Technical Sessions
Key to Session/Paper Numbers

A Coatings for Use at High Temperature

B Hard Coatings and Vapor Deposition Technology

C Optical Thin Films

D Carbon and Nitride Materials: Synthesis-Structure-Property Relationships

E Tribology and Mechanical Behavior of Coatings and Thin Films

F Characterization: Linking Synthesis Properties and Microstructure

G Applications, Manufacturing, and Equipment

H New Horizons in Coatings and Thin Films

TS1 Atomistics of Thin Films Growth

TS2 Coatings for Fuel Cells

TS3 Bioactive Coatings and Biofunctionalization

TS4 Surface Engineering for Thermal Management

Program Numbers are listed with the symposium letter first, the session number second, and the number of the paper last (i.e., A1-1-1 = Symposium A, session 1 denotes the half day session in which the talk is being presented, morning or afternoon, and the paper number slot is -1). Sessions sponsored by two divisions are labeled with both acronyms (i.e., C2/E5, E5/C2).

SYMPOSIUM SCHEDULE POINTERS:

- All morning sessions begin at 8:00 am (except Monday, when the sessions begin at 10:00 am following the 8:00 am Plenary Session).
- All afternoon sessions begin at 1:30 pm, following the lunch break that starts at 12:10 pm.
- Invited speakers, (marked invited in the program) have 40 minutes; contributed speakers have a 20-minute limit.

IF YOU ARE MAKING AN ORAL PRESENTATION:

All technical session rooms are equipped with computers, LCD projectors, screens, laser pointers and microphones. Please test your presentation materials to be certain they are compatible with the equipment being provided in the technical sessions. The Presenter's Preview Room is the Dover Room near the Tiki Pavilion. Please allow ample time for this; preferably the day before you are scheduled to present – not immediately before your talk. The hours are Sunday, 3:30 - 6:30 pm and Monday – Thursday, 8:00 am - 5:30 pm.

IF YOU ARE MAKING A POSTER PRESENTATION:

Boards will be available the morning of the scheduled presentation, and must be removed by 9:00 p.m. that evening. An informal sign having the paper number, title and author listing will aid you in the location of which board your poster is to be displayed. Poster Presenters need to be at the side of their presentation during the entire Poster Session; this is to encourage discussions with interested persons attending the session.

	Hard Coatings and Vapor Deposition Technology **Room: Golden West - Session B2-1** **Arc and E-Beam Coatings and Technologies** **Moderators:** V.I. Gorokhovsky, Arcomac Surface Engineering, LLC, J. Vetter, Sulzer Metaplas GmbH	**Hard Coatings and Vapor Deposition Technology** **Room: Royal Palm 1-3 - Session B3-1** **CVD Coatings and Technologies** **Moderators:** M. Pons, SIMAP, Grenoble-INP, CNRS, H. Holzschuh, Walter AG
10:00 am	**B2-1-1 Invited** Metallic Film Deposition using a Vacuum Arc Plasma Source with a Refractory Anode, I. BEILIS, **R.L. BOXMAN**, Tel Aviv University, Israel	**B3-1-2** Microstructural Investigation of CVD Alumina Coatings Grown on Single Crystals of α-Al_2O_3, **S. CANOVIC**, Chalmers University of Technology, Sweden, B. LJUNGBERG, C. BJÖRMANDER, Sandvik Tooling, Sweden, M. HALVARSSON, Chalmers University of Technology, Sweden
10:20 am	Invited talk continued.	**B3-1-3** Residual Stress Evolution in CVD Multilayer Coatings on Cutting Tools at High Temperature, **M. KLAUS**, Technische Universität Berlin, Germany, CH. GENZEL, Helmholtz-Centre Berlin for Materials and Energy, Germany, H. HOLZSCHUH, Walter AG, Germany
10:40 am	**B2-1-3** Investigation of Electrochromic Properties of Nickel Oxide Films Prepared by Electrom Beam Evaporation, **D.R. SAHU**, T.-J. WU, J.-L. HUANG, National Cheng Kung University, Taiwan	**B3-1-4** Surface Treatments for Enhancing the Adhesion of Nanocrystalline Diamond Coatings to Tungsten Carbide Micro End Mills, **P.J. HEANEY**, C.D. TORRES, University of Wisconsin - Madison, M.A. HAMILTON, University of Pennsylvania, A.V. SUMANT, Argonne National Laboratory, K. SRIDHARAN, University of Wisconsin - Madison, R.W. CARPICK, University of Pennsylvania, F.E. PFEFFERKORN, University of Wisconsin - Madison
11:00 am	**B2-1-4** Surface Modification of Iron Containing Aluminum Alloys by Treating with Copper Plasma Produced with Cathodic Arc, **B. CORLU**, M. URGEN, Istanbul Technical University, Turkey	
11:20 am	**B2-1-6** Optical Reflective Performance of Copper Alloy Thin Films with Ti-Zr and Al-Zr Additions, **C.-Y. SU**, National Taipei University of Technology, Taiwan, C.-Y. TSAY, Feng Chia University, Taiwan, C.-W. CHANG, National Taipei University of Technology, Taiwan, C.-H. HSU, Tatung University, Taiwan, C.-K. LIN, Feng Chia University, Taiwan	

Monday Morning, April 27, 2009

	Carbon and Nitride Materials: Synthesis-Structure-Property Relationships - Room: Royal Palm 4-6 – Session D2-1 **Diamond and Diamond-Like Carbon Materials** **Moderators:** O. Shenderova, International Technology Center, R. Hauert, Empa	**Tribology and Mechanical Behavior of Coatings and Thin Films - Room: California - Session E2-1** **Mechanical Properties and Adhesion** **Moderators:** R. Chromik, McGill University, J. Michler, Empa
10:00 am	**D2-1-1** Preparation and Comparison of a-C:H Coatings using Reactive Sputter Techniques, **M. KEUNECKE**, K. WEIGEL, K. BEWILOGUA, Fraunhofer Institute for Surface Engineering and Thin Films, Germany, R. CREMER, H.-G. FUSS CemeCon A.G., Germany	**E2-1-1 Invited** Fracture and Failure in Polymer – Metal Systems for Flexible Electronics, **D.F. BAHR**, J.D. YEAGER, Washington State University, M.S. KENNEDY, Clemson University, N.R. MOODY, Sandia National Laboratories
10:20 am	**D2-1-2** Effect of Boron Incorporation on the Structure and Electrical Properties of Diamond-Like Carbon Films Deposited by Femtosecond and Nanosecond Pulsed Laser Ablation, A. SIKORA, Université Jean Monnet, France, O. BOURGEOIS, J.L. GARDEN, CNRS et Université de Grenoble, France, J.N. ROUZAUD, Ecole Normale Supérieure, France, J.C. SANCHEZ-LOPEZ, T.C. ROJAS, Instituto de Ciencia de Materiales de Sevilla, Spain, A.S. LOIR, F. GARRELIE, **C. DONNET**, Université Jean Monnet, France	Invited talk continued.
10:40 am	**D2-1-3 Invited** Retrospective Lifetime Prediction of Failed and Explanted DLC Coated Hip Joint Balls, **G. TÄGER**, L.E. PODLESKA, University Essen, Germany, C.V. FALUB, G. THORWARTH, M. STIEFEL, R. HAUERT, Empa, Switzerland	**E2-1-4** Mechanical Behavior and Stability of Self Standing Films and Film on Substrate Systems, **M. IGNAT**, SIMAP Grenoble INP, France, C. SEGUINEAU, Novamems France, A. CHOUAF, ENSEM Casablanca Maroc, France
11:00 am	Invited talk continued.	**E2-1-5** Procedures and Equations for the Next Generation of Surface Testers Solving the Problem of Pile-Up, Sink-In and Making Area-Function-Calibration Obsolete, **N. SCHWARZER**, Saxonian Institute of Surface Mechanics, Germany
11:20 am	**D2-1-5** Properties of Nanostructured Surfaces of DLC Thin Films Prepared by Pulsed-DC PECVD, **C. CORBELLA**, S. PORTAL, M. RUBIO-ROY, E. BERTRAN, M.C. POLO, E. PASCUAL, J.L. ANDÚJAR, University of Barcelona, Spain	**E2-1-6** Interfacial Fracture of Thin Hard Films on Compliant Substrates, **N.R. MOODY**, Sandia National Laboratories, M.S. KENNEDY, Clemson University, M.J. CORDILL, Erich Schmid Institute, D.P. ADAMS, J.A. EMERSON, Sandia National Laboratories, D.F. BAHR, Washington State University, E.D. REEDY, Sandia National Laboratories
11:40 am	**D2-1-6** Topography Development of Toluene Based a-C:H Coatings Deposited by RF-PECVD, **C. HORMANN**, S. MEIER, Fraunhofer Institute of Mechanics of Materials IWM, Germany	Focused Topic Session Micro Materials 12:15 – 1:15 pm

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G3
Atmospheric Plasma, Hollow Cathode, and Hybrid Technologies - Moderators: V.H. Baggio-Scheid, General-Command of Aerospace Technology, A. Leyland, University of Sheffield

10:00 am	**G3-1 Invited** Synthesis of Amorphous Carbon Films using Large-scale Atmospheric Pressure Plasma CVD Equipment, **H. KODAMA**, Kanagawa Academy of Science and Technology, Japan, T. HORIUCHI, M. KUMAGAI, Kanagawa Industrial Technology Center, Japan, T. SUZUKI, Keio University, Japan	
10:20 am	Invited talk continued.	
10:40 am	**G3-3** Atmospheric Pressure Plasma Coatings to Improve the Tribological Behaviour of Rubbers Against Metal Counter Bodies, **B. VERHEYDE**, VITO-MAT, Belgium, A. VANHULSEL, D. HAVERMANS, VITO Belgium, M. WANGENHEIM, IDS Germany	
11:00 am	**G3-4** Fatigue Life Determination of Plasma Nitrided Medical Grade CoCrMo Alloy, **Ö. BAYRAK**, F. YETIM, A. ALSARAN, A. ÇELIK, Ataturk University, Turkey	
11:20 am	**G3-5 Invited** Mass and Energy Spectrometry of Plasmas at Atmospheric Pressure, **J.A. REES**, D.L. SEYMOUR, T.D. WHITMORE, Hiden Analytical Ltd., United Kingdom	
11:40 am	Invited talk continued.	Tutorial Oral and Poster Presentations 12:15 – 1:15 pm Sunrise Room

NOTES

Monday Afternoon, April 27, 2009

	Hard Coatings and Vapor Deposition Technology **Room: Golden West - Session B2-2** **Arc and E-Beam Coatings and Technologies** **Moderators:** V.I. Gorokhovsky, Arcomac Surface Engineering, LLC, J. Vetter, Sulzer Metaplas GmbH	**Hard Coatings and Vapor Deposition Technology** **Room: Royal Palm 1-3 - Session B3-2** **CVD Coatings and Technologies** **Moderators:** M. Pons, SIMAP, Grenoble-INP, CNRS, H. Holzschuh, Walter AG
1:30 pm	**B2-2-1 Invited** Trends and Applications of Cathodic Arc Evaporation, **O. ZIMMER**, Fraunhofer Institute for Materiasl and Beam Technology, Germany, V. WEIHNACHT, H.-J. SCHEIBE, Fraunhofer Institute for Materials and Beam Technology, Germany	**B3-2-1** Mechanical Behavior of PECVD Silicon Oxide, Oxynitride and Nitride: A Comparative Micro-Cantilever Deflection Study, **K. MATOY**, KAI Kompetenzzentrum automobil- und Industrie-Elektronik GmbH, Austria, R. PIPPAN, Erich Schmid Institute Leoben, Austria, G. DEHM, University of Leoben, Austria, H. SCHÖNHERR, T. DETZEL, Infineon Technologies AG, Austria
1:50 pm	Invited talk continued.	**B3-2-2** Properties and Tribological Behaviour of CVD TiBN-Coated Cutting Tools, **D. STIENS**, Walter AG, Germany, I. DREILING, University of Tuebingen, Germany, H. HOLZSCHUH, Walter AG, Germany, T. CHASSE, University of Tuebingen, Germany
2:10 pm	**B2-2-3** Zirconium and Aluminum Arc Behavior and Ion Current Generation During Metal and Oxide Deposition, **I. ZUKERMAN**, Tel Aviv University / NRC-Negev, Israel, V.N. ZHITOMIRSKY, Tel Aviv University, Isreal, A. RAVEH, NRC-Negev, Israel, R.L. BOXMAN, Tel Aviv University, Israel, S.K. KIM, University of Ulsan, Korea	**B3-2-3 Invited** Experimental Thermodynamics for the Evaluation of ALD Growth Processes, **E. BLANQUET**, P VIOLET, SIMAP, Grenoble-INP, CNRS, France, D. MONNIER, STMicroelectronics, France, I. NUTA, SIMAP, Grenoble-INP, CNRS, France, C. CHATILLON, SIMAP, Grenoble-INP, CNRS
2:30 pm	**B2-2-4** Phase Formation in Oxide Layers Synthesized by Pulsed Arc Evaporation, M. DOEBELI, Paul Scherrer Inst & ETH Zuerich, A. DOMMANN, Centre Suissed, J. HERRÁN, Centre Suissed & Tecnun San Sebastian, E. KALCHBRENNER, OC Oerlikon Balzers AG, A. NEELS, Univ of Neuchâtel, J. RAMM, **H. RUDIGIER**, OC Oerlikon Balzers AG, J. THOMAS, IFW Dresden, B. WIDRIG, OC Oerlikon Balzers AG	Invited talk continued.
2:50 pm	**B2-2-5** Influence of Bias Pulsing on Structure and Properties of Arc-Evaporated $(Al_{1-x}Cr_x)_2O_3$ Hard Coatings, **M. POHLER**, G.A. FONTALVO, R. FRANZ, University of Leoben, Austria, D. KURAPOV, OC Oerlikon Balzers AG, Liechtenstein, C. POLZER, PLANSEE Composite Materials GmbH, Austria, C. MITTERER, University of Leoben, Austria	**B3-2-5** Effect of Air Exposure on Cu Diffusion Barrier Properties of TaN Thin Films Grown by Atomic Layer Deposition, **O.H. KIM**, D.J. KIM, K.C. KIM, H.M. AJMERA, T.J. ANDERSON, J. KOLLER, L. MCELWEE-WHITE, D.P. NORTON, University of Florida
3:10 pm	**B2-2-6** Deposition of ZrO_2/Al_2O_3 Thin Films by Cathodic Arc Plasma Deposition, **S.K. KIM**, V.V. LE, University of Ulsan, Korea, J.W. LEE, KAIST, Korea, V.N. ZHITOMIRSKY, R.L. BOXMAN, Tel Aviv University, Israel	**B3-2-6** DLI-CVD of M-TiO_2 (M = Ag, Cu) Antibacterial Thin Films, J. MUNGKALASIRI, Cirimat - Cea, France, L. BEDEL, F. EMIEUX, CEA, France, J. DORÉ, F. RENAUD, Nosoco.tec, France, **F. MAURY**, CNRS ENSIACET, France
3:30 pm	**B2-2-8** Hollow Cathode Plasma Assisted AlTiSiN-Thin Film Deposition by Vacuum Arc P+-Technology for Advanced Wear Protection of Tools, **M. HOLZHERR**, T. SCHMIDT, M. FALZ, K.-D. STEINBORN, VTD Vakuumtechnik Dresden GmbH, Germany	**B3-2-7** Evaluation of the Tungsten Diphenylhydrazido Complex $Cl_4(CH_3CN)W(NNPh_2)$ as a Precursor for CVD of WN_xC_y, **D.J. KIM**, O.H. KIM, T.J. ANDERSON, J. KOLLER, L. MCELWEE-WHITE, L.C. LEU, D.P. NORTON, University of Florida
3:50 pm	**B2-2-9** Synthesis and Mechanical Properties of CrN_x Coatings Deposited by Arc Ion Plating, M. ZHANG, **K.H. KIM**, Pusan National University, Korea, G. LIN, School of Physics and Optoelectronic Technology, China, C. DONG, School of Material Science and Engineering, China	**B3-2-8** Silicon Carbide CVD for Polycrystalline Growth: Thermodynamic Evaluation vs. Film Morphology, J. TREVARTHEN, SIMAP, Grenoble-INP, CNRS, France, **A. CLAUDEL**, R. MARTIN, ACERDE, France, G. CHICHIGNOUD, M. MORAIS, SIMAP, Grenoble-INP, CNRS, France, E. BLANQUET, SIMAP, Grenoble-INP, CNRS, Framce, M. PONS, SIMAP, Grenoble-INP, CNRS, France
4:10 pm	**B2-2-10** Characterization of Worn Ti-Si Cathodes used for Reactive Cathodic Arc Evaporation, J. ZHU, A. ERIKSSON, N. GHAFOOR, Linköping University, sweden, M. JOHANSSON, SECO Tools AB, Sweden, F. GIULIANI, Linköping University, Sweden, J. SJÖLÉN, SECO Tools AB, Sweden, L. HULTMANN, J. ROSÉN, **M. ODÉN**, Linköping University, Sweden	**B3-2-9** Adhesion and Homogeneity of a-C:H:Si Films Deposited in a Modified Plasma Nitriding System for Industrial Application, **C. FORSICH**, D. HEIM, University of Applied Sciences, Austria, T. MUELLER, Rubig GmbH & Co KG Anlagentechnik, Austria
4:30 pm	**B2-2-11** Structure and Composition of Arc Deposited $(Ti_{1-x}Si_x)(C_{1-y}N_y)_z$ Coatings, **A. ERIKSSON**, J. ZHU, M. ODÉN, Linköping University, Sweden, M. JOHANSSON, J. SJÖLÉN, SECO Tools AB, Sweden, L. HULTMAN, J. ROSÉN, Linköping University, Sweden	**B3-2-11** Molecular Beam Mass Spectrometry and Modelling of CH_4-CO_2 Plasmas in Relation with Polycrystalline and Nano-Smooth Diamond Deposition, T. GRIES, **L. VANDENBULCKE**, S. DE PERSIS, C. MET, ICARE-CNRS, France, O. AUBRY, GREMI-Polytech/Université d'Orléans, France, J.L. DELFAU, ICARE-CNRS, France
	Welcome Mixer Atlas Foyer 6:00 – 7:00 pm	Focused Topic Session Vamas TWA 22 5:30 – 6:30 pm

Monday Afternoon, April 27, 2009

	Carbon and Nitride Materials: Synthesis-Structure-Property Relationships **Room: Royal Palm 4-6 - Session D2-2** **Diamond and Diamond-Like Carbon Materials** **Moderators:** O. Shenderova, International Technology Center, R. Hauert, Empa	**Tribology and Mechanical Behavior of Coatings and Thin Films** **Room: California - Session E2-2** **Mechanical Properties and Adhesion** **Moderators:** R. Chromik, McGill University, J. Michler, Empa
1:30 pm	**D2-2-1** Raman Analysis of DLC Coated Engine Components with Complex Shape: Understanding of Wear Mechanisms, **O. JARRY**, Sorevi, C. JAOUL, P. TRISTANT, M. COLAS, T. MERLE-MEJEAN, C. DUBLANCHE-TIXIER, University of Limoges, CNRS, SPCTS, France	**E2-2-2** Parametric Study on the Behavior of a Film/Substrate System to Limit Crack Propagation by the Finite Element Method, **N.K. FUKUMASU**, R.M. SOUZA, University of São Paulo, Brazil, M. IGNAT, SIMAP Grenoble INP, France
1:50 pm	**D2-2-2** Hydrogen-Free Hard Amorphous Carbon Coatings: Properties by Cathodic Vacuum Arc Evaporation and Magnetron Sputter Deposition, M. STUEBER, Forschungszentrum Karlsruhe, Germany, **J. VETTER**, Sulzer Metaplas GmbH, Germany, H. LEISTE, S. ULRICH, C. ZIEBERT, Forschungszentrum Karlsruhe, Germany, J. MUELLER, G. ERKENS, Sulzer Metaplas GmbH, Germany	**E2-2-3** Nano-Impact – a Reliable Tool to Predict Coating Performance, **B. BEAKE**, Micro Materials Ltd, United Kingdom
2:10 pm	**D2-2-3 Invited** Non-Destructive Characterization of Carbon Films, **A. FERRARI**, University of Cambridge, United Kingdom	**E2-2-4** Adhesion of Diamond Like Carbon (DLC) Coatings on Metalic Biomedical Implants, **C.V. FALUB**, R. HAUERT, G. THORWATH, U. MÜLLER, M. PARLINSKA-WOJTAN, C. AFFOLTER, P. SCHMUTZ, J. MICHLER, Empa, Switzerland, M. TOBLER, IonBond AG, C. VOISARD, Synthes GmbH
2:30 pm	Invited talk continued.	**E2-2-5 Invited** A Micro-FEM Modelling Based Fracture Mechanics Approach to Wear Resistance Assessment of Thin Hard Coatings, **K. HOLMBERG**, VTT Technical Research Centre of Finland
2:50 pm	**D2-2-5** Characterisation of a Plasma Beam Source During Duplex Treatment of High Speed Steel for Depositing DLC Coatings, **M. FENKER**, M. BALZER, N. BERMAYER, Forschungsinstitut Edelmetalle & Metallchemie, Germany, M. RINKE, M. STÜBER, S. ULRICH, J. YE, Forschungszentrum Karlsruhe GmbH, Germany	Invited talk continued.
3:10 pm	**D2-2-6** Correlation of Plasma Properties and Microstructures of DLC Films Deposited by Laser Induced High Current Pulsed Arc, J.-B. WU, **C.-Y. CHEN**, Industrial Technology Research Institute, Taiwan, M.-Y. LI, National Nano Device Laboratories, Taiwan, M.-S. LEU, A.-K. LI, Industrial Technology Research Institute, Taiwan	**E2-2-13** Structural and Mechanical Properties of Graded and Multilayered $Al_xTi_{1-x}N$/CrN Coatings Synthesized by a Cathodic-Arc Deposition Process, **Y.-Y. CHANG**, C.-P. CHANG, C.-Y. HSIAO, D.-Y. WANG, Mingdao University, Taiwan
3:30 pm	**D2-2-7** Amorphous Carbon Deposited By Sputtering And Plasma Enhanced Chemical Vapor Deposition Without Vacuum Pumping During Deposition, G.A. VIANA, **F.C. MARQUES**, University of Campinas, Brazil	**E2-2-9** Effect of Aging on Adhesion of Black Anodic Coatings for Space Applications, **Y. GOUEFFON**, CNES, France, C. MABRU, Universite de Toulouse, ISAE, France, L. ARURAULT, Universite de Toulouse, CIRIMAT-Institut Carnot, France, C. TONON, Astrium, France, P. GUIGUE, CNES, France
3:50 pm	**D2-2-8** Thermal Annealing Effect on Tribological, Electrical, and Corrosion Properties of DLC Coatings, L. WANG, Z.J. PENG, University of Windsor, Canada, J. HOUSDEN, E. SPAIN, Tecvac Ltd., **X. NIE**, University of Windsor, Canada	**E2-2-10** Effect of Hydrogen on the Mechanical and Structural Properties of SiC-Derived Carbon Films, D.-S. LIM, **H.-J. CHOI**, Korea University, Korea, M.J. MCNALLAN, University of Illinois at Chicago, Y.H. SOHN, University of Central Florida
4:10 pm		**E2-2-11** Tensile Testing of Amorphous Diamond-Like Carbon and Crystalline Diamond Coatings, J. SCHAUFLER, **K. DURST**, K. KELLERMANN, S. ROSIWALL, M. GÖKEN, University Erlangen Nürnberg, Germany
4:30 pm		**E2-2-12** The Mechanical Strength of Micro-to-Nanoscale Porous Ag Coatings, A. JANKOWSKI, **H. AHMED**, Texas Tech University
	Welcome Mixer Atlas Foyer 6:00 – 7:00 pm	Welcome Mixer Atlas Foyer 6:00 – 7:00 pm

Monday Afternoon, April 27, 2009

	Applications, Manufacturing, and Equipment **Room: Sunrise - Session G5/H5** **Coatings for Renewable Energy Systems** **Moderators:** L. Bardos, Uppsala University, S. Roy, Newcastle University	**Bioactive Coatings and Biofunctionalization** **Room: Sunset - Session TS3-1** **Bioactive Coatings and Surface Biofunctionalization** **Moderators:** D.V. Shtansky, State Technological University, Moscow Institute of Steel and Alloys, S. Kumar, University of South Australia
1:30 pm	**G5/H5-1 Invited** Electrochemical Energy Conversion: Coatings for Fuel Cells, Electrolysers and Batteries, **E. ROBERTS**, Unversity of Manchester, United Kingdom	**TS3-1-1 Invited** Nitrogen-Rich Plasma Polymer Coatings ("PPE:N") for Bio-Medical Applications in Orthopaedic and Vascular Therapies, **M.R. WERTHEIMER**, P.L. GIRARD-LAURIAULT, F. TRUICA-MARASSESCU, J.-C. RUIZ, École Polytechnique, Canada, H.T. WANG, J. ANTONIOU, F. MWALE, McGill University, Canada, S. LEROUGE, Notre-Dame Hospital, Canada
1:50 pm	Invited talk continued.	Invited talk continued.
2:10 pm	**G5/H5-3 Invited** Thin-Film Based Superconducting Wires for Electric Power Applications, **V. SELVAMANICKAM**, University of Houston	**TS3-1-4** Characterisation of Ca- and P-Containing PEO Coatings on Ti Substrates, **A. YEROKHIN**, A. PILKINGTON, A. MATTHEWS, University of Sheffield, United Kingdom
2:30 pm	Invited talk continued.	**TS3-1-5** Effects of Sterilization and Heat Treatment on Alumina Coated Surgical Instruments, **A.C. RUIZ**, M. KAVDIA, University of Arkansas, S. KUMAR, University of South Australia, M.H. GORDON, University of Arkansas, H.H. ABU-SAFE, Lebanese American University, Lebanon, S. DAVIS, Philander smith College
2:50 pm	**G5/H5-5** Deposition and Characterisation of NiO_x Films by Reactive Sputtering for Application in Dye Sensitized Solar Cells, **M. AWAIS**, M. RAHMAN, D. MACELROY, University College Dublin, Ireland, N. COBURN, D. DINI, J.G. VOS, Dublin City University, Ireland, D.P. DOWLING, University College Dublin, Ireland	**TS3-1-7** Spray-Pyrolyzed Hydroxyapatite Thin-Film Coatings, **M. AGUILAR-FRUTIS**, CICATA-IPN, Mexico, S. KUMAR, University of South Australia, C. FALCONY, CINVESTAV-IPN, Mexico
3:10 pm	**G5/H5-6 Invited** Renewable Energy Systems - What Coatings Do We Need?, **H. BERNHOFF**, M. BERGKVIST, Uppsala University, Sweden	**TS3-1-8** The Influence of Electrochemical Parameters on the Microstructures and Bactericidal Abilities of Anodized Titanium Dioxide Nanotube Arrays, **L.-S. LIU**, Y.-J. CHANG, J.-W. LEE, H.-P. CHEN, Tungnan University, Taiwan
3:30 pm	Invited talk continued.	**TS3-1-9** Biocompatibility of Niobium-Based Coatings, G. RAMÍREZ, **S.E. RODIL**, H. ARZATE, Universidad Nacional Autónoma de México, J.J. OLAYA, Universidad Nacional de Colombia, E. CAMPS, Instituto Nacional de Investigaciones Nucleares, México, S. MUHL, Universidad Nacional Autónoma de México
3:50 pm		**TS3-1-10** Low-temperature Deposition of Anatase TiO_2 on Medical Grade Polyetheretherketone to Assist Osseous Integration, **H.-K. TSOU**, Feng Chia University, Taichung Veterans General Hospital & Jen-The Junior College of Medicine, P-Y. HSIEH, Feng Chia Univ, C.-J. CHUNG, Central Taiwan Univ of Science and Technology, C.-H. TANG, China Medical Univ, T.-W. SHYR, J.-L. HE, Feng Chia Univ
4:10 pm		**TS3-1-11** Investigation of Microstructure and Properties of Plasma-Sprayed HAp-TiO_2 graded Coatings, **A. IWANIAK**, M. SOZANSKA, The Silesian University of Technology, Poland
	Welcome Reception Atlas Foyer 6:00 – 7:00 pm	Welcome Reception Atlas Foyer 6:00 – 7:00 pm

NOTES

Tuesday Morning, April 28, 2009

	Coatings for Use at High Temperature **Room: Royal Palm 1-3 - Session A1-1** **Coatings to Resist High Temperature Oxidation** **Moderators:** Y. Zhang, Tennessee Technological University, K. Stiller, Chalmers University of Technology	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B5-1** **Properties and Characterization of Hard Coatings and Surfaces - Moderators:** Y.W. Chung, Northwestern University, G. Abadias, Universite de Poitiers-CNRS, M. Fenker, Forschungsinstitut Edelmetalle & Metallchemie
8:00 am	**A1-1-1** Platinum Nucleation Mechanisms and their Role in Platinum Aluminide Coatings, **M.A. CRAIG**, L. CHIRIVI, K. LONG, M. ROBINSON, Cranfield University, United Kingdom, D. RICKERBY, Rolls Royce, United Kingdom, J.R. NICHOLLS, Cranfield University, United Kingdom	**B5-1-1 Invited** Contribution of an In Situ Microscopic Approach for a Better Understanding of the Oxidation Phenomena of TiSiN Nanocomposite Films Deposited on Steel, **P. STEYER**, Laboratoire MATEIS, France, A. MÈGE-REVIL, G. THOLLET, INSA de Lyon, France, R. CHIRIAC, UCBL-LMI, France, C. SIGALA, C. ESNOUF, INSA de Lyon, France
8:20 am	**A1-1-2** Oxidation and Volatilization of Coatings for Power Generation: New Super Sultra Critical Steam Turbines, **F.J. PEREZ**, S.I. CASTANEDA, M.P. HIERRO, F.J. BOLIVAR, Universidad Complutense, Spain	Invited talk continued.
8:40 am	**A1-1-3 Invited** High Temperature Coatings for Gas Turbine Engines, **B.T. HAZEL,** J. RIGNEY, M. GORMAN, B. BOUTWELL, D.WORKMAN AND R. DAROLIA., GE AircraftEngines	**B5-1-3** Synthesis and Oxidation Resistance of TiAlSiN and Multilayer TiAlSiN/CrAlN Coating, **N. FUKUMOTO**, H. EZURA, T. SUZUKI, Keio University, Japan ****STUDENT FINALIST****
9:00 am	Invited talk continued.	**B5-1-4** Non-Linear Finite Element Modeling of Ultra-Hard Nanocomposites: The Ratio of Hardness to Yield Strength, Origin of the Ultra-Hardness and the Limits of Nanoindentation to its Measurement, M.G.J. VEPREK-HEIJMAN, Technical University Munich, Germany, R.G. VEPREK, Integrated Systems Laboratory, ETH, Switzerland, A.S. ARGON, D.M. PARKS, Massachusetts Institute of Technology, **S. VEPREK**, Technical University Munich, Germany
9:20 am	**A1-1-6** Computational Investigation into the Phase Stability and Phase Distribution in the Ni-Cr-Al Ternary System, **K. MA**, J.M. SCHOENUNG, UC Davis	**B5-1-5 Invited** Secondary Ion Mass Spectrometry and Auger Electron Spectroscopy Used at nm Range Depth Resolutions and Tens of nm Range Lateral Resolution for Ultimate Characterization of Nanostructured Hard Coatings, **D. DUDAY**, P. CHOQUET, V. VALLE, J.N. AUDINOT, J. GUILLOT, V. HODY, H.N. MIGEON, CRP-Gabriel Lippmann, Luxembourg
9:40 am	**A1-1-7** New Generation HIPIMS Nanostructured Coatings to Perform in Severe High Temperature Environment, **P.EH. HOVSEPIAN**, A.P. EHIASARIAN, Sheffield Hallam University, United Kingdom, C. LEYENS, TU-Cottbus, Germany, R. BRAUN, German Aerospace Centre, Germany, F.J. TRUJILLO, Universidad Complutense de Madrid, Spain	Invited talk continued.
10:00 am	**A1-1-9** Investigation of an Oxidation Barrier for IN 617 at Low pO_2, **E.A. CLARK**, J.Y. YANG, University of California, Santa Barbara, A.G. EVANS, University of California Santa Barbara, C.G. LEVI, University of California, Santa Barbara	**B5-1-7** Solid Solution Nanocomposite Cr-Si-N Thin Films, **L. CASTALDI**, Empa, Switzerland, V. SHKLOVER, ETH Zürich, Switzerland, P. SCHWALLER, Empa, Switzerland, R. SANJINES, EPFL, Switzerland, J. PATSCHEIDER, Empa, Switzerland
10:20 am	**A1-1-10** Stable Electrodes and Passivation Coatings for High Temperature Acoustic Wave Sensors, **B. STURTEVANT**, D.J. FRANKEL, G. BERNHARDT, T. MOONLIGHT, M. PEREIRA DA CUNHA, R.J. LAD, University of Maine	**B5-1-8** Microstructure and Erosion Resistance of Nanocomposite Ti-Si-C-N Coatings Deposited using HMDSN Precursor, **R. WEI**, C. RINCON, E. LANGA, Southwest Research Institute
10:40 am	**A1-1-5** High Temperature Oxidation Behavior of RF Magnetron Sputtered NiCrAl Coatings on Superalloys, **R.A. MAHESH**, R. JAYAGANTHAN, S. PRAKASH, Indian Institute of Technology, India	**B5-1-10** Mechanical and Tribological Properties of Nano-Scale Multilayered TiN/CrN Coatings, **M. KONCHADY**, S. YARMOLENKO, D. PAI, J. SANKAR, North Carolina A&T State University
11:00 am		**B5-1-11** The Effects of Interlayer Grading and Pulsed Biasing on Chromium Nitride Films, **J.A. FREEMAN**, P.J. KELLY, G. WEST, Manchester Metropolitan University, United Kingdom, N.M. RENEVIER, University of Central Lancashire, United Kingdom, J.W. BRADLEY, University of Liverpool, Untied Kingdom
11:20 am		**B5-1-12** A New Approach to the Synthesis of Adherent Hard Coatings with High Toughness, **A.N. RANADE**, Northwestern University, L.R. KRISHNA, International Advanced Research Centre (ARCI), India, Y.W. CHUNG, Northwestern University ****STUDENT FINALIST****
11:40 am	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm	**B5-1-9** Microstructural and Mechanical Properties of Sputter Deposited TiN/SiN_x Multilayer Coatings on Si Substrate, **V. CHAWLA**, R. JAYAGANTHAN, R. CHANDRA, Indian Institute of Technology Roorkee, India

	Carbon and Nitride Materials: Synthesis-Structure-Property Relationships **Room: Royal Palm 4-6 - Session D2-3** **Diamond and Diamond-Like Carbon Materials** **Moderators:** O. Shenderova, International Technology Center, R. Hauert, Empa	**Tribology and Mechanical Behavior of Coatings and Thin Films** **Room: California - Session E2-3** **Mechanical Properties and Adhesion** **Moderators:** R. Chromik, McGill University, J. Michler, Empa
8:00 am	**D2-3-1 Invited** Diamond Functionalizatin for Biosensing Applications and the Detection of NO_x, **J.T. GLASS**, J.B. SUND, C.P. CAUSEY, S.D. WOLTER, C.B. PARKER, Duke University, B.R. STONER, RTI International, E.J. TOONE, Duke University, P. NATISHAN, US Naval Research Laboratory	**E2-3-1 Invited** Mechanical Properties of Metals Below 100 nm - Effects of Size, Temperature and Geometry, **R. SPOLENAK**, ETH Zurich, Switzerland
8:20 am	Invited talk continued.	Invited talk continued.
8:40 am	**D2-3-3** CVD-Diamond Coating on Steel Substrates: Basics and Applications, **J. FANDREY**, K. KELLERMANN, S.M. ROSIWAL, R.F. SINGER, University Erlangen-Nuremberg, Germany	**E2-3-3** Hardness Mapping and Nanoindentation of Cold-Spray Coatings, **D. GOLDBAUM**, T. SHARIFF, A. REZAEIAN, McGill University, Canada, E. IRISSOU, J.-G. LEGOUX, National Research Council Canada (NRC), Canada, R. CHROMIK, S. YUE, McGill University, Canada
9:00 am	**D2-3-4** CVD-Diamond on Titanium Substrate as Electrode for Wastewater Treatment, **K. BAYERLEIN**, University Erlangen-Nuremberg, Germany, M. FORETA, Diaccon GmbH, Germany, S.M. ROSIWAL, R.F. SINGER, University Erlangen-Nuremberg, Germany	**E2-3-6** Ultra Low Indentation Approach to Stress-Strain Behavior of Thin Films, J. NOHAVA, G. FAVARO, CSM Instruments SA, Swizerland, **N. RANDALL**, CSM Instruments Inc.
9:20 am	**D2-3-5** Manufacturing and Properties of Self-Supporting Nano-Crystalline Diamond Foil, **M.A. LODES**, S.M. ROSIWAL, R.F. SINGER, University Erlangen-Nuremberg, Germany	**E2-3-5** Mechanical Properties of TiN/Ti Multilayer Coating Deposited by LAFAD Technology, **Y. CHENG**, T. BROWNE, B. HECKERMAN, American Eagle Instruments, Inc., C. BOWMAN, V.I. GOROKHOVSKY, Arcomac Surface Engineering, LLC.
9:40 am	**D2-3-6** Mechanical Properties of Ultrananocrystalline Diamond Films with Different Nucleation Densities, G. FAVARO, CSM Instruments SA, Switzerland, **C. POPOV**, University of Kassel, Germany, W. KULISCH, European Commission Joint Research Centre, Italy	**E2-3-4** Small-Scale Tests for Investigating Plastic Flow in Brittle Crystals, **S. KORTE**, W.J. CLEGG, University of Cambridge, United Kingdom
10:00 am	**D2-3-7 Invited** Recent Developments in the Application of Single-Nano Diamond Particles, **E. ŌSAWA**, Shinshu University, Japan	**E2-3-7 Invited** Mechanical and Electrical Behavior of Oxide-Dispersion Strengthened Au Microcontacts, **R. VINCI**, T. BANNURU, W.L. BROWN, T. HUMPLIK, K. MONGKOLSUTTIRAT, Lehigh University
10:20 am	Invited talk continued.	Invited talk continued.
10:40 am	**D2-3-12** Influence of Surface Roughness on the Tribological Properties of HF-CVD Diamond Coated Heat-Treatable Steel, **K. KELLERMANN**, S. EHRHARDT, J. FANDREY, S.M. ROSIWAL, R.F. SINGER, University Erlangen-Nuremberg, Germany	**E2-3-9** Factors Affecting the Critical Indenter Penetration for the Measurement of the Hardness and Young's Modulus of Coatings, **S.J. BULL**, J. CHEN, Newcastle University, United Kingdom
11:00 am	**D2-3-11** The Thermostability of Detonation Nanodiamond, **A.N. PANOVA**, G.P. BOGATYREVA, National Academy of Sciences of Ukraine, V.J. ZABUGA, G.G TSAPYUK, National Taras Shevchenko University of Kyiv, Ukraine, S.A. LISOVENKO, National Academy of Sciences of Ukraine	**E2-3-10** Influence of Intrinsic Stress on the Measurement of the Onset of Yielding for Thin Films Using Depth-Sensing Spherical Indentation, **M. HERRMANN**, A. CLAUSNER, F. RICHTER, Chemnitz University of Technology, Germany
11:20 am	**D2-3-9** Comparative Study of Polishing Processes of Semi-Conductor Crystals and other Materials Surfaces by Detonation Nanodiamonds (DNA) and Colloidal Silica Compositions, **A.S. ARTEMOV**, Russian Academy of Sciences, Russia, V.M. BOGATYREV, National Academy of Sciences of Ukraine, S.B. FARAFONOV, State Technological University, Russia, I.G. RUZAVIN, Lomonosov Moscow State Academy of Fine Chemical Technology, Russia	**E2-3-11** Influence of the Nitriding and TiAlN Coating Thickness on the Mechanical and Tribological Properties of a Duplex Coating System on H13 Die Steel, **R. TORRES**, PUCPR, Brazil, L. SUZUKI, Neodent Co, Brazil, P. SOARES, Pontificia Universidade Católica do Paraná, Brazil, C. LEPIENSKI, Universidade Federal do Paraná, Brazil, J.J. MOORE, Colorado School of Mines
11:40 am	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm	**E2-3-12** Deformation Mechanisms in TiN/NbN Multilayer Thin Films, **K.A. RZEPIEJEWSKA-MALYSKA**, Empa, Switzerland, A. BARANSKA, A. SZERLING, Institute of Electron Technology, Poland, S. KORTE, W.J. CLEGG, University of Cambridge, United Kingdom, J. MICHLER, Empa, Switzerland

Tuesday Morning, April 28, 2009

	Applications, Manufacturing, and Equipment **Room: Sunrise - Session G6** **Coatings, Pre-Treatment, Post-Treatment and Duplex Technology** **Moderators:** K. Bobzin, RWTH Aachen University, E. Kusano, Kanazawa Institute of Technology	**New Horizons in Coatings and Thin Films** **Room: Tiki Pavilion - Session H1-1** **Nanotube, Nanowire and Nanoparticle Thin-Films and Coatings** **Moderator:** R. Martel, Université de Montréal
8:00 am	**G6-1** Exploring the Potential of Triode Plasma Diffusion Treatments to Improve the Wear Behaviour of Titanium Alloys, **G. CASSAR**, A. LEYLAND, A. MATTHEWS, The University of Sheffield, United Kingdom	**H1-1-1 Invited** Chiral Architectures in Thin Films: Fabrication and Applications, **M.J. BRETT**, University of Alberta, Canada
8:20 am	**G6-2** RF-Plasma Technique for Duplex Coatings on Aluminum, **S. MEIER**, M. KÖNIG, C. HORMANN, Fraunhofer Institute for Mechanics of Materials IWM, Germany	Invited talk continued.
8:40 am	**G6-3** Plasma Nitrocarburizing and Laser Hardening Duplex Treatment of AISI 4340 Steel, **V.H. BAGGIO-SCHEID**, G.DE VASCONCEDOS, A.J. ABDALLA, General-Command of Aerospace Technology, Brazil	**H1-1-3** Shape Modification of Silica Submicroparticles Assembled in Compact Monolayer by Dry Etching, **S. PORTAL**, M.A. VALLVÉ, M. RUBIO-ROY, C. CORBELLA, J. IGNES-MULLOL, B. BERTRAN, University of Barcelona, Spain
9:00 am	**G6-4 Invited** Dedicated Coatings for High Precision Cutting Tools – Coating Design and Architecture Adjusted with Most Modern Process Technology, **T. LEYENDECKER**, R. CREMER, CemeCon AG, Germany	**H1-1-4** Self-Organized Functional Metallic Nano-Arrays by Combined Ion Beam Sputtering and Plasma Deposition, **T.W.H. OATES**, J. ROSEN, Linköping University, Sweden, A. KELLER, S. FACSKO, Forschungszentrum Dresden-Rossendorf, Germany, S. NODA, University of Tokyo, Japan
9:20 am	Invited talk continued.	**H1-1-6** Nano Multilayer Transparent SiO_x-Based PECVD Coatings for Barrier and Protective Applications, **A. PATELLI**, S. VEZZU', S. COSTACURTA, L. ZOTTAREL, M. COLASUONNO, Associazione CIVEN, Italy
9:40 am	**G6-6** Edge Honing for High Performance Drilling Operations, **M. MAES**, KOMET GROUP GmbH, Germany	**H1-1-7** Patterning of Semiconductor Surfaces with Ordered Metallic Nanowires and Nanoparticles via Self-Assembly, **J.M. BURIAK**, J. CHAI, University of Alberta, Canada
10:00 am	**G6-7 Invited** Process Temperature and Post Annealing Effects on Microstructure and Mechanical Behavior of the Sputtering Ni-P-Al Coatings, Y.M. SU, J.C. WU, **F.B. WU**, National United University, Taiwan	**H1-1-9 Invited** Carbon Nanotube Films as Transparent, Conductive and Flexible Electrodes, **C. ZHOU**, University of Southern California
10:20 am	Invited talk continued.	Invited talk continued.
10:40 am	**G6-9** Influence of Different Post-Treatment Techniques on Arc Coated Carbide Drills, **S. SATTEL**, I. GARRN, Guehring oHG, Germany	**H1-1-12** Structural Characteristics and Photocatalytic Capabilities of Visible Light Responsible Nanoporous TiO_2/ITO Composite Thin Films, **C.H. HUNG**, National Kaohsiung First University of Science and Technology, Taiwan, K.R. WU, National Kaohsiung Marine University, Taiwan, W.C. LO, National Kaohsiung First University of Science and Technology, Taiwan, T.P. CHO, Metal Industries Research and Development Centre, Taiwan
11:00 am		
	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm

	Bioactive Coatings and Biofunctionalization **Room: Sunset - Session TS3-2** **Bioactive Coatings and Surface Biofunctionalization** **Moderators:** D.V. Shtansky, State Technological University, Moscow Institute of Steel and Alloys, S. Kumar, University of South Australia	
8:00 am	**TS3-2-1** Surface Functionalization of High Strength Biomedical Ceramics, **J.R. PIASCIK**, RTI International, J.Y. THOMPSON, NOVA Southeastern University, E.J. SWIFT, University of North Carolina at Chapel Hill, S. GREGO, B.R. STONER, RTI International	
8:20 am	**TS3-2-2** Osteoblast Adhesion to Metal-Oxide Incorporated Diamond-Like Carbon Films, **L.K. RANDENIYA**, A. BENDAVID, S. AMIN, P.J. MARTIN, CSIRO Materials Science and Engineering, Australia, R. ROHANIZADEH, University of Sydney, Australia	
8:40 am	**TS3-2-3** Tribology and Corrosion Behavior of Alpha Alumina-coated Ti-6Al-4V for Surgical Implantation, **M.T. MONTGOMERY**, University of Arkansas, P. MOHANTY, University of Michigan, H.H. ABU-SAFE, Lebanese American University, Lebanon, M.H. GORDON, University of Arkansas	
9:00 am	**TS3-2-4** Fabrication of Hierarchical Wrinkled Micro-Pillars using Diamond-Like Carbon Coating, **Y. RAHMAWAN**, Seoul National University, Korea, SK.F. AHMED, Korea Institute of Science and Technology, Korea, K.J. JANG, Seoul National University, Korea, K.S. KIM, Brown University, M.W. MOON, K.R. LEE, Korea Institute of Science and Technology, Korea, K.Y. SUH, Seoul National University, Korea	
9:20 am	**TS3-2-5** Anti-Microbial Properties of Silver Modified Amorphous Carbon Films, **A ALMAGUER-FLORES**, Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico, R OLIVARES-NAVARRETE, Department of Biomedical Engineering, Georgia Institute of Technology, L.A. XIMÉNEZ-FYVIE, O. GARCIA, S.E. RODIL, Universidad Nacional Autónoma de México	
9:40 am	**TS3-2-6** Rapid Antimicrobial Susceptibility Testing of Escherichia Coli and Staphylococcus Aureus by Using the Cr-Cu-N Nanocomposite Thin films with Gradient Copper Content, Y.-J. CHANG, K.-J. WENG, J.-S. LU, J.-T. CHEN, J.-L. LI, **J.-W. LEE**, Tungnan University, Taiwan	
10:00 am	**TS3-2-7** Electrochemical Characterization of Biocompatible TiO_x and NbO_x Films, **P.N. ROJAS**, S.E. RODIL, H. ARZATE, S. MUHL, Universidad Nacional Autónoma de México	
10:20 am	**TS3-2-8** Structural Characterisation of Multifunctional Biocompatible Ti(CON) Based Coatings, S. RIBEIRO, N. JORDÃO, Universidade do Minho, Portugal, A.P. PIEDADE, Grupo de Materials, Portugal, M. HENRIQUES, R. OLIVEIRA, Universidade do Minho, Portugal, R. ESCOBAR-GALINDO, Instituto de Ciencia de Materiales de Madrid (ICMM -CSIC), Spain, **S. CARVALHO**, Universidade do Minho, Portugal	
10:40 am	**TS3-2-9** Preparation of Plasma-Polymerized Para-xylene as an Alternative to Parylene Coating for Biomedical Surface Modification, **C.-M. CHOU**, Feng Chia University; Taichung Veterans General Hospital, Taiwan, K.-C. HSIEH, Feng Chia University, Taiwan, R.O.C., C.-J. CHUNG, Central Taiwan University of Science and Technology, Taiwan, J.-L. HE, Feng Chia University, Taiwan	
11:00 am	**TS3-2-10** Micro-arc Oxidation of β Titanium Alloy: Structural Characterization and Osteoblast Compatibility, **H.-T. CHEN**, Feng Chia University; China Medical University Hospital, Taiwan, C.-H. HSIAO, H.-Y. LONG, Feng Chia University, Taiwan, C.-J. CHUNG, Central Taiwan University of Science and Technology, R.O.C., C.-H. TANG, China Medical University, Taiwan, K.-C. CHEN, J.-L. HE, Feng Chia University, Taiwan	
11:20 am	**TS3-2-11** Nanoindentation and Corrosion Studies of TiN/NiTi Thin Films for Biomedical Applications, **D. KAUR**, A. KUMAR, Indian Institute of Technology Roorkee, India	
	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm	Exhibition Opens Today Town & Country/San Diego Rooms 11:00 am – 7:00 pm

Tuesday Afternoon, April 28, 2009

	Coatings for Use at High Temperature **Room: Royal Palm 1-3 - Session A1-2** **Coatings to Resist High Temperature Oxidation** **Moderators:** Y. Zhang, Tennessee Technological University, K. Stiller, Chalmers University of Technology	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B5-2** **Properties and Characterization of Hard Coatings and Surfaces - Moderators:** Y.W. Chung, Northwestern University, G. Abadias, Universite de Poitiers-CNRS, M. Fenker, Forschungsinstitut Edelmetalle & Metallchemie
1:30 pm	**A1-2-1 Invited** Compositional, Geometrical and Manufacturing Related Parameters Affecting the Oxidation Resistance of MCrAlY-Coatings, **D. NAUMENKO**, J. TOSCANO, M. SUBANOVIC, L. SINGHEISER, W.J. QUADAKKERS, Forschungszentrum Jülich GmbH, Germany	**B5-2-1** Structure, Stability and Bonding of Ternary Transition Metal Nitrides, **P. PATSALAS**, G.M. MATENOGLOU, L.E. KOUTSOKERAS, CH.E. LEKKA, University of Ioannina, Greece, G. ABADIAS, Universite de Poitiers-CNRS, France, C. KOSMIDIS, G.A. EVANGELAKIS, University of Ioannina, Greece
1:50 pm	Invited talk continued.	**B5-2-2** Ab-Initio Calculations of the Effect of Si and C on Elastic Properties of Metal Nitrides, **J. HOUSKA**, J.E. KLEMBERG-SAPIEHA, L. MARTINU, Ecole Polytechnique de Montreal, Canada
2:10 pm	**A1-2-3** Microstructure and Oxidation Resistance of Nanocrystalline 304SS-Al Coatings, **N.S. CHERUVU**, R. WEI, Southwest Research Institute, R. GOVINDARAJU, Karta Technologies, D.W. GANDY, Electric Power Research Institute	**B5-2-3 Invited** Growth Behavior and Microstructure of Arc Ion Plated Titanium Dioxide, C.-J. CHUNG, Central Taiwan University of Science and Technology, Taiwan, H.-I. LIN, P-Y. HSIEH, K.-C. CHEN, **J.-L. HE**, Feng Chia University, Taiwan, A. LEYLAND, A. MATTHEWS, University of Sheffield, United Kingdom
2:30 pm	**A1-2-5** Oxidation Resistance of a Zr-Doped NiAl Bondcoat Thermochemically Deposited on a Nickel-Based Superalloy, **S. HAMADI**, M.P. BACOS, M. POULAIN, ONERA, France, V. MAURICE, P. MARCUS, ENSCP/CNRS, France	Invited talk continued.
2:50 pm	**A1-2-6** Reactive Element-Modified Aluminide and Platinum Aluminide Coatings on Ni-Base Superalloys, **M.S. PRIEST**, Y. ZHANG, Tennessee Technological University, B.A. PINT, J.A. HAYNES, Oak Ridge National Laboratory, B.T. HAZEL, B.A. NAGARAJ, GE Aircraft Engines	**B5-2-5** Structure-Property Relations of arc-Evaporated $Al_xNb_{1-x}N$ Hard Coatings, **R. FRANZ**, University of Leoben, Austria, M. LECHTHALER, OC oerlikon Balzers AG, Liechtenstein, C. POLZER, PLANSEE Composite Materials GmbH, Austria, C. MITTERER, University of Leoben, Austria
3:10 pm	**A1-2-7** NiW Diffusion Barrier Influence on the Oxidation Behaviour of β-(Ni,Pt)Al Coated Fourth Generation Ni-Base Superalloy, **E. CAVALETTI**, S. NAVEOS, S. MERCIER, P. JOSSO, M.P. BACOS, ONERA, France, D. MONCEAU, ENSIACET-INPT, France	**B5-2-13** Wear Resistant Coatings Via Directed Vapor Deposition, **S. EUSTIS**, D. HASS, Directed Vapor Technologies International, Inc.
3:30 pm	**A1-2-8** Formation and Oxidation Performance of Low-Temperature Pack Aluminide Coatings on Ferritic-Martensitic Steels, **Y. ZHANG**, B. BATES, Tennessee Technological University, B.A. PINT, Oak Ridge National Laboratory	**B5-2-8** Development of B4C Sputter-Deposited Coatings for Mechanical Applications, C. PATACAS, **C. LOURO**, J.C. OLIVEIRA, A. CAVALEIRO, University of Coimbra, Portugal
3:50 pm	**A1-2-9 Invited** Pt Modified Nickel Aluminides, MCrAlY-Base Multilayer Coatings and TBC Systems Fabricated by Sparks Plasma Sintering (SPS) for the Protection of Nickel Base Superalloys, **D. MONCEAU**, ENSIACET-INPT, France, D. OQUAB, CIRIMAT Laboratory - Toulouse, France, C. ESTOURNES, CIRIMAT and PNF2	**B5-2-9 Invited** Influence of the Chemical Composition, Electronic Structure and Phase Evolution on the Electric and Optical Behaviors of Decorative Zirconium Oxynitride Thin Films, **F. VAZ**, P. CARVALHO, L. CUNHA, Minho University, Portugal, N. MARTIN, Institut FEMTO-ST, France, J.P. ESPINÓS, A.R. GONZÁLEZ-ELIPE, Instituto
4:10 pm	Invited talk continued.	Invited talk continued.
4:30 pm	**A1-2-11** Role of Al Oxide PVD Coatings in the Protection Against Metal Dusting, **J. ALVAREZ**, ITESM-CCV, B. BAUTISTA, D. MELO, IPN, Mexico, O. SALAS, ITESM-CEM, Mexico, R. REICHELT, Wilhelms-Universitaet, Germany, J. OSEGUERA, ITESM-CEM, Mexico, . LÓPEZ, IPN, Mexico	**B5-2-11** Sub-Microstructure and Surface Topography of Reactive Unbalanced Magnetron Sputtered Titanium and Titanium Compound Thin Films, **S.D. CARPENTER**, P. KELLY, Manchester Metropolitan University, United Kingdom
4:50 pm	**A1-2-12** Observation of High-Temperature Phase Transformation in the Si-Modified Aluminide Coating on Mild Steel Using EBSD, **W.-J. CHENG**, C.-J. WANG, National Taiwan University of Science and Technology, Taiwan	**B5-2-12** Effect of Substrate Positioning for TiAlN Films Deposited by an Inverted Cylindrical Magnetron Sputtering System, **H.H. ABU-SAFE**, Lebanese American University, Lebanon, M.H. GORDON, University of Arkansas
5:10 pm		**B5-2-7** Carbon Occupancy of Interstitial Sites in Vanadium Carbide Films Deposited by Reactive Magnetron Sputtering, E. PORTOLAN, C.L.G. AMORIM, G.V. SOARES, C. AGUZZOLI, C.A. PEROTTONI, I.J.R. BAUMVOL, **C.A. FIGUEROA**, Universidade de Caxias do Sul, Brazil
	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm

Tuesday Afternoon, April 28, 2009

	Carbon and Nitride Materials: Synthesis-Structure-Property Relationships - Room: Royal Palm 4-6 - Session D1 **Boron Nitride and Carbon Nitride and Group-III (Al, Ga, In) Nitride Materials** **Moderators:** A.B. Djurišić, The University of Hong Kong, M. Keunecke, Fraunhofer Institute for Surface Engineering & Thin Films	**Tribology and Mechanical Behavior of Coatings and Thin Films - Room: California - Session E5** **Nano- and Microtribology Instrumentation and Characterization** **Moderators:** N. Randall, CSM Instruments Inc., J. Hahn, Korea Research Institute of Standards and Science
1:30 pm	**D1-1 Invited** Cubic Boron Nitride Based Metastable Coatings and Nanocomposites, **J. YE**, S. ULRICH, M. STÜBER, C. ZIEBERT, Forschungszentrum Karlsruhe, Germany	**E5-1 Invited** Nanotribology, Nanomechanics and Materials Characterization Studies and Applications to Bio/Mamotechnology and Biomimetics, **B. BHUSHAN**, The Ohio State University
1:50 pm	Invited talk continued.	Invited talk continued.
2:10 pm	**D1-3** Machining of High Performance Workpiece Materials with cBN Coated Cutting Tools, E. UHLMANN, **J.A. OYANEDEL FUENTES**, Institute for Machine Tools and Factory Management, Germany, M. KEUNECKE, Fraunhofer Institute for Surface Engineering and Thin Films, Germany	**E5-3** Nanotribology of WSC and MoSeC Self-Lubricant Coatings, **T. POLCAR**, SEG-CEMUC University of Coimbra & Czech Technical University - Prague, Czech Republic, M. EVARISTO, University of Coimbra, Portugal, R. COLAÇO, Instituto Superior Tecnico, Portugal, A. CAVALEIRO, University of Coimbra, Portugal
2:30 pm	**D1-4** Magnetron Sputter Deposition of Low-Stress Cubic Boron Nitride Films using Ar-N_2-CH_4 Gas Mixtures, **S. ULRICH**, J. YE, M. STÜBER, C. ZIEBERT, Forschungszentrum Karlsruhe, Germany	**E5-4** Micro-Tribological Performance of Metal/ MoS_2 Lubricants, **P. STOYANOV**, R. CHROMIK, McGill University, Canada, J.R. LINCE, The Aerospace Corporation
2:50 pm	**D1-5** Texture Development and Microstructure Characterization of AlN Thin Films Fabricated by Pulsed Closed Field Unbalanced Magnetron Sputtering, F. WANG, A. FELDMAN, J. LIN, J.J. MOORE, B. MISHRA, **M. HASHEMINIASARI**, Colorado School of Mines	**E5-5 Invited** Application of Diamond-Like Nanocomposite Coatings for Microsystems Tribology[1], **S. PRASAD**, Sandia National Laboratories, T. SCHARF, The University of North Texas
3:10 pm	**D1-6** Theoretical and Spectroscopic Investigations on the Structure and Bonding in B-C-N Thin Films, **E. BENGU**, M.F. GENISEL, O. GULSEREN, R. OVALI, Bilkent University, Turkey	Invited talk continued.
3:30 pm	**D1-7 Invited** Atomic and Electronic Structure of GaN Surfaces, **J.E. NORTHRUP**, Palo Alto Research Center	**E5-7** Nanotribological Properties of Topographically-Chemically Modified Silicon Surfaces, D.C. PHAM, R.A. SINGH, K. NA, S. YANG, **E.-S. YOON**, Korea Institure of Science and Technology, Korea
3:50 pm	Invited talk continued.	**E5-8** Nano-Tribological Properties of Undulated Ultra Thin Diamond Patterns, D.-S. LIM, **S.K. LEE**, J.H. KIM, Korea University, Korea
4:10 pm	**D1-9** Highly Reflective and Thermally Stable Ni/Ag Multi-Layer Ohmic Contact to p-Type GaN, **J.H. SON**, Y.H. SONG, J.-L. LEE, Pohang University of Science and Technology (POSTECH), Korea	**E5-9** Nanotribology of Multilayered Nano Thin Films by Molecular Dynamics Simulations, **J.-C. HUANG**, C.-C. CHO, Tungnan University, Taiwan
4:30 pm	**D1-10** Influence of rf Power on the Electrical and Mechanical Properties of CN Thin Films Deposited by Reactive RF Magnetron Sputtering, **I. BANERJEE**, N. KUMARI, M. KUMAR, S.K. MAHAPATRA, Birla Institute of Technology, India, S.K. PABI, Indian Institute of Technology Kharagpur, India, P.K. BARHAI, Birla Institute of Technology,	
	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm

Tuesday Afternoon, April 28, 2009

	Characterization: Linking Synthesis Properties and Microstructure **Room: Sunrise - Session F1-1** **Advances in Characterization of Coatings and Thin Films** **Moderators:** P. Schaaf, TU Ilmenau, M. Baker, University of Surrey	**New Horizons in Coatings and Thin Films** **Room: Tiki Pavilion - Session H1-2** **Nanotube, Nanowire and Nanoparticle Thin-Films and Coatings** **Moderator:** R. Martel, Université de Montréal
1:30 pm	**F1-1-2** Effect of Stoichiometry on Structure and Texture of $Zr_{1-x}Si_xN$ Thin Films, X. ZHANG, M.S. BYRNE, **R.J. LAD**, University of Maine	**H1-2-1 Invited** Temperature Dependent Conduction in ZnO Nanowires, **J. LU**, University of Southern California
1:50 pm	**F1-1-4 Invited** Atom Probe Tomography of Thin Films, **D. LARSON**, Imago Scientific Instruments	Invited talk continued.
2:10 pm	Invited talk continued.	**H1-2-3** Growth and Characterizations of ZnO Nanorod/Film Structures, **W.Y. WU**, M.D. CHEN, J.M. TING, National Cheng Kung University, Taiwan
2:30 pm	**F1-1-6** Three-Dimensional Atom Probe Tomography of Nanocomposite Diamond-like Carbon Films, **T.W. SCHARF**, M.C. ROMANES, R. BANERJEE, The University of North Texas, R.D. EVANS, G.L. DOLL, Timken Company	**H1-2-4** Sputter Deposited ZnO Nanowire/Thin Film Structures, T.L. CHOU, W.Y. WU, **J.M. TING**, National Cheng Kung University, Taiwan
2:50 pm	**F1-1-7** Tip Enhanced Raman Spectroscopy for High Resolution Assessment of Strained Silicon Devices, **L. SANDERSON**, P. DOBROSZ, S.H. OLSEN, S.J. BULL, Newcastle University, United Kingdom, S. MANTL, D.M. BUCA, FZ-Juelich, Germany	**H1-2-5** Formation of Titanium Germanosilicide Nanocrystals for Nonvolatile Memory Application, **L.-W. FENG**, National Chiao Tung University, Taiwan, T.-C. CHANG, National Sun Yat-Sen University, C.-H. TU, P.-S. WANG, National Chiao Tung University, D.-S. GAN, N.-J. HO, H.-J. HUANG, M.-C. CHEN, National Sun Yat-Sen University, C.-Y. CHANG, National Chiao Tung University
3:10 pm	**F1-1-8** Depth Profile Analysis of Thin Layers on Glasses and Ceramics by RF GD OES, **P. HUNAULT**, Horiba Jobin Yvon Inc., P. CHAPON, C. TAUZIEDE, A. TEMPEZ, Horiba Jobin Yvon Inc., France, M. GANCIU, NILPRP - Bucharest, Romania, P. GUILLOT, University JF Champollion - Albi, France, P. BELENGUER, Laplace CNRS, France	**H1-2-6** Fabrication of Molybdenum Oxide Nanowires Grown by Thermal CVD, H.C. HSIH, Chinese Culture University, & National Tsing Hua University, Taiwan, **C.C. CHANG**, National Tsing Hua University, Taiwan, M.W. HUANG, National Chung Hsing University, Taiwan, Y.T. HSIEH, National Tsing Hua University, Taiwan, B.J. WEI, National Chung Hsing University, Taiwan
3:30 pm	**F1-1-9** Local-Order Information of the Decomposition of Nanocrystalline Supersaturated TiAlN by X-ray Absorption Spectroscopy, **J. ENDRINO**, R. GAGO, Instituto de Ciencia de Materiales de Madrid, Spain, G. FOX-RABINOVICH, McMaster University, Canada, A. GUTIERREZ, Universidad Autonoma de Madrid, Spain	**H1-2-7** Thermally Evaporated Sb-Doped SnO_2 Nanobelts for Ethanol Gas Sensors, **P.-S. LEE**, Y.-H. LIN, C.-C. KUO, National Tsing Hua University, Taiwan, J.-M. WU, Feng-Cha University, Taiwan, Y.-S. CHANG, H.C. SHIH, National Tsing Hua University, Taiwan
3:50 pm	**F1-1-10** Testing Thin Films by Microcompression: Benefits and Limits, D. KIENER, H.P. WOERGOETTER, Erich Schmid Institute of Materials Science, Austria, **G. DEHM**, University of Leoben, Austria	**H1-2-9** Hybrid Manganese Oxide Films for Supercapacitor Application Prepared by Sol-Gel Technique, C.-K. LIN, C.-H. CHENG, Y.-H. TIEN, **C.-Y. CHEN**, Feng Chia University, Taiwan, S.-C. WANG, Southern Taiwan University, Taiwan, W.T. TSAI, National Cheng Kung University, Taiwan
4:10 pm	**F1-1-11** Contact Angle Analysis of Sol-Gel Derived Zirconia Based Hybrid Coatings on 304 Stainless Steel Substrates, **S. ASHOKKUMAR**, J. HINKE, Accoat A/S, Denmark, J.A. NISSEN, P. MOLLER, Technical University of Denmark	
	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm

Tuesday Afternoon, April 28, 2009

Atomistics of Thin Films Growth
Room: Sunset - Session TS1
Computational & Experimental Studies of Thin Film Growth: An Atomistic View
Moderators: S. Kodambaka, University of California - Los Angeles, J. Rosén, Linköping University

1:30 pm	**TS1-1 Invited** In Situ, Real-Time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Corssoin Barrier, and Steering, **M.J. ROST**, Leiden University, Netherlands	
1:50 pm	Invited talk continued.	
2:10 pm	**TS1-3 Invited** Evolution of Residual Stress During Thin Film Growth: Effect of Competing Kinetic Processes, **E. CHASON**, Brown University	
2:30 pm	Invited talk continued.	
2:50 pm	**TS1-6** Atomistic Simulations of Plasma Sprayed Thin Film Growth, **E. WEBB**, Sandia National Laboratories	
3:10 pm	**TS1-7 Invited** Tunable Molecular Beams: A New Frontier in Vacuum Deposition of Organic Semiconductors, **A. AMASSIAN**, Cornell University, Ithaca	
3:30 pm	Invited talk continued.	
3:50 pm	**TS1-9** A First-Principles Investigation of the Phase Stability of Ti_2AlC Upon Oxygen Incorporation, **M. DAHLQVIST**, B. ALLING, I.A. ABRIKOSOV, J. ROSÉN, Linköping University, Sweden	
4:10 pm	**TS1-10** A Formula for Increased Hardness and/or Ductility in TiN-Based Thin Films, D.G. SANGIOVANNI, **V. CHIRITA**, L. HULTMAN, Linköping University, Sweden	
4:30 pm	**TS1-11 Invited** DFT Studies of Graphene Thin Films, **C.V. CIOBANU**, Colorado School of Mines	
4:50 pm	Invited talk continued.	
	Exhibition Reception Town & Country/San Diego Rooms 5:30 – 7:00 pm	

Wednesday Morning, April 29, 2009

	Hard Coatings and Vapor Deposition Technology **Room: Golden West - Session B1-1** **Sputtering Coatings and Technologies** **Moderators:** C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B4** **Laser and Ion Beam Surface Engineering** **Moderators:** C. Muratore, Air Force Research Laboratory/UTC, Inc., W. Waldhouser, Johanneum Research
8:00 am	Session B4	**B4-2** Excimer and Femtosecond Laser Microstructuring of Superhard Pulsed Laser Deposited ta-C Films - Effects on Friction and Wear Resistance, **S. WEISSMANTEL**, G. REISSE, M. NIEHER, R. BOETTCHER, A. ENGEL, K. GUENTHER, University of Applied Sciences Mittweida, Germany
8:20 am		**B4-3** Control of Femtosecond Pulsed Laser Deposited Thin Films through Plasma Optimization by Temporal Shaping Based on Genetical Algorithm, M. GUILLERMIN, C. LIEBIG, **F. GARRELIE**, R. STOIAN, A.S. LOIR, Université Jean Monnet, France, S. VALETTE, Ecole Centrale de Lyon, France, E. AUDOUARD, Université Jean Monnet, France
8:40 am		**B4-4** Deposition of Nanocomposite DNA Thin Films by Through Thin Film Ablation, **S. FAIRCHILD**, Air Force Research Laboratory, P. MURRAY, University of Dayton Research Institute, O. SHENDEROVA, International Technology Center, F. OUCHEN, University of Dayton Research Institute
9:00 am		**B4-5 Invited** Broad-Area Ion Beam Systems for Materials Processing: An Overview of Technology and Applications, **L.J. MAHONEY**, D.E. SIEGFRIED, B.A. KNOLLENBERG, V. KANAROV, A.J. DEVASAHAYAM, Veeco Instruments, Inc.
9:20 am		Invited talk continued.
9:40 am		**B4-7 Invited** Ion Beam Irradiation Effects in Polymers Confined at Nanometer Scale and Nanomaterials, **M. CHIPARA**, K. LOZANON, M.D. CHIPARA, A. ADHIKARI, M. MIHUT, The University of Texas Pan American
10:00 am		Invited talk continued.
10:20 am	**B1-1-9** Composition-Property Relations of Reactive Magnetron Sputtered VC_xN_{1-x} Coatings, **N. FATEH**, C. MITTERER, University of Leoben, Austria	Session B1-1
10:40 am	**B1-1-10** Experimental and Computational Study on the Phase Stability of Cubic Al Containing Transition Metal Nitrides, **F. ROVERE**, M. TO BABEN, S. ERSHOV, RWTH Aachen University, Germany, H.-G. FUSS, CemeCon AG, Germany, P.H. MAYRHOFER, Montanuniversität Leoben, Austria, D. MUSIC, J.M. SCHNEIDER, RWTH Aachen University, Germany ****STUDENT FINALIST****	
11:00 am	**B1-1-11 Invited** Combinatorial Sputter Deposition for Thin Film Materials Selection and -Design, **J.M. SCHNEIDER**, RWTH Aachen University	
11:20 am	Invited talk continued.	
	Exhibition Closes Today 2:00 pm	Exhibition Closes Today 2:00 pm

Wednesday Morning, April 29, 2009

	Optical Thin Films **Room: Royal Palm 4-6 - Session C2-1** **Optical Thin Films for Active Devices and Microstystems** **Moderators:** M. Cremona, PUC-Rio, T. Miyata, Kanazawa Institute of Technology	**Tribology and Mechanical Behavior of Coatings and Thin Films - Room: California - Session E1-1** **Friction and Wear of Coatings: Lubrication, Surface Effects and Modeling - Moderators:** E. Broitman, Carnegie Mellon University, A. Fernandez-Camacho, CSIC-University Sevilla, O.L. Eryilmaz, Argonne National Laboratory
8:00 am	**C2-1-1 Invited** Conjugated Polymer Based Organic Solar Cells: State of the Art and Future Challenges, G. DENNIER, C.J. BRABEC, **N. DROLET**, Konarka Technologies Inc.	**E1-1-1** Low-Friction and Wear Mechanisms for Sputtered Ti-C-N Coatings, **M. REBELO DE FIGUEIREDO**, G.A. FONTALVO, Univ of Leoben, Austria, C. MURATORE, AFRL/UTC, Inc., R. FRANZ, Univ of Leoben, , A.A. VOEVODIN, AFRL/Univ of Dayton, M. O'SULLIVAN, Plansee Composite Matls GmbH, , M. LECHTHALER, OC Oerlikon Balzers AG, C. MITTERER, Univ of Leoben,
8:20 am	Invited talk continued.	**E1-1-10** Role of Transfer Phenomena on Tribological Behaviour of of Al-Cu Thin Films, M. RUET, Ecole Centrale de Lyon, France, T. DUGUET, Ecole des Mines de Nancy, France, **J. FONTAINE**, Ecole Centrale de Lyon, France, V. FOURNEE, Ecole des Mines de Nancy, France, K. ITO, Ecole Centrale de Lyon, France, J. LEDIEU, Ecole des Mines de Nancy, France, M. BELIN, Ecole Centrale de Lyon, France
8:40 am	**C2-1-3** Electrochromic Performance of PECVD-Synthesized WO_xC_y Thin Films on Flexible PET/ITO Substrates for Flexible Electrochromic Devices, **Y.-S. LIN**, Feng Chia University, Taiwan	**E1-1-3 Invited** Tribology of the Head/Disk Interface, **F.E. TALKE**, Univ. of California, San Diego
9:00 am	**C2-1-5 Invited** Large, Room-Temperature Magnetoresistance in Organic Light-Emitting Diodes, **M. WOHLGENANNT**, T.D. NGUYEN, J. RYBICKI, Y. SHENG, University of Iowa	Invited talk continued.
9:20 am	Invited talk continued.	**E1-1-5** Comparative Study of the Friction Coefficient and Wear Volume with Silane and Carbonitrided Interlayer in 316L Stainless Steel Samples in Hybrid Lubrication Conditions, **R.P.C.C. STATUTI**, INPE – Inst Nacional de Pesquisas Espaciais, Brazil, P.A. RADI, L.V SANTOS, V.J. TRAVA-AIROLDI, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil
9:40 am	**C2-1-7** Improved Multi-layer OLED Architecture Using Evolutionary Genetic Algorithm, **M. CREMONA**, PUC-Rio, Brazil, W.Q. GIANINI, C. LEGNANI, K.C. TEIXEIRA, CeDO - Inmetro, Brazil, B. MESSER, O.P. VILELA NETO, M.A.C. PACHECO, ICA - DEE - PUC-Rio, Brazil	**E1-1-6** How Can H Determine the Tribological Behavior in Lubricated Contact of Cu/W – C:H Sputtered Coatings, **M. EVARISTO**, SEG-CEMUC, University of Coimbra, Portugal, T. POLCAR, SEG-CEMUC University of Coimbra & Czech Technical University - Prague, Portugal, A. CAVALEIRO, SEG-CEMUC University of Coimbra, Portugal
10:00 am	**C2-1-8** The Effects of Post Annealing on Electrochromic Nb-Doped WO_3 Films, J.L HUANG, **C.K. WANG**, National Cheng Kung University, Taiwan, S.-C. WANG, Southern Taiwan University, Taiwan, D.R. SAHU, National Cheng Kung University, Taiwan	**E1-1-7 Invited** In Orbit Tribological Tests of a Novel Solid Lubricating Film at the International Space Station, **M. BRIZUELA**, A. GARCIA-LUIS, INASMET-Tecnalia, Spain, J.I. OÑATE, Fundacion INASMET-Tecnalia, Spain, I. GARMENDIA, INASMET-Tecnalia, Spain, C. MARTINEZ, R. FERNANDEZ-SANZ, INTA, Spain
10:20 am	**C2-1-9** Suppressing Coherent Thermal Transport of Photons by Stacking Multi-layer Photonic Crystals, **W. T. LAU**, J.-T. SHEN, S. FAN, Stanford University	Invited talk continued.
10:40 am	**C2-1-10** Effect of Power Density on ITO Thin Films by Facing Targets Sputtering, **Y.J. KIM**, S.B. JIN, S.I. KIM, Y.S. CHOI, I.S. CHOI, J.G. HAN, Center for Advanced Plasma Surface Technology, Korea	**E1-1-9** Low Earth Orbit Space Tribometer, **B. KRICK**, University of Florida, J.G. JONES, Air Force Research Laboratory/RXBT, J.K. LENOFF, Air Force Research Laboratory/RXBN, A.A. VOEVODIN, Air Force Research Lab/University of Dayton, W.G. SAWYER, University of Florida
	Exhibition Closes Today 2:00 pm	Focused Topic Session CSM Instruments 12:15 – 1:15 pm

Wednesday Morning, April 29, 2009

	Characterization: Linking Synthesis Properties and Microstructure **Room: Sunrise - Session F1-2** **Advances in Characterization of Coatings and Thin Films** **Moderators:** P. Schaaf, TU Ilmenau, M. Baker, University of Surrey	**Characterization: Linking Synthesis Properties and Microstructure** **Room: Sunrise - Session F3** **Characterisation by Electron and Ion Beam Microscopy** **Moderators:** M. Watanabe, Lawrence Berkeley National Laboratory, J.-G. Wen, University of Illinois
8:00 am	**F1-2-1 Invited** Thin Film Studies by the Application of Complementary Techniques in Real Time, **A. VANTOMME**, D. SMEETS, J. DEMEULEMEESTER, Katholieke University Leuven, Belgium, C. DETAVERNIER, Universiteit Gent, Belgium, C.M. COMRIE, University of Cape Town, South Africa, C.C. THERON, iThemba Labs, South Africa, C. LAVOIE, T.J.	Session F1-2
8:20 am	Invited talk continued.	
8:40 am	**F1-2-4** Cathodoluminescent Properties and Phase Identification in Kappa- and Gamma-Alumina Coatings, **G. POZINA**, F. GIULIANI, L. HULTMAN, Linköping University, Sweden, H. BLOMQVIST, M. COLLIN, I. REINECK, Sandvik Tooling AB, Sweden	
9:00 am	Session F3	**F3-5 Invited** Characterization of Interfaces Using Scanning Transmission Electron Microscopy Techniques, **S. STEMMER**, J.M. LEBEAU, University of California, Santa Barbara
9:20 am		Invited talk continued.
9:40 am		**F3-7** Thermal Stability of PVD Nitride Multilayer Coatings: Effect of Nitrogen Inter-Diffusion, I. ROSS, **W. RAINFORTH**, University of Sheffield, United Kingdom, P.EH. HOVSEPIAN, Sheffield Hallam University, United Kingdom
10:00 am		**F3-8** Raman Multi Wavelength and Energy Filtred Tem Study of Diamond-Like Carbon Films Deposited by Femtosecond and Nanosecond Pulsed Laser Ablation, **A. SIKORA**, Université Jean Monnet, France, B. VACHER, Ecole Centrale de Lyon, France, A.S. LOIR, F. GARRELIE, C. DONNET, Université Jean Monnet, France
10:20 am		**F3-9 Invited** The Stability of Thin Metal Films in Contact with Oxides, **W. KAPLAN**, M. BARAM, Technion, Israel
10:40 am		Invited talk continued.
11:00 am		**F3-11** Characteristic Analysis of Supporting Precious Metal Nanoparticles on Carbon Nanotubes for Electrocatalysts, M. CHEN, **S.-C. LU**, H.-Y. CHEN, Minghsin University of Science and Technology, Taiwan, H.-W. YU, C.-M. CHEN, National Chiao-Tung University, Taiwan
11:20 am		**F3-12** The Effect of Surface Mechanical Attrition Treatment on Phase Transformation in Surface Layer of NiTi Alloy, **T. HU**, W. ZHANG, S. WU, Y. XIN, City University of Hong Kong, C.W CHU, Southeast University, R.K.Y. FU, Plasma Technology Limited, P.K. CHU, City University of Hong Kong, K.W. YEUNG, City University of Hong Kong, J. LU, The Hong Kong Polytechnic University, Hong Kong
	Exhibition Closes Today 2:00 pm	Exhibition Closes Today 2:00 pm

Wednesday Morning, April 29, 2009

	Applications, Manufacturing, and Equipment **Room: Royal Palm 1-3 - Session G1-1** **Innovations in Surface Coatings and Treatments** **Moderators:** R. Wei, Southwest Research Institute, M. Rodmar, Sandvik Tooling	**New Horizons in Coatings and Thin Films** **Room: Tiki Pavilion - Session H3** **Surface Engineering of Coatings: Tribo, Bio and Nano-Corrosion Effects** **Moderators:** M.M. Stack, Strathclyde University, E. Meletis, University of Texas at Arlington
8:00 am	**G1-1-1** High Ionization Plasma for Advanced Coatings: ANew Approach for Coating Deposition on Temperature Sensitive and Complex-Shaped Substrates, **J. ALAMI**, G. ERKENS, J. VETTER, J. MUELLER, T. RASA, W. JUNG, Sulzer Metaplas GmbH, Germany	**H3-1** Potentiodynamic and Potentiostatic Characterization of CVD Alumina Coating for Orthopaedic Implant Wear Reduction, **R. OVERHOLSER**, E. GULLEY, B. SMITH, DePuy, a Johnson & Johnson Company
8:20 am	**G1-1-2** Industrial Scale Deposition of Oxides, **L.P. NIELSEN**, K.P. ALMTOFT, I.H. ANDERSEN, Danish Technological Institute, Denmark, P. EKLUND, I. HANSEN-BRUHN, B. THOMSEN, J. BOTTIGER, Aarhus University, Denmark	**H3-2** Corrosion Behavior of Magnetron Sputtered Al-Mn Coatings in Neutral Saline Solution, **M. REFFASS**, C. BERZIOUB, C. REBERE, University of La Rochelle, France, A. BILLARD, University of Technology Belfort Montbeliard, France, J. CREUS, University of La Rochelle, France
8:40 am	**G1-1-3 Invited** High Power Pulsed Sputtering and Plasma-Assisted Hybrid Processes for Industrial Scale Hard Layer Deposition, **H. KLOSTERMANN**, F. FIETZKE, R. LABITZKE, T. MODES, O. ZYWITZKI, Fraunhofer FEP, Germany	**H3-3 Invited** Tribocorrosion Mechanisms: Materials and Coatings for Bio-Implants, **M.T. MATHEW**, University Medical Centre (RUMC), L.A. ROCHA, Universidade do Minho, Portugal, A FISCHER, University of Duisburg-Essen, Germany, M.A. WIMMER, Rush University Medical Centre (RUMC)
9:00 am	Invited talk continued.	Invited talk continued.
9:20 am	**G1-1-6** Nanocomposite PVD Coatings to Prevent Failure of Molten Aluminium Forming Dies, **E.A. TORRES MIRANDA**, Z. BRYTAN, D. UGUES, Politecnico di Torino, Italy, M. PERUCCA, Clean NT Lab - Environment Park S.p.A., Italy	**H3-5** Functionalization of Ti for Dental Implants Applications, A.C. ALVES, **L.A. ROCHA**, Universidade do Minho, Portugal
9:40 am	**G1-1-7** Magnetron Sputtering of Free-Standing Aluminium-Scandium Alloys for Use in Cold Forming Processes, **I. EISBRECHER**, H.-R. STOCK, Stiftung Institut fuer Werkstofftechnik, Germany	**H3-6** Characterisation and Evaluation of the Erosion-Corrosion Resistance of HIPIMS-HIPIMS Deposited Nanoscale CrN/NbN Multilayer Coatings, Y.P. PURANDARE, A.P. EHIASARIAN, Sheffield Hallam University, United Kingdom, **M.M. STACK**, Strathclyde University, United Kingdom, P.EH. HOVSEPIAN, Sheffield Hallam University, United Kingdom
10:00 am	**G1-1-8** Effect of Dry Micro-Blasting Conditions on Pvd Films' Properties and on Coated Tools Cutting Performance, **K.-D. BOUZAKIS**, G. SKORDARIS, S. GERARDIS, G. KATIRTZOGLOU, S. MAKRIMALLAKIS, N. MICHAILIDIS, Aristoteles University of Thessaloniki, Greece, F. KLOCKE, E. BOUZAKIS, Technical University of Aachen, Germany	**H3-7 Invited** Nanoscale Properties of In-Situ Formed Tribofilms from Thiophosphate Compounds, **P. ASWATH**, University of Texas at Arlington
10:20 am	**G1-1-10** Characterization and Antibacterial Properties of TaON–Ag Nanocomposite Thin Film, **J.H. HSIEH**, C.C. CHANG, Mingchi University of Technology, Taiwan	Invited talk continued.
10:40 am	**G1-1-11** Structure Effect on the Improvement of the Corrosion Resistance in a 4140 Nitrided Steel by Post-Discharge, **A. MEDINA**, UMSNH-Instituto de Investigaciones Metalúrgicas, Mexico, J. OSEGUERA, TESM-CEM, Mexico, H. CARREÓN, UMSNH-Instituto de Investigaciones Metalúrgicas, Mexico, L. BÉJAR-GÓMEZ, UMSNH-ngenieria Mecanica, Mexico	**H3-9** Nanostructured Aluminium Based Coatings Deposited by Electron-Beam Evaporative PVD, F. SANCHETTE, C. DUCROS, CEA de Grenoble, France, A. BILLARD, University of Technology Belfort Montbeliard, France, C. REBERE, University of La Rochelle, France, C. BERZIOU, Université de la Rochelle, France, M. REFFASS, **J. CREUS**, University of La Rochelle, France
11:00 am	**G1-1-12** Influence of Residual Stresses on the Growth Kinetics of Boride Layers, **M. ORTIZ-DOMÍNGUEZ**, I. CAMPOS, A. MENESES-AMADOR, J. MARTÍNEZ-TRINIDAD, Instituto Politécnico Nacional, México	**H3-10** Superhydrophilic Textured-Surfaces on Stainless Steel Substrates, H. WANG, **M. ZOU**, University of Arkansas, R. WEI, Southwest Research Institute
11:20 am	**G1-1-13** Improvement on Low-Temperature Deposited HfO_2 High-Pressure Oxygen/Ozone Treatment, **P.-C. YANG**, T.-C. CHANG, National Sun Yat-Sen University, Taiwan	**H3-11** Influence of Substrate Microstructures of Squeeze Cast Magnesium Alloy AJ62 on Corrosion Resistance of Plasma Electrolytic Oxidation Coating, L. HAN, P. ZHANG, J. NI, **X. NIE**, H. HU, University of Windsor, Canada
11:40 am		**H3-12** Corrosion Behavior of Nickel Doped Diamond-Like Carbon Thin Films in NaCl Solution, E. LIU, **N.W. KHUN**, Nanyang Technological University, Singapore
	Exhibition Closes Today 2:00 pm	Exhibition Closes Today 2:00 pm

Surface Engineering for Thermal Management
Room: Sunset - Session TS4
Surface Engineering for Thermal Interfaces, Heat Flow Control and Thermal Energy Management
Moderators: A.A. Voevodin, Air Force Research Laboratory, T.S. Fisher, Purdue University

8:00 am	**TS4-1 Invited** Thermal Conductivity Control with Thin Films, **D.G. CAHILL**, University of Illinois Urbana-Champaign	
8:20 am	Invited talk continued.	
8:40 am	**TS4-3** Thermal Conductivity Anisotropy in Molybdenum Disulfide Thin Films, **R. MCLAREN**, D.G. CAHILL, W. KING, University of Illinois Urbana-Champaign, C. MURATORE, Air Force Research Lab/UTC, Inc., J. HU, A.A. VOEVODIN, Air Force Research Laboratory	
9:00 am	**TS4-4** Fabrication and Characterisation of Nanoscale Heating Sources ('NanoHeaters') for Thermal Nanomanufacturing, **C. REBHOLZ**, K. FADENBERGER, I.E. GUNDUZ, M. KOKONOU, C.C. DOUMANIDIS, University of Cyprus, T. ANDO, Northeastern University, J. CHEN, Z GU, University of Massachusetts Lowell, P. WONG, Tufts University	
9:20 am	**TS4-6** Processing and Characterization of Carbon Nanotubes on Diamond/Si Substrates for Thermal Management Applications, **C. VARANASI**, University of Dayton Research Institute, J. PETRY, AFRL, J. BURKE, L. BRUNKE, Univ of Dayton Research Inst, J. BULMER, K. YOST, AFRL, W. LANTER, Innovative Scientific Solutions, Inc., J. SCOFIELD, P. BARNES, AFRL	
9:40 am	**TS4-7 Invited** Strain, Electronic Structure, Phonons and Thermal Properties of ScN:ZrN Superlattices: A First-Principles Study, **U.V. WAGHMARE**, J Nehru Centre for Advanced Scientific Research, India	
10:00 am	Invited talk continued.	
10:20 am	**TS4-9** GaN-on-Diamond Wafers for HEMT, the Diamond Side of the Story, **C. ENGDAHL**, E. FRANCIS, Crystallume	
10:40 am	**TS4-11** Modeling of Anisotropic Thermal Transport Behavior in Molybdenum Disulphide (MoS_2), **V. VARSHNEY**, Air Force Research Laboratory/RXBT, S. PATNAIK, Air Force Research Laboratory, A. ROY, Air Force Research Laboratories/RXBT, A.A. VOEVODIN, Air Force Research Lab/University of Dayton, B. FARMER, Air Force Research Laboratory	
11:00 am	**TS4-13** Models of Thermal Condutivity of Multilayer Coatings for Cutting Applications, L. BRAGINSKY, **V. SHKLOVER**, ETH Zürich, Switzerland, A. GUSAROV, ENISE, France	
	Exhibition Closes Today 2:00 pm	Exhibition Closes Today 2:00 pm

NOTES

Wednesday Afternoon, April 29, 2009

	Hard Coatings and Vapor Deposition Technology **Room: Golden West - Session B1-2** **Sputtering Coatings and Technologies** **Moderators:** C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University	**Optical Thin Films** **Room: Royal Palm 4-6 - Session C2-2** **Optical Thin Films for Active Devices and Microstystems** **Moderators:** M. Cremona, PUC-Rio, T. Miyata, Kanazawa Institute of Technology
1:30 pm	**B1-2-1 Invited** Development of Multi-Element Nitride/Oxide Coatings, **F.-S. SHIEU**, D.-C. TSAI, R.-S. YU, National Chung Hsing University, Taiwan	**C2-2-1** ZnO:Al Transparent Conducting Oxide Films of Very High Quality Synthesized by Filtered Cathodic Arc Deposition, **A. ANDERS**, Lawrence Berkeley National Laboratory, S. LIM, University of Sydney, Australia, K.M. YU, Lawrence Berkeley National Laboratory, J. ANDERSSON, Ångstrom Laboratory, Sweden, J. ROSÉN, Linköping University, Sweden, M. MCFARLAND, J. BROWN, Acree Technologies Inc.
1:50 pm	Invited talk continued.	**C2-2-2** Influence of Thermal Annealing on Electrical and Optical Properties of Ga-Doped ZnO Thin Films, **H. MAKINO**, N. YAMAMOTO, A. MIYAKE, T. YAMADA, Kochi University of Technology, Japan, Y. HIRASHIMA, H. IWAOKA, T. ITOH, Geomatec Co., Ltd., Japan, H. HOKARI, H. AOKI, Casio Computer Co., Ltd., Japan, T. YAMAMOTO, Kochi University of Technology, Japan
2:10 pm	**B1-2-3** Preferential Sputtering of Oxides and Target Design for Stable Reactive Magnetron Deposition of Oxides, **T. KUBART**, T. NYBERG, S. BERG, Uppsala University, Sweden	**C2-2-3 Invited** Attractive Potential Substitute for ITO in Flat Panel Display, **K. UTSUMI**, Tosoh Corporation, Japan
2:30 pm	**B1-2-4** Low Temperature Deposition of Anatase TiO_2 Film by Dual Magnetron Sputtering, J. SICHA, **P. BAROCH**, M. MEISSNER, R. CERSTVY, J. MUSIL, University of West Bohemia, Czech Republic	Invited talk continued.
2:50 pm	**B1-2-5** Photocatalysis and Phase Transformation of Oxygen-Deficient Carbon-Containing Titania Films, C.C. LIN, **Y.S. WANG**, P.W. CHOU, M.S. WONG, National Dong Hwa University, Taiwan	**C2-2-5** Thermopower and Optical Studies on Undoped and Manganese Doped Indium Tin Oxide Films, **S.R KUMAR**, V. DAMODARA DAS, S. KASIVISWANATHAN, Indian Institute of Technology Madras, India
3:10 pm	**B1-2-6** Nucleation and Crystal Growth Behavior of α-Alumina by Reactive Magnetron Sputtering, **H. TAMAGAKI**, Y. IKARI, H. FUJII, K. YAMAMOTO, Kobe Steel Ltd., Japan, T. KOHARA, Kohara Ltd., Japan	**C2-2-6** Reactively Co-Sputtering Transparent Conducting Oxide Films of Nb-Doped Titania, K.H. HONG, **P.W. LEE**, W.C. HSU, H.C. CHANG, M.S. WONG, National Dong Hwa University, Taiwan
3:30 pm	**B1-2-7** Low-Temperature Deposition of Alpha Alumina by Physical Vapor Deposition without a Chrome Template Layer, **A. BARITO**, University of Arkansas, H.H. ABU-SAFE, Lebanese American University, Lebanon, D.E. SPEAROT, M.H. GORDON, University of Arkansas	**C2-2-7** Structural, Electrical and Optical Properties of Transparent $Zn_{1-x}Mg_xO$ Nanocomposite Thin Films, **A. KAUSHIK**, D. KAUR, Indian Institute of Technology, India
3:50 pm	**B1-2-8** RF Sputtered Piezoelectric Zinc Oxide Layers for Microfluidic Applications, **K.F. WÄTJE**, A. WIXFORTH, University of Augsburg, Germany	Session D3-1
4:10 pm	**B1-2-9** Phase Transition and Mechanical Properties of Zr(N,O) Thin Films on AISI 304 Stainless Steel, **J.H. HUANG**, T.C. LIN, G.P. YU, National Tsing Hua University at Hsinchu, Taiwan	
4:30 pm	**B1-2-10** Preparation of Artificial $BiFeO_3/SrTiO_3$ Superlattices by rf Sputtering, **S.-J. CHIU**, G.P. YU, J.H. HUANG, National Tsing Hua University, Taiwan, H.-Y. LEE, National Synchrotron Radiation Research Center, Taiwan	
4:50 pm	**B1-2-11** PVD Module for High Quality AC Reactive Sputtering of Oxide and Nitride Thin Films, **V.V. FELMETSGER**, P.N. LAPTEV, S.M. TANNER, Tegal Corporation	Awards Convocation California Room 5:30 – 7:00 pm
5:10 pm	**B1-2-12** Adhesion Improvement of Reactive RF-Sputtered Zirconium Nitride Films on WC Substrate, **A. RIZZO**, M.A. SIGNORE, L. MIRENGHI, L. TAPFER, D. VALERINI, ENEA, UTS FIM, CR Brindisi, Italy, U. GALIETTI, DIMEG - Politecnico of Bari, Italy, D. ALTAMURA, University of Lecce, Italy	Awards Buffet Reception Poolside near Tiki Pavilion 7:30

Wednesday Afternoon, April 29, 2009

	Carbon and Nitride Materials: Synthesis-Structure-Property Relationships - Room: Royal Palm 4-6 - Session D3-1 **Carbon and Nitrogen-Containing Nanostructured Composite and Nanolaminated Films** **Moderators:** U. Jansson, Uppsala University, Y. Pauleau, Grenoble Polytechnic Institute, J.M. Ting, National Cheng Kung University	**Tribology and Mechanical Behavior of Coatings and Thin Films - Room: California - Session E1-2** **Friction and Wear of Coatings: Lubrication, Surface Effects and Modeling - Moderators:** E. Broitman, Carnegie Mellon University, A. Fernandez-Camacho, CSIC-University Sevilla, O.L. Eryilmaz, Argonne National Laboratory
1:30 pm	Session C2-2	**E1-2-1** Interfacial Scratch Adhesion Behavior of Multilayered Ti(BN):Ti(MoS_2) Based PVD Coatings, **I. EFEOGLU**, Ataturk University, Turkey, B. PRAKASH, J. HARDELL, Luleå University of Technology, Sweden
1:50 pm		**E1-2-2** Optimization of AlN Coatings for Tribological Applications, **A. ROJO**, ITESM-TOL, Mexico, J. OSEGUERA, O. SALAS, J. SOLIS, ITESM-CEM, Mexico
2:10 pm		**E1-2-3 Invited** Surface Chemical Mechanisms of Lubricants Under Sliding Conditions, **W. TYSOE**, UW-Milwaukee
2:30 pm		Invited talk continued.
2:50 pm		**E1-2-7** Expanding the Range of Temperature-Adaptive Solid Lubricant Coatings Beyond 25-700°C with the Aid of In Situ Wear and Surface Analysis Techniques, **C. MURATORE**, J. HU, J. BULTMAN, Air Force Research Laboratory, A.A. VOEVODIN, Air Force Research Lab/University of Dayton
3:10 pm		**E1-2-8** Optimization of the Coating Parameters for Micro Arc oxidation of Cp-Ti, **Y. VANGOLU**, A. ALSARAN, E. ARSLAN, Y. TOTIK, Ataturk University, Turkey
3:30 pm		**E1-2-10** Effects of Sicking-Sliding Zones on the Tool Wear and Coating Delamination When Machining Hard Materials, **S. BAHI**, A. MOUFKI, M. NOUARI, Ecole Nationale Supérieure des Mines de Nancy, France, M. EL MANSORI, Arts et Métiers Paristech, France, A. MOLINARI, Ecole Nationale Supérieure des Mines de Nancy, France
3:50 pm	**D3-1-8 Invited** Applications of Nanocomposites in Organic Electronic Devices, **T.P. NGUYEN**, Institut des Materiaux Jean Rouxel, France, C.W. LEE, University of Chiao Tung, Taiwan	
4:10 pm	Invited talk continued.	
4:30 pm	**D3-1-11** Cermet Structured a-c:H/Pt Thin Films as High Temperature Selective Solar Absorber Coatings, **Y.H. LAN**, W.Y. WU, J.M. TING, National Cheng Kung University, Taiwan	
4:50 pm	**D3-1-12** Microstructure and Mechanical Properties of CrAlN/Si_3N_4 Nanostructure Multilayered Coatings, J.-G. DUH, **S.-H. TSAO**, National Tsing-Hua University, Taiwan	Awards Convocation California Room 5:30 – 7:00 pm
5:10 pm	**D3-1-10** Sol Gel Derived Hybrid Dielectric Thin films for ULSI Applications, **A.M. MAHAJAN**, B.N. JOSHI, North Maharashtra University, India	Awards Buffet Reception Poolside near Tiki Pavilion 7:30

Wednesday Afternoon, April 29, 2009

	Characterization: Linking Synthesis Properties and Microstructure - Room: Sunrise - Session F2/B7 **In Situ Characterization for Deposition Proceess and Film Properties Modeling** **Moderators:** M. Beckers, Linköping University, D. Depla, Ghent University	**Applications, Manufacturing, and Equipment Room: Royal Palm 1-3 - Session G7** **Advances in Industrial PVD & CVD Deposition** **Moderators:** R. Cremer, KCS Europe GmbH, K. Yamamoto, Kobe Steel Ltd.
1:30 pm	**F2/B7-1** Quantification of the Incorporation Coefficient of a Reactive Gas on a Metallic Film During Magnetron Sputtering: the Method and Results, **W.P. LEROY**, S. MAHIEU, Ghent University, Belgium, R. PERSOONS, Flemish Institute for Technological Research (VITO), Belgium, D. DEPLA, Ghent University, Belgium	**G7-1** Innovative Applications Enabled by Large Area Hot Filament Diamond Deposition, **J.W. ZIMMER**, D. AIDALA, J HERLINGER, sp3 Diamond Technologies
1:50 pm	**F2/B7-2** Experimental and Numerical Plasma Characterization in a Deep Reactive Ion Etch System, **S.P. KOIRALA**, M.H. GORDON, University of Arkansas, S.L. BURKETT, The University of Alabama	**G7-2** Industrial Scale Production of CVD-Diamond Coated Mechanical Seals and Electrodes, **M. RÜFFER**, M. FORETA, J. HOLZKE, M. NIERADA, DiaCCon GmbH, Germany, S. ROSIWALL, University Erlangen Nürnberg, Germany
2:10 pm	**F2/B7-3 Invited** Initial Stages of Polycrystalline Thin Film Growth as Seen by Scanning Probe Microscopy, **T. MICHELY**, Universität zu Köln, Germany	**G7-3** Characterization of Industrial-Scale LAFAD Technology and Applications, **V.I. GOROKHOVSKY**, Arcomac Surface Engineering, LLC
2:30 pm	Invited talk continued.	**G7-4** Development of a Magnetically Steered Cathodic Arc Evaporation Source, **K. YAMAMOTO**, S. TANIFUJI, S. NAKAKUBO, H. FUJII, Y. KUROKAWA, S. KUJIME, Kobe Steel Ltd., Japan
2:50 pm	**F2/B7-5** Surface Mound Formation During Epitaxial Growth of CrN(001), **X.Y. ZHANG**, D. GALL, Rensselaer Polytechnic Institute	**G7-7** Integration Aspects of High Impulse Magnetron Sputtering Plus (HIPIMS+) Technology in an Industrial PVD Coating Machine, **F. PAPA**, R. TIETEMA, T. KRUG, C. STRONDL, I. KOLEV, Hauzer Techno Coating BV, Netherlands
3:10 pm	**F2/B7-6** In Situ Stress Evolution in TiZrN and TiTaN Thin Films Grown by Reactive Magnetron Sputtering, **G. ABADIAS**, Universite de Poitiers, France, PH. GUERIN, Université de Poitiers-CNRS, Laboratoire PHYMAT, France, L.E. KOUTSOKERAS, P. PATSALAS, University of Ioannina, Greece	**G7-8** The New Vacotec/Eifeler Alpha 400/900P PVD Deposition System as a Basis for Innovative Coatings for Industries and Research, **H. HRUBÝ**, E. VOSS, G. KEIREN, Eifeler Werkzeuge GmbH, Germany, J. ANKLAM, Vacotec S.A., Switzerland
3:30 pm	**F2/B7-7** In Situ AFM Investigation on Tribo-Corrosion of CrSiN Film Adherent Tool Steel, **H.-H. LIN**, C.-C. CHOU, National Taiwan Ocean University, Taiwan, J.-C. HUANG, J.-W. LEE, Y.-C. CHANG, Y.-B. LIN, Tungnan University, Taiwan	**E4/G4-9** Hot Filament Diamond CVD – Technology and Applications, **W. REICHERT**, R. CREMER, O. LEMMER, CemeCon AG, Germany
3:50 pm	**F2/B7-8** Fast Characterization of Reaction Waves in Exothermic, Metal-Metal Multilayer Nanolaminates, **D.P. ADAMS**, J. MCDONALD, M HOBBS, Sandia National Laboratories	
4:10 pm	**F2/B7-9** Quantitative Measurement of Ion Energy Distributions Impinging onto Arbitrarily Biased Substrates During Plasma Deposition, **T. BALONIAK**, A. VON KEUDELL, Ruhr-Universität Bochum, Germany	
	Awards Convocation California Room 5:30 – 7:00 pm	Awards Buffet Reception Poolside near Tiki Pavilion 7:30

Wednesday Afternoon, April 29, 2009

	New Horizons in Coatings and Thin Films **Room: Tiki Pavilion - Session H4** **Thin Films for Photovoltaics: Synthesis and Characterization** **Moderators:** S. Khare, University of Toledo, S. Fairchild, Air Force Research Laboratory	**Coatings for Fuel Cells** **Room: Sunset - Session TS2** **Coatings for Fuel Cells** **Moderators:** J.W. Stevenson, Pacific Northwest National Laboratory, D. Mumm, University of California
1:30 pm	**H4-1 Invited** Manufacturing Photovoltaic Systems, **L. KAZMERSKI**, NREL	**TS2-1 Invited** Electroplated Coatings on Ferritic Steels for SOFC Interconnect Application, **J.H. ZHU**, Tennessee Technological University
1:50 pm	Invited talk continued.	Invited talk continued.
2:10 pm	**H4-3** Steady State and Transient Photoconductivity of a-$Se_{90-x}Sb_{10}In_x$ ($0 \leq x \leq 15$) Thin Films, **M. KAMBOJ**, F. MOHAMMADI, Ryerson University Toronto, Canada	**TS2-3** Improved Properties of SOFCs using Pre-Coated Sandvik Sanergy HT 22% Cr Ferritic Interconnect Steel, **U. BEXELL**, M. SCHUISKY, AB Sandvik Materials Technology, Sweden
2:30 pm	**H4-5** Glancing-Angle Deposited Titania Films for Dye-Sensitized Solar Cells, H.Y. YANG, **M.F. LEE**, M.S. WONG, National Dong Hwa University, Taiwan	**TS2-5** Development of Spinel Protection Layers for Steel-Based SOFC Interconnects, **J.W. STEVENSON**, Z.G. YANG, G.B. XIA, X.S. LI, Z.M. NIE, C.M. WANG, Pacific Northwest National Laboratory
2:50 pm	**H4-6 Invited** High Performance Polymer Soloar Cells and Optical Sensors, **Y. YANG**, UCLA	**TS2-7** Structure and Conductivity of Apatite-Like Lanthanum Silicate Films for SOFcs Electrolytes, **J.C. OLIVEIRA**, University of Coimbra, Portugal, M. VIEIRA, Polytechnic Institute of Leiria, Portugal, A.L. SHAULA, A. CAVALEIRO, University of Coimbra, Portugal
3:10 pm	Invited talk continued.	**TS2-8** The Corrosion Properties and Interfacial Contact Resistance of TiN, TiAlN and CrN PVD Coatings in Simulated PEM Fuel Cell Environments, **L. WANG**, D.O. NORTHWOOD, University of Windsor, Canada, J. HOUSDEN, E. SPAIN, Tecvac Ltd., X. NIE, University of Windsor, Canada
3:30 pm	**H4-8** Hybrid Solar Cells Based on Poly (3-Hexylthiophene):Fullerene Blend and TiO_2 Porous Film, M.K. FUNG, C.T. YIP, **K.Y. CHEUNG**, A.B. DJURIŠIĆ, W.K. CHAN, The University of Hong Kong	
3:50 pm	**H4-10** Photoelectrochemical Properties of Nitrogen-Doped Indium Tin Oxide Thin Films Prepared by Reactive DC Magnetron Sputtering Technique, **K.R. WU**, National Kaohsiung Marine University, Taiwan, C.Y. YEH, Kao Yuan University, Taiwan, C.H. HUNG, National Kaohsiung First University of Science and Technology, Taiwan, C.Y. CHUNG, National kaohsiung Marine University, Taiwan	
	Awards Convocation California Room 5:30 – 7:00 pm	Awards Buffet Reception Poolside near Tiki Pavilion 7:30

Thursday Morning, April 30, 2009

	Coatings for Use at High Temperature **Room: Royal Palm 1-3 - Session A2** **Coatings for Use in Harsh Environments** **Moderators:** J.R. Nicholls, Cranfield University, M. Schütze, DECHEMA e.V.	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B1-3** **Sputtering Coatings and Technologies** **Moderators:** C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University
8:00 am	**A2-1** Application of PVD Coatings in Food Processing Tools, **M. ROSTAGNO**, S. DURANTE, DIAD, Italy, M. PERUCCA, Clean NT Lab - Environment Park S.p.A., Italy, E.A. BOOT, F. CARTASEGNA, Environmental Park, Italy	**B1-3-1** Study of the Effect of Plasma Current Density on Nitrides and Oxynitrides Titanium Thin Films Prepared by Reactive DC Magnetron Sputtering, **P.K. BARHAI**, N. KUMARI, I. BANERJEE, Birla Institute of Technology, India, S.K. PABI, Indian Institute of Technology Kharagpur, S.K. MAHAPATRA, Birla Institute of Technology, India
8:20 am	**A2-3** Electrochemical Impedance Spectroscopy (EIS) Study on Corrosion Performance of CrAlSiN Coated Steels in 3.5 wt.% NaCl Solution, **C.H. LIN**, J.G. DUH, National Tsing-Hua University, Taiwan	**B1-3-2** Chromium Containing Amorphous Hydrogenated Carbon Thin Films (a-c:H/Cr) as Selective Solar Absorber Coatings, **H.Y. CHENG**, W.Y. WU, J.M. TING, National Cheng Kung University, Taiwan
8:40 am	**A2-4** A Dry Drilling Process Contribution with Solid Carbide TiAlN + AlCrN Coated Drill, **W. MATTES**, SENAI-SC, ALVES, Brazil, S. MARTINS, SOCIESC, Brazil	**B1-3-3 Invited** Investigation of the O^- Ion Emission During Reactive Magnetron Sputtering, **S. MAHIEU**, W.P. LEROY, D. DEPLA, Ghent University, Belgium
9:00 am	**A2-5 Invited** High Temperature Corrosion Behaviour of Materials in Heavy-Duty Gas Turbines with Fuel Flexibility, **B. BORDENET**, Alstom (Switzerland) Ltd., Switzerland	Invited talk continued.
9:20 am	Invited talk continued.	**B1-3-5** A Satisfactory Explanation for the Discharge Voltage Behaviour During Reactive Magnetron Sputtering, **D. DEPLA**, S. MAHIEU, R. DE GRYSE, Ghent University, Belgium
9:40 am	**A2-7** Diffusion Coatings for Oxidizing High-Chlorine Environments at Elevated Temperatures, **B. RAMMER**, T. WEBER, M. SCHÜTZE, DECHEMA e.V., Germany	**B1-3-6** Ion Energy Distributions in AZO Magnetron Sputtering from Planar and Rotatable Magnetrons, **F. RICHTER**, T. WELZEL, TU Chemnitz, Germany, R. KLEINHEMPEL, Southwall Europe GmbH, Germany, T. DUNGER, TU Chemnitz, Germany, T. KNOTH, M. DIMER, F. MILDE, von Ardenne Anlagentechnik GmbH, Germany
10:00 am	**A2-10** Production, Characterization and Evaluation of Protective Cr Oxide Coatings Against Metal Dusting, D. MELO, IPN, Mexico, D. SINGÜENZA, **O. SALAS**, ITESM-CEM, Mexico, R. REICHELT, Wilhelms-Universitaet, Germany, J. OSEGUERA, ITESM-CEM, Mexico, . LÓPEZ, IPN, Mexico	**B1-3-7** Investigation of the Role of Hydrogen in Silicon Deposition Using an Energy-Resolve Mass Spectrometer in an Ar/H2 Radio Frequency Magnetron Discharge, **S.L. MENSAH**, University of Arkansas, H.H. ABU-SAFE, Lebanese American University, Lebanon, H.A NASEEM, M.H. GORDON, University of Arkansas
10:20 am	**A2-9** Factors Affecting the Performance of a Porous, High Temperature Abradable Coating, **D. ALLEN**, Siemens Energy	**B1-3-8** Simulation of Layer Sequence in Multilayer Coatings Prepared by Sputtering, **M. PANJAN**, Jozef Stefan Institute, Slovenia, T. PETERMAN, University of Ljubljana, Slovenia, P. PANJAN, M. CEKADA, Jozef Stefan Institute, Slovenia
10:40 am	**A2-12** An Overview of Sulzer Metco Abradable Coatings and Some New Developments in Blade Tipping, **S. WILSON**, Sulzer Metco, Switzerland, D. SPORER, Sulzer Metco Europe, M. DORFMAN, Sulzer Metco, USA	**B1-3-9** Improving the Oxidation Resistance of AlCrn Coatings by Tailoring Chromium Out-Diffusion, **R. ESCOBAR GALINDO**, Universidad Autonoma de Madrid, Spain, J. ENDRINO, Instituto de Ciencia de Materiales de Madrid, Spain, G. FOX-RABINOVICH, McMaster University, Canada, J.M. ALBELLA, Instituto de Ciencia de Materiales de Madrid, Spain
11:00 am	**A2-8** Microstructure and Cutting Mechanics in Abradable Seal Coatings, S. GOERGEN, Cranfield University, United Kingdom, C. SELLARS, Rolls Royce Plc, United Kingdom, I. WALTON, D.J. STEPHENSON, **J.R. NICHOLLS**, Cranfield University, United Kingdom	**B1-3-10** Phase Transformation, Thermal Stability, Morphological and Mechanical Characteristics of the Ni- Al and Ni-P-Al Alloy Coating Systems, **C.C. WU**, F.B. WU, National United University, Taiwan
11:20 am	**A2-11** Degradation of Hot-Dip Aluminized Coating on the Cyclic Oxidation of the Ferritic 430 Stainless Steel, **M. BADARUDDIN**, C.J. WANG, National Taiwan University of Science and Technology (NTUST), Taiwan	**B1-3-11** Oxidation Study of Mo-Ru Hard Coatings, **Y.-I. CHEN**, National Taiwan Ocean University, Taiwan
	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm

Thursday Morning, April 30, 2009

	Hard Coatings and Vapor Deposition Technology **Room: Sunrise - Session B6-1** **Hard and Multifunctional Nano-Structured Coatings** **Moderators:** M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Laboratories, U.S. Army ARDEC, R. Sanjines, EPFL	**Carbon and Nitride Materials: Synthesis-Structure-Property Relationships - Room: Royal Palm 4-6 - Session D3-2** **Carbon and Nitrogen-Containing Nanostructured Composite and Nanolaminated Films** **Moderators:** U. Jansson, Uppsala University, Y. Pauleau, Grenoble Polytechnic Institute, J.M. Ting, National Cheng Kung University
8:00 am	**B6-1-1 Invited** Processes, Properties and Application Potential of Ti-Al-X-N and Cr-Al-X-N Thin Films, **P.H. MAYRHOFER**, Montanuniversität Leoben, Austria	**D3-2-1 Invited** The MAX Phases and Kinking Non-Linear Elastic Solids; A Newly Identified Class of Solids, **M.W. BARSOUM**, Drexel University
8:20 am	Invited talk continued.	Invited talk continued.
8:40 am	**B6-1-3** Thermal Decomposition of Arc Evaporated ZrAlN Thin Films, **L. ROGSTRÖM**, L.J.S. JOHNSON, Linköping University, Sweden, M. JOHANSSON, SECO Tools AB, Sweden, T. MYRTVEIT, Sandvik Tooling AB, Sweden, L. HULTMAN, M. ODÉN, Linköping University, Sweden	**D3-2-3** Electronic Structure Investigation of MAX-Phases by Soft X-ray Emission Spectroscopy, **M. MAGNUSON**, Linköping University, Sweden, U. JANSSON, Uppsala University, Sweden, L. HULTMAN, Linköping University, Sweden
9:00 am	**B6-1-4** Growth, Annealing Behaviour and Cutting Performance of (Ti,Si)(C,N) Coatings, **L.J.S. JOHNSON**, L. ROGSTRÖM, Linköping University, Sweden, M. JOHANSSON, SECO Tools AB, Sweden, M. COLLIN, Sandvik Tooling AB, Sweden, J. SJÖLÉN, SECO Tools AB, Sweden, M. ODÉN, L. HULTMAN, Linköping University, Sweden	**D3-2-4** A Computational Study of the Effects of Impurities on MAX Phase Elastic Properties, **M.F. COVER**, M.M.M. BILEK, D.R. MCKENZIE, University of Sydney, Australia
9:20 am	**B6-1-5** Microstructure, Mechanical and Tribological Properties of Cr-C-N Coatings Deposited by Pulsed Closed Field Unbalanced Magnetron Sputtering, **Z.L. WU**, Colorado School of Mines, and Dalian University of Technology, China, J. LIN, J.J. MOORE, Colorado School of Mines, M.K. LEI, Dalian University of Technology, China	**D3-2-5** Mechanical Deformation Properties of Ti_2AlC MAX Phase Thin Films with High Oxygen Content, **A. MOCKUTE**, P. PERSSON, F. GIULIANI, L. HULTMAN, Linköping University, Sweden, M.M.M. BILEK, University of Sydney, Australia, J. ROSÉN, Linköping University, Sweden
9:40 am	**B6-1-6** Comparative Investigation of TiAlC(N), TiCrAlC(N), and CrAlC(N) Coatings Deposited by Sputtering of MAX-Phase $Ti_{2-x}Cr_xAlC$ Targets, **D.V. SHTANSKY**, PH.V. KIRYUKHANTSEV-KORNEEV, A.N. SHEVEYKO, D.I. SOROKIN, State Tech Univ "Moscow Inst of Steel & Alloys", Russia, B.N. MAVRIN, C. ROJAS, A. FERNANDEZ, E.A. LEVASHOV	**D3-2-6** The Effects of Surface Structure, Incident Ion Energy, and Impurity Incorporation on MAX Phase Nucleation and Growth, **M.D. TUCKER**, M.C. GUENETTE, P.O.Å. PERSSON, J. ROSÉN, M.M.M. BILEK, D.R. MCKENZIE, University of Sydney, Australia
10:00 am	**B6-1-7 Invited** The Effect of Magnetron Pulsing on the Structure and Properties of Nanostructured Multifunctional Tribological Coatings, **J.J. MOORE**, J. LIN, B. MISHRA, Colorado School of Mines, W.D. SPROUL, Reactive Sputtering, Inc., J.A. REES, Hiden Analytical, LTd., United Kingdom	**D3-2-7** Microstructural Modifications of TiX(X=C or N)/TiAl Multilayers During Thermal Annealing and Ion-Irradiation, **T. CABIOCH**, M. BUGNET, M. JAOUEN, University of Poitiers, France
10:20 am	Invited talk continued.	
10:40 am	**B6-1-10** Thermally Enhanced Mechanically Properties of Arc Evaporated TiN/TiAlN Multialyer Thin Films, **A. KNUTSSON**, Linköping University, Sweden, M. JOHANSSON, SECO Tools AB, Sweden, M. ODÉN, Linköping University, Sweden	
11:00 am	**B6-1-11** Characterization of the Adhesion and Tribology of Ti/TiAlN Multilayer PVD Coatings Deposited on Pre-Nitrided Tool Steels, W. TILLMANN, E. VOGLI, **S. MOMENI**, Dortmund University of Technology, Germany	
11:20 am	**B6-1-12** Characteristics of Cr_2N/Cu Multilayered Thin Films with Different Bilayer Thickness, **C.-L. LI**, J.-W. LEE, Tungnan University, Taiwan, L.-C. CHANG, Mingchi University of Technology, Taiwan	
	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm

Thursday Morning, April 30, 2009

	Tribology and Mechanical Behavior of Coatings and Thin Films - Room: California - Session E4/G4 **Coatings for Machining Advanced Materials and Advanced Manufacturing Methods** **Moderators:** W. Kalss, Oerlikon Balzers Coatings, H.-G. Fuss, CemeCon A.G.	**New Horizons in Coatings and Thin Films** **Room: Sunset - Session H2-1** **High Power Impulse Magnetron Sputtering** **Moderators:** A.P. Ehiasarian, Sheffield Hallam University, K. Marchev, P&G Company
8:00 am	**E4/G4-3 Invited** Adaptive PVD Coating for Machining of Hard to Cut Materials, **G. FOX-RABINOVICH**, McMaster University, Canada, K. YAMAMOTO, Kobelco, Japan, S. VELDHUIS, McMaster University, Canada	**H2-1-1 Invited** Target-Plasma-Film Interactions in High Power Pulsed Magnetron Sputtering, **K. SARAKINOS**, RWTH Aachen University, Germany
8:20 am	Invited talk continued.	Invited talk continued.
8:40 am	**E4/G4-5** Crystalline γ-Alumina Deposited in an Industrial Coating Unit for Demanding Turning Operations, K. BOBZIN, N. BAGCIVAN, P. IMMICH, **M. EWERING**, RWTH Aachen University, Germany	**H2-1-3** Time Resolved Optical Imaging of HIPIMS Discharges: Selecting Pulse Parameters Based on Target Mass, **J.G. JONES**, C. MURATORE, Air Force Research Laboratory, A.N. REED, Air Force Research Laboratory/University of Dayton Research Institute, A.R. WAITE, Air Force Research Laboratory/UTC, Inc./University of Dayton, C.A. CERBUS, Air Force Research Laboratory/University of Dayton Research Institute, S.F. NOSS, A.A. VOEVODIN, Air Force Research Laboratory
9:00 am	**E4/G4-6** Investigating the Performance of TiN and AlTiN Coatings on Milling Cutter Used for Machining Bimetal Steel, **M. SARWAR**, J. HAIDER, Northumbria University, United Kingdom, M. PERSSON, H. HELLBERGH, SNA Europe, Sweden	**H2-1-4** Characterization of HIPIMS Discharge for Next Generation Semiconductor Fabrication, **A.N. CLOUD**, R.E. FLAUTA, M.J. NEUMANN, S.L. ROHDE, D.N. RUZIC, University of Illinois at Urbana-Champaign
9:20 am	**E4/G4-7** Process Variability in Honing of Cylinder Liner with Vitrified Bonded Diamond Tools, **L. SABRI**, Renault, France, S. MEZGHANI, M. EL MANSORI, Arts et Métiers Paris Tech, France	**H2-1-5** Distance Dependent Plasma Composition and Ion Energy Distribution Functions in High Power Impulse Magnetron Sputtering of Ti and Cr, **A.P. EHIASARIAN**, Sheffield Hallam University, United Kingdom, J. ANDERSSON, Ångstrom Laboratory, Sweden, A. ANDERS, Lawrence Berkeley National Laboratory
9:40 am	**E4/G4-8** Influence of Nanostructured CVD Diamond Coatings During Dry Turning of a SiC Particle-Reinforced Metal Matrix Composite, **A. KREMER**, M. EL MANSORI, Arts et Métiers Paristech, France	**H2-1-6** Mass/Energy Analysis of the Plasma During MPP and Conventional DC Sputter Deposition of Cr and CrN Films, **W.D. SPROUL**, Reactive Sputtering, Inc., J. LIN, J.J. MOORE, Colorado School of Mines, Z.L. WU, Colorado School of Mines, and Dalian University of Technology, China, X. ZHANG, Colorado School of Mines, R. CHISTYAKOV, Zond, Inc., B. ABRAHAM, Zpulser, LLC, J.A. REES, Hiden Analytical, Ltd.
10:00 am	**E4/G4-10** Coating Thickness Effects on Diamond Coated Cutting Tools, **F. QIN**, Y. CHOU, The University of Alabama, D. NOLEN, R. THOMPSON, Vista Engineering	**H2-1-7** Stress in TiN Coatings Grown by HIPIMS, **R. MACHUNZE**, Delft University of Technology,, Netherlands, A.P. EHIASARIAN, Sheffield Hallam University, United Kingdom, G.C.A.M. JANSSEN, Delft University of Technology, Netherlands
10:20 am		**H2-1-8** Cutting Performance Improvement of PVD TiaIn-Based Coatings, Produced by HPPMS Technology, K.-D. BOUZAKIS, G. SKORDARIS, S. GERARDIS, G. KATIRTZOGLOU, S. MAKRIMALLAKIS, M. PAPPA, Aristoteles University of Thessaloniki, Greece, **R. CREMER**, H.-G. FUSS, W. KOELKER, J. DUKWEN, CemeCon A.G., Germany
	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm	2010 ICMCTF Planning Meeting (open to all interested attendees) California Room, 12:00 – 1:15 pm

NOTES

Thursday Afternoon, April 30, 2009

	Coatings for Use at High Temperature **Room: Royal Palm 1-3 - Session A3-1** **Thermal Barrier Coatings** **Moderators:** A. Bolcavage, Rolls-Royce Corporation, R. Mevrel, ONERA, K. Murphy, Howmet Castings	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B6-2** **Hard and Multifunctional Nano-Structured Coatings** **Moderators:** M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Labs, U.S. Army ARDEC, R. Sanjines, EPFL
1:30 pm	**A3-1-1** Toughening Mechanisms in 7YSZ Thermal Barrier Coatings, N.R. PHILIPS, **E.M. DONOHUE**, University of California, Santa Barbara, D. KOHL, Universität Stuttgart, Germany / summer internship at University of California, Santa Barbara, C.G. LEVI, A.G. EVANS, University of California, Santa Barbara	**B6-2-1 Invited** Metal Carbide/Amorphous C-Based Nanocomposite Coatings for Tribological Applications, **J.C. SANCHEZ-LOPEZ**, D. MARTINEZ-MARTINEZ, M.D. ABAD, A. FERNANDEZ, Instituto de Ciencia de Materiales de Sevilla, Spain
1:50 pm	**A3-1-2** Nondestructive Microstructural Characterization of Thermal Barrier Coatings by Laser Flash Technique, **F. CERNUSCHI**, Cesi Ricerca, Italy, P. BISON, CNR-ITC, Italy, A. MOSCATELLI, Politecnico di Milano, Italy	Invited talk continued.
2:10 pm	**A3-1-3 Invited** Recent Results on Advanced Thermal Barrier Coatings, D. STÖVER, **R. VAßEN**, O. JARLIGO, H. KASSNER, Y. ZHANG, D. MACK, G. MAUER, Institute of Energy Research, Germany	**B6-2-3** XPS Analysis of Binary and Ternary TiC-Based Alloy and Nanocomposite Coatings, **E. LEWIN**, Uppsala University, Sweden, M. GORGOI, BESSY GmbH, Germany, U. JANSSON, Uppsala University, Sweden
2:30 pm	Invited talk continued.	**B6-2-4 Invited** Fabrication and Tribological Properties of Composite Coatings Produced by Lithographic and Microbeading Methods, **J.E. KRZANOWSKI**, University of New Hampshire
2:50 pm	**A3-1-5** The Relation Between Morphology, Phase and Thermal Conductivity Changes in the Thermally Loaded EB-PVD TBCs, **R. OCHROMBEL**, German Aerospace Center, Germany, V. RYUKHTIN, Technical University of Berlin, Germany, B. SARUHAN, German Aerospace Center, Germany	Invited talk continued.
3:10 pm	**A3-1-6** The Behavior of High-Purity, Low-Density Air Plasma Sprayed Thermal Barrier Coatings, G.H. MEIER, **M.A. HELMINIAK**, N.M. YANAR, F.S. PETTIT, University of Pittsburgh, T.A. TAYLOR, Praxair Surface Technologies	**B6-2-6** Microstructures and Corrosion Resistance of Pulsed DC Reactive Magnetron Sputtered nanocomposite Zr-Si-N Thin Films, **Y.-B. LIN**, J.-W. LEE, Tungnan University, Taiwan, L.-C. CHANG, Mingchi University of Technology, Taiwan
3:30 pm	**A3-1-7** Thermo-Mechanical Properties and Gradient Testing of Thermal Barrier Coatings Subject to Spallation Due to CMAS (Calcium-Magnesium-Alumino-Silicate) Penetration, **S. FAULHABER**, A.G. EVANS, University of California Santa Barbara	**B6-2-7** Hard and Decorative Coatings Based on Al-N + Au Nanocomposite Structures, **A. CAVALEIRO**, C. LOURO, N. FIGUEIREDO, University of Coimbra, Portugal
3:50 pm	**A3-1-8** Effect of Low Level CMAS Attack on EB PVD TBCs, **R.G. WELLMAN**, G. WHITMAN, J.R. NICHOLLS, Cranfield University, United Kingdom	**B6-2-8** Microstructure – Property Relationships in Nitride-Based Coatings on Steel Substrates Prepared by Pulsed Laser Deposition, **A. JAHJA**, P. MUNROE, University of New South Wales, Australia
4:10 pm	**A3-1-9** The Role of Crystallization in Arresting CMAS Infiltration into TBCs, **E.M. VOGEL**, S KRÄMER, C.G. LEVI, University of California, Santa Barbara	**B6-2-9** Chromium-Aluminum Oxide Coatings Deposited by Reactive Magnetron Sputtering, **P. EKLUND**, Linköping University, Sweden, K. PEDERSEN, University of Aarhus, Denmark, K.P. ALMTOFT, L.P. NIELSEN, Danish Technological Institute, Denmark, M. SRIDHARAN, M. SILLASSEN, J. BOTTIGER, University of Aarhus, Denmark
4:30 pm	**A3-1-10** Electrophoretically Deposited Alumina as Protective Overlay for Thermal Barrier Coatings Against CMAS Degradation, **P. MOHAN**, T. PATTERSON, Y.H. SOHN, University of Central Florida	
4:50 pm	**A3-1-11** Dependence of Microstructure and Mechanical Properties with Starting Powder Morphology in Zirconia-Based Thermal Barrier Coatings, **Y.G. JUNG**, S.I. JUNG, J.Y. KWON, Y.S. SIM, U. PAIK, Changwon National University, Korea, K.S. LEE, Kookmin University, Korea	
	Poster Session Town and Country/San Diego Rooms 5:00 - 7:00 pm	Poster Reception Town and Country/San Diego Rooms 5:30 - 7:00 pm

Thursday Afternoon, April 30, 2009

	Optical Thin Films **Room: Royal Palm 4-6 - Session C1** **Recent Advances in Optical Thin Films** **Moderators:** J. Bellum, Sandia National Laboratory, R. Sczupak, Reynard Corporation	**Tribology and Mechanical Behavior of Coatings and Thin Films** **Room: California - Session E3-1** **Tribology of Nanostructured and Amorphous Films** **Moderators:** J. Fontaine, Ecole Centrale de Lyon, T.W. Scharf, The University of North Texas
1:30 pm	**C1-1 Invited** Unusual Optical Thin Film Solutions Based on Advances in Materials and Deposition Processes, **J.A. DOBROWOLSKI**, NRC, Canada	**E3-1-1** Friction Behavior of Nanocrystalline Diamond Coatings at Variable Sliding Speeds, **N. THEODORE**, Naval Research Laboratory / North Carolina State University, K. WAHL, Naval Research Laboratory
1:50 pm	Invited talk continued.	**E3-1-2** Tribology of Deuterated Diamond-Like Carbon Films : An Imaging TOF-SIMS Study, **O.L. ERYILMAZ**, A. ERDEMIR, G. KARTAL, Argonne National Laboratory
2:10 pm	**C1-3** Thermal Stability of SiO_xN_y Thin Films with Tailored Refraction Index: Microstructural, Chemical and Mechanical Properties, V. GODINHO, C. FERNANDEZ-RAMOS, M.C. JIMENEZ DE HARO, M.P. DELPLANCKE-OGLETREE, **A. FERNANDEZ**, Instituto de Ciencia de Materiales de Sevilla, Spain	**E3-1-3 Invited** Super-Low Friction of Carbon-Based Coatings in Nitrogen Gas, **K. ADACHI**, Tohoku University
2:30 pm	**C1-4** Improved Contrast and Reflectivity of Near Normal Incidence Reflective Mjltilayer Optics for FLASH and Next Generation Soft X-ray Lithography, **T. TSARFATI**, E. ZOETHOUT, R.W.E. VAN DE KRUIJS, F. BIJKERK, FOM Institute for Plasma Physics Rijnhuizen, Netherlands	Invited talk continued.
2:50 pm	**C1-5 Invited** Multi-Layer Dielectric Coatings and Processing for Production of Optical Gratings on Large Substrates, **D.J. SMITH**, Plymouth Grating Laboratory, Inc.	**E3-1-5** Tribological Behavior of DLC Coated Spinal Disk Implants, **G. THORWARTH**, U. MÜLLER, C.V. FALUB, R. HAUERT, B. WEISSE, Empa, Switzerland, C. VOISARD, Synthes GmbH, Switzerland, M. TOBLER, IonBond AG, Switzerland
3:10 pm	Invited talk continued.	**E3-1-6** Tribological and Mechanical Properties of Nanostructured Hydrogenated Amorphous Carbon and Titanium Diboride Films, **B. ZHAO**, Y.W. CHUNG, Northwestern University
3:30 pm	**C1-7** Visible Light Photocatalysis of N-Doped TiO_2 Films Prepared by Reactive Sputtering Using Air/Ar Mixtures, **M.-H. CHAN**, National Chung Hsing University, Taiwan, F.-H. LU, National Cheng Kung University, Taiwan	**E3-1-7** When and Why a-c:H Films are Hydrophobic?, **L.V SANTOS**, National Institute for Space Research - INPE, Brazil, V.J. TRAVA-AIROLDI, A.F. AZEVEDO, R.P.C.C. STATUTI, P.A. RADI, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil
3:50 pm	**C1-8** Low Temperature HIPIMS Deposition of AZO Coatings on Polymeric Web, **P.J. KELLY**, P. BARKER, G. WEST, Manchester Metropolitan University, United Kingdom, J.W. BRADLEY, University of Liverpool, Untied Kingdom	**E3-1-8** Friction and Wear Behavior of Hydrogenated Amorphous Diamond-Like Carbon (a-C:H) by Reactive Magnetron Sputtering in Water Environment, **B. HILKER**, H.-R. STOCK, M. DIESSELBERG, Stiftung Institut fuer Werkstofftechnik, Germany
4:10 pm	**C1-9** Fabrication of ZnO Thin Films by Atomic Layer Deposition Using Interrupted Flow-Rate Method, C.-S. KU, National Synchrotron Radiation Research Center, Taiwan, J.-M. HUANG, National Hsinchu University of Education, Taiwan, **C.-M. LIN**, National Hsinchu University of Education,, Taiwan, H.-Y. LEE, National Synchrotron Radiation Research Center, Taiwan	**E3-1-9** Water Adsorption on Phosphorus Carbide Thin Films, E. BROITMAN, Carnegie Mellon University, **A FURLAN**, G.K. GUEORGUIEV, IFM, Linköping University, Sweden, ZS CZIGÁNY, Research Institute for Technical Physics and Materials Science, Hungary, A.M. TARDITI, Universidad Nacional del Litoral, Argentina, A.J. GELLMAN, Carnegie Mellon University, S. STAFSTRÖM, IFM, Linköping University, Sweden, L. HULTMAN, Linköping University, Sweden
4:30 pm		**E3-1-10** Measured and Simulated Wear and Friction Maps of DLCH20% and DLCH35% Films, **P.A. RADI**, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil, L.V SANTOS, M.C.M FARIAS, L.F. BONETTI, V.J. TRAVA-AIROLDI
4:50 pm	Poster Session Town and Country/San Diego Rooms 5:00 - 7:00 pm	**E3-1-11** Synthesis and Characterization of Nanocomposite and Nanolaminate Multilayers by High-Power Ion Beam Ablation, **T.J. RENK**, T.E. BUCHHEIT, S. PRASAD, Sandia National Laboratories
5:10 pm	Poster Reception Town and Country/San Diego Rooms 5:30 - 7:00 pm	**E3-1-12** Wear-Resistant and Low-Friction Diamond-Like-Carbon (DLC)-Layers for the Wood Machining Industry, W. TILLMANN, Technische Univeristaet Dortmund, Germany, E. VOGLI, **F. HOFFMANN**, Technische Universitaet Dortmund, Germany

Thursday Afternoon, April 30, 2009

	Applications, Manufacturing, and Equipment **Room: Sunrise - Session G2-1** **Coatings for Automotive and Aerospace Applications** **Moderators:** G. Dadheech, General Motors, H. Rudigier, OC Oerlikon Balzers AG	**New Horizons in Coatings and Thin Films** **Room: Sunset - Session H2-2** **High Power Impulse Magnetron Sputtering** **Moderators:** A.P. Ehiasarian, Sheffield Hallam University, K. Marchev, P&G Company
1:30 pm	**G2-1-3 Invited** Atmospheric Plasma - Cleaning, Activation and Coating of Materials Surfaces by Openair® Plasma, **C. BUSKE**, Plasmatreat GmbH, Germany	**H2-2-1** Modulated Pulse Power Sputtered Chromium and Chromium Nitride Coatings, **J. LIN**, J.J. MOORE, Colorado School of Mines, W.D. SPROUL, Reactive Sputtering, Inc., B. MISHRA, Colorado School of Mines, Z.L. WU, Colorado School of Mines, and Dalian University of Technology, China, M. HASHEMINIASARI, S. MYERS, Colorado School of Mines, R. CHISTYAKOV, Zond, Inc., B. ABRAHAM, Zpulser, LLC
1:50 pm	Invited talk continued.	**H2-2-3** Physical Properties and Potential Applications of High Power Impulse Magnetron Sputtering Plus (HIPIMS+) Deposited Chromium Nitride and Titanium Nitride Coatings, F. PAPA, **C. STRONDL**, I. KOLEV, T. KRUG, R. TIETEMA, Hauzer Techno Coating BV, Netherlands
2:10 pm	**G2-1-5** Low Temperature Reactive Magnetron Sputtered nc-Ti(N,C)/a-C:H Coatings for Automotive Applications, **C. TSOTSOS**, K. POLYCHRONOPOULOU, University of Cyrpus, N. DEMAS, Univ of Illinois at Urbana-Champaign, M. BAKER, Y. CHEN, Univ of Surrey, UK, K. KANAKIS, A. LEYLAND, A. MATTHEWS, Univ of Sheffield, UK C. REBHOLZ, Univ of Cyprus, A.A. POLYCARPOU, Univ of Illinois at Urbana-Champaign	**H2-2-4** Low Friction CrN/TiN Multilayer Coatings Prepared by a Hybrid HIPIMS/UBMS Process, **J. PAULITSCH**, Materials Center Leoben Forschung GmbH, Austria, P.H. MAYRHOFER, University of Leoben, Austria, M. SCHENKEL, SVS Vacuum Coatings Technologies, Germany
2:30 pm	**G2-1-6** Gas Flow Sputtering - An Approach to Coat Complex Geometries and Non-Line-of-Sight Areas, **S. TANG**, U. SCHULZ, German Aerospace Center, Germany	**H2-2-5** Industrial-Scale Deposition of Highly Adherent CN_x Films on Steel Substrates, **E. BROITMAN**, Carnegie Mellon University, ZS CZIGÁNY, Research Institute for Technical Physics and Materials Science, Hungary, R. CREMER, CemeCon AG, Germany, X. ZHOU, SKF Engineering and Research Center, Netherlands, L. HULTMAN, Linköping University, Sweden
2:50 pm	**G2-1-7** Thick Plasma Enhanced PVD Coatings for Weapons Applications, **S.L. LEE**, M. TODARO, US Army ARDEC-Benét Labs, R. WEI, E. LANGA, Southwest Research Institute	**H2-2-6** Development of Ready to use Cr_2AlC Max-Phase on Complex Geometries, **O. SCHROETER**, A. FLORES RENTERIA, Brandenburg University of Technology at Cottbus, Germany, C. LEYENS, TU-Cottbus, Germany
3:10 pm	**G2-1-8** Enabling Lightweight, High Load Aero-Bearings, J. AVELAR-BATISTA WILSON, S. BANFIELD, Tecvac Ltd, United Kingdom, B. KARADIA, N. VAHEGLA, Airbus, United Kingdom, P. SMITH, Cranfield University, United Kingdom, G. CASSAR, A. LEYLAND, A. MATTHEWS, The University of Sheffield, United Kingdom, **J. HOUSDEN**, TECVAC Ltd, United Kingdom	**H2-2-7** Impact Behavior of (Ti,Al,Si)N Deposited by HPPMS, K. BOBZIN, **N. BAGCIVAN**, S. BOLZ, RWTH Aachen University, Germany
3:30 pm		**H2-2-8** Modulated Pulse Power Deposition of Optical Coatings, **R. CHISTYAKOV**, Zond, Inc., B. ABRAHAM, Zpulser, LLC, W.D. SPROUL, Reactive Sputtering, Inc., J.J. MOORE, J. LIN, Colorado School of Mines
3:50 pm		**H2-2-9** Arbitrary Voltage Pulse Shape Plasma Generator with RF Capabilities for Material Processing, R. CHISTYAKOV, **B. ABRAHAM**, Zond Inc.
4:10 pm		**H2-2-10** An Investigation of Magnetron Magnetic Field Strength Requirements for HIPIMS, P.J. KELLY, P. BARKER, Manchester Metropolitan University, United Kingdom, D. OCHS, Hüttinger Elektronik GmbH, Germany, A.G. SPENCER, Alacritas Consultancy Ltd., United Kingdom, **G. HINTZ,** Hüttinger Elektronik GmbH, Germany
	Poster Session Town and Country/San Diego Rooms 5:00 - 7:00 pm	Poster Reception Town and Country/San Diego Rooms 5:30 - 7:00 pm

NOTES

Thursday Afternoon Poster Sessions

Coatings for Use at High Temperature
Room: Town & Country - Session AP

Symposium A Poster Session
5:00 – 7:00 pm

AP-1

Hard Protective Si-Zr-O Coatings Resistant to Thermal Cycling in Air up to 1400ºC, J. MUSIL, **V. SATAVA**, P. ZEMAN, R. CERSTVY, University of West Bohemia, Czech Republic

AP-4

Analysis of Hf-Rich Precipitates in NiAl-Hf Bond Coats via Transmission Electron Microscopy and Atom Probe Tomography, **M.A. BESTOR**, M.S. KIRSCH, R.L. MARTENS, M.L. WEAVER, The University of Alabama

AP-5

Properties of the Thermally Stable Si-B-C-N Coatings Prepared by Reactive dc Magnetron Co-Sputtering, **P. CALTA**, J. CAPEK, P. ZEMAN, P. STEIDL, R. CERSTVY, J. VLCEK, University of West Bohemia, Czech Republic

AP-6

Growth of Intermetallic Layer in the Aluminide Mild Steel During Hot-dipping, **W.-J. CHENG**, C.-J. WANG, National Taiwan University of Science and Technology, Taiwan

AP-7

Control of Coating Thickness Ratio for Optimizing Adhesive Strength and Thermal Shock Resistance in Air-Plasma Sprayed Zirconia Based Thermal Barrier Coatings, Y.G. JUNG, **S.I. JUNG**, Y.S. SIM, J.Y. KWON, J.H. LEE, U. PAIK, Changwon National University, Korea

AP-8

Diffusion Aluminide Coatings for TiAl Intermetallic Turbine Blades, **M. GORAL**, L. SWADZBA, G.J. MOSKAL, The Silesian University of Technology, Poland, G. JARCZYK, ALD Vacuum Technologies AG, Germany

AP-9

Dry Drilling of Austempered Ductile Iron (ADI) with Different Coated Drill, **W. MATTES**, SENAI-SC, ALVES, Brazil, S. MARTINS, SOCIESC, Brazil

AP-10

The Oxidation of a Air Plasma Sprayed Thermal Barrier Coating, **P. SMITH**, R.G. WELLMAN, Cranfield University, United Kingdom, R. JONES, M. WYBROW, Rolls Royce, United Kingdom, J.R. NICHOLLS, Cranfield University, United Kingdom

AP-11

Influence of Deposition Parameters on Structure and Oxidation Resistance of Diffusion Aluminide Coatings Obtained by CVD Method on Ni-Base Superalloys, **L. SWADZBA**, B. MENDALA, M. HETMANCZYK, B. WITALA, M. GORAL, The Silesian University of Technology, Poland

AP-12

Characterization of Microstructure and Properties of Plasma Sprayed Ceramic Coatings on Mg Alloys, **A. IWANIAK**, G.J. MOSKAL, A. KIEBUS, T. RZYCHON, The Silesian University of Technology, Poland

AP-13

High Temperature Wear Behaviour of Aluminum Oxide Coatings Produced by Ac Micro Arc Oxidation, E. ARSLAN, Y. TOTIK, **Y. VANGOLU**, A. ALSARAN, I. EFEOGLU, Ataturk University, Turkey

AP-14

Thermal Barrier Coatings by Electron Beam-Hysical Vapor Deposition of Zirconia Co-Doped with Yttria and Niobia, **D.S. ALMEIDA**, C.A.A. CAIRO, Centro Tecnologico de Aeronautica, Brazil, D.A.P. REIS, Instituto Tecnologico de Aeronautica, Brazil, V. HENRIQUES, F. PIORINO NETO, Centro Tecnologico de Aeronautica, Brazil

AP-15

Effects of Granulometry on Properties of Plasma Sprayed Yttria-Stabilized Zirconia Coatings, **S. LISCANO**, L. GIL, UNEXPO, Venezuela, M.H. STAIA, Universidad Central de Venezuela, O. LEÓN, UNEXPO, Venezuela

AP-16

Effect of Hf Alloy Addition on Aluminide Coating Performance, **B.A. PINT**, K.L. MORE, J.A. HAYNES, Oak Ridge National Laboratory

Hard Coatings and Vapor Deposition Technology
Room: Town & Country - Session BP

Symposium B Poster Session
5:00- 7:00 pm

BP-1

Deposition of Superhard Nanolayered CrAlBN Thin Films by Cathodic arc Plasma Deposition, **S.K. KIM**, V.V. LE, University of Ulsan, Korea, J.W. LEE, KAIST, Korea

BP-2

A Cylindrical Form of the Hot Refractory Anode Vacuum Arc (HRAVA), **S. MUHL**, IIM-UNAM, I. CAMPS, O. PEÑA, Universidad Nacional Autonoma de Mexico, E. CAMPS, L. ESCOBAR-ALARCON, Instituto Nacional de Investigaciones Nucleares, Mexico, S.E. RODIL, Universidad Nacional Autonoma de Mexico

BP-3

TiN/Ti

Thin Film on Flexible PET Substrate Deposited by RF Magnetron Sputtering, J.H. HUANG, **J.L. LIN**, G.P. YU, National Tsing Hua University at Hsinchu, Taiwan

BP-4

Preparation, Structure and Characteristic Study of TiVCrAlN Thin Film, **D.-C. TSAI**, M.-J. DENG, F.-S. SHIEU, National Chung Hsing University, Taiwan

BP-5

Interlayer Effect on the Detaching Mechanism for Mo-Ru Hard Coatings, **Y.-I. CHEN**, National Taiwan Ocean University, Taiwan, L.-C. CHANG, Mingchi University of Technology, Taiwan, B.-N. TSAI, National Taiwan Ocean University

BP-6

Growth of α-$(Al,Cr)_2O_3$ Thin Films by Reactive r.f. Magnetron Sputtering, **D. DIECHLE**, M. STUEBER, H. LEISTE, S. ULRICH, Forschungszentrum Karlsruhe, Germany, V. SCHIER, Walter AG, Germany

BP-7

Optoelectronic and Structural Properties of ZnO:Ga Thin Films Prepared by Pulsed DC Magnetron Sputtering, **W.-T. YEN**, Y.-C. LIN, National Changhua University of Education, Taiwan

BP-8

Formation, Characterization and Properties of Al-doped Vanadium Pentoxide Nanorods by Chemical Vapor Deposition, **M.W. HUANG**, National Chung Hsing University, Taiwan, Y.C SU, L.W CHANG, National Tsing Hua University, Taiwan, F.-S. SHIEU, National Chung Hsing University, Taiwan, H.-C. SHIH, Chinese Culture University, Taiwan

BP-9

Characterization of Superlattice CrN/AlN Coating for Semiconductor Packaging Applications, D.-Y. WANG, **W.-H. TZENG**, Mingdao University, Taiwan

BP-10

Effects of Dopant Ion and Mn Valence State in the $La_{1-x}A_xMnO_3$ (A=Sr, Ba, Ca) CMR Films for Infrared Sensor, **S.G. CHOI**, A.S. REDDY, Yonsei University, Korea, H. RYU, B.-G. YU, Electronics and Telecommunication Research Institute, Korea, H.-H. PARK, Yonsei University, Korea

BP-11

Composition-Constitution-Morphology Relationship of Al_2O_3 Thin Films Deposited by Plasma Assisted Chemical Vapor Deposition, **J.M. SCHNEIDER**, K. JIANG, K. SARAKINOS, D. MUSIC, J. MAYER, RWTH Aachen University, Germany, R. SNYDERS, Université de Mons, Belgium, ST. KONSTANTINIDIS, University of Mons-Hainaut, Belgium, T. MARKUS, Forschungszentrum Jülich, Germany

BP-12

Characterization of Binary, Ternary and Quaternary Hard Coatings in the Material System V-Al-C-N Produced by Industrial Scale Reactive Magnetron Sputter Deposition, **SZ. KOLOZSVÁRI**, P. PESCH, TZO Rheinbreitbach GmbH, Germany, C. ZIEBERT, M. STUEBER, S. ULRICH, Forschungszentrum Karlsruhe, Germany

BP-14

Influence of Bias Voltage on the Microstructure and Physical Properties of Magnetron Sputtered ZrSiN Nanocomposite Thin Films, N. CUSNIR, D. OEZER, C.S. SANDU, **R. SANJINES**, A. KARIMI, EPFL, Switzerland, J. PATSCHEIDER, EMPA, Switzerland

BP-15

Wear Resistance of PVD Magnetron Sputtered ZrTiBN Thin Films, **O. JIMENEZ**, M. AUDRONIS, K. KANAKIS, A. LEYLAND, A. MATTHEWS, University of Sheffield, United Kingdom

BP-16

Synergy Between High Temperature and Wear In CrSiN Nanocomposite Coatings Deposited by Hybrid Arc /Magnetron Process, A. MÈGE-REVIL, **P. STEYER**, INSA de Lyon, France, J. FONTAINE, M. GUIBERT, Ecole Centrale de Lyon, France, J.-F. PIERSON, Ecole des Mines de Nancy, France, C. ESNOUF, INSA de Lyon, France

BP-17

Mechanical and Electrochemical Properties of CrZr-Si-N Coatings, **Y.S. KIM**, G.S. KIM, S.Y. LEE, S.C. OH, Korea Aerospace University, Korea

BP-18

Phonon Anomalies in Multiferroic $BiFeO_3$ Epitaxial Thin Films Prepared by Using Pulsed Laser Deposition, M.K. SINGH, S. DUSSAN, **G.L. SHARMA**, R. KATIYAR, University of Puerto Rico

BP-19

Characteristics of Silicon-Nitride Films Deposited by Internal Linear Inductively Coupled Plasma Source, **G.H. GWEON**, K.N. KIM, J.H. LIM, G.Y. YEOM, SungKyunKwan University, Korea

BP-21

Boron-10 Coating of Textured Semiconductor Surfaces for Neutron Detector Integrated Circuits, **C.C. KLEPPER**, O.R. MONTEIRO, R.C. HAZELTON, J.J. MOSCHELLA, J.M. WILLIAMS, E.P. CARLSON, M.D. KEITZ, HY-Tech Research Corporation

BP-22

Preparation and Mechanical Properties of Reactively Sputtered Complex $Al_xCoCrCuFeNi$ Oxide Films, T.K. CHEN, C.S. LIN, **M.S. WONG**, National Dong Hwa University, Taiwan

BP-23

Nucleation and Growth of Diamond by Pulsed Liquid Injection CVD Using Tequila as Precursor, **L.M. APATIGA**, Campus Juriquilla, Mexico, J. MORALES, Universidad Autonoma de Nuevo Leon, Mexico

BP-24

Ion Energy Distribution and Langmuir Probe Studies in an Ar/O_2 Discharge During Al_2O_3 Deposition, **S.L. MENSAH**, University of Arkansas, H.H. ABU-SAFE, Lebanese American University, Lebanon, M.H. GORDON, University of Arkansas

BP-25

Characterization and Mechanical Properties of Magnetron Sputtered Iron Nitride Coatings, H.-R. STOCK, **I. EISBRECHER**, H. TEUBER, Stiftung Institut fuer Werkstofftechnik, Germany

BP-26

Multilayer Chromium Based Coatings Grown by Direct Liquid Injection CVD, **F. MAURY**, ENSIACET, France, A. DOUARD, S. DELCLOS, D. SAMELOR, C TENDERO, CIRIMAT, France

BP-27

Characteristics and Homogeneity of Direct and Pulse Current Electrodeposited High Phosphorus Content Ni-P Coatings, **C.C. WU**, H.S. HUANG, K.J. CHANG, F.B. WU, National United University, Taiwan

BP-28

Effect of Pulse Plating with Ultrasonic Agitation on the Corrosion and Wear Properties of Eco-friendly Trivalent Chromium Layers Prepared in Chromium Sulfate Bath, S.C. KWON, Korea Institute of Materials Science, Korea, **J.J. LEE**, Y. CHOI, Sunmoon University, Korea

BP-29

BGA Cutter Improvement Utilizing Nano-TiAlN Coating Layers Synthesized by Cathodic Arc Ion Plating Process, **S.H. HUANG**, National Chiao Tung University, Taiwan, T.-E. HSIEH, National Chiao Tung Univesity, Taiwan, C.-W. CHEN, Gigastorage Corporation, Taiwan

BP-30

Mechanical Properties and Oxidation Resistance of (Cr,Al)N Based Films Synthesized by Radio-Frequency Magnetron Sputtering Method, **H. HASEGAWA**, T. MIYAKE, Okayama University, Japan, S. KUNITSUGU, Industrial Technology Center of Okayama Prefecture, Japan, K. OHASHI, S. TSUKAMOTO, Okayama University, Japan

BP-31

Tribological Characterisation of TiN_x Coatings Synthesized by Cathodic ARC Evaporation Technique, **J. MENGHANI**, K.B. PAI, M.K. TOTLANI, SVNIT Surat, India, N. JALGOANKAR, Multi-Arc India Ltd., India

BP-32

NiTi Memory Alloy Sculptured Thin Film by Glancing Angle Electron Beam Evaporation Technique, **K. KAZMANLI**, L. TRABZON, M. URGEN, G. GURLUK, Istanbul Technical University, Turkey

BP-33

High Temperature Oxidation Resistance of Multicomponent Cr-Ti-Al-Si-N Coatings, **C.-Y. HSIAO**, Y.-Y. CHANG, D.-Y. WANG, Mingdao University, Taiwan, W. WU, National Chung Hsing University, Taiwan

BP-34

Optical, Morphological and Electrochemical Properties of Niobium Oxide Thin Films, **G. RAMÍREZ**, S.E. RODIL, S. MUHL, Universidad Nacional Autónoma de México, J.J. OLAYA, Universidad Nacional de Colombia, M. RIVERA, Universidd Nacional Autónoma de México, E. CAMPS, L. ESCOBAR-ALARCON, Instituto Nacional de Investigaciones Nucleares, México

BP-36

Effects of Low-Temperature Duplex Coatings on Corrosion Behavior of Austempered Ductile Iron, **C.-H. HSU**, C.-Y. LEE, K.-L. CHEN, K.-C. LU, Tatung University, Taiwan

BP-37

Thermal Evolution, Mechanical and Corrosion Properties of Al Implantation into TiSiN Nanocomposite Coatings by MPII Hybrid System, C.-L. CHANG, **L.-Y. TSENG**, C.-W. WU, Y.-C. LIU, W.-Y. LIN, MingDao University, Taiwan

BP-38

Using High Work Function Ni Metal to Improve the Stress Reliability of $CaCu_3Ti_4O_{12}$ MIM Capacitors, **L.-C. CHANG**, Mingchi University of Technology, Taiwan, C.-H. YANG, H.-L. KAO, Chang Gung University, Taiwan, F.B. WU, National United University, Taiwan

BP-40

Phase Transformation, Thermal Stability and Indentation Behavior of Ni-P-Based Interlayer Enhanced CrN Composite Coatings, Y.Y. LI, Y.C. HSIAO, J.C. WU, **F.B. WU**, National United University, Taiwan

BP-41

Superhard Coatings Prepared by Pulsed Magnetron Sputtering, **M. KEUNECKE**, K. WEIGEL, K. BEWILOGUA, Fraunhofer Institute for Surface Engineering and Thin Films, Germany, W. KÖLKER, P. JASCHINSKI, Cemecon AG, Germany

BP-43

Evaluation of the Electrochemical Behaviour of Tantalum Oxide Thin Films for Biomedical Applications, **P.N. ROJAS**, S. MUHL, S.E. RODIL, Universidad Nacional Autónoma de México

BP-44

Microstructure and Properties of Laser-Cladded Cr-Ni Coatings Prepared on Steel, A. IWANIAK, **E. AUGUSTYN**, J. ADAMIEC, The Silesian University of Technology, Poland

BP-45

Development of Temperature Sensor Thin Films to Monitor Turning Processes, W. TILLMANN, **E. VOGLI**, TU Dortmund University, Germany, K. PANTKE, D BIERMANN, Institute of Machining Technology, Germany

BP-46

Why a TiN Coating is not Suitable for Corrosion Protection of Steel, J.P.M. GROENEWEGEN, **R. MACHUNZE**, G.C.A.M. JANSSEN, TU Delft, Netherlands

BP-47

Raman Spectroscopy as a Tool to Study Composition in Cubic Ti-C-N CVD Coatings, **I. DREILING**, A. HAUG, University of Tuebingen, Germany, H. HOLZSCHUH, Walter AG, Germany, T. CHASSE, University of Tuebingen, Germany

Optical Thin Films
Room: Town & Country - Session CP

Symposium C Poster Session
5:00 – 7:00 pm

CP-1

Preparation of TiO_2 Photoelectrode by Spray Pyrolysis Technique for the Photovoltaic Application, M.F. HOSSAIN, S. BISWAS, M. SHAHJAHAN, **T. TAKAHASHI**, University of Toyama, Japan

CP-2

Effects of Oxygen Flow Ratios and Annealing Temperatures on Raman and Photoluminescence of Titanium Oxide Thin Films Deposited by Reactive Magnetron Sputtering, C.K. CHUNG, M.W. LIAO, **C.W. LAI**, National Cheng Kung University, Taiwan

CP-3

Transparent Thermally Stable Poly(etherimide) Film as Flexible Substrate for OLEDs, C. LEGNANI, W.G. QUIRINO, CeDO - Inmetro, Brazil, **M. CREMONA**, PUC-Rio, Brazil, V.L. CALIL, CeDO - Inmetro, Brazil, G.F. MOREIRA, C.A. ACHETE, C. VILANI, Dimat - Inmetro, Brazil

CP-4

Holographic Grating Formation in PVB Doped Polymer Dispersed Lilquid Crystal Based on PUA, **E.H. KIM**, Y.G. JUNG, U. PAIK, Changwon National University, Korea

CP-5

Study of Structural and Optical Properties of TiO_2:Tb Coatings Prepared by High Energy Reactive Magnetron Sputtering, D. KACZMAREK, **J. DOMARADZKI**, Wroclaw University of Technology, Poland, Z.J. RADZIMSKI, Silicon Quest International

CP-6

The Carbon Effects on the Phase Transformation of Visible-Light Responsive Carbon Containing TiO_2 Nanoparticles, **Y.-H. CAI**, S.-J. CAI, V. YEH, S.-B. WU, C.-L. CHENG, National Dong Hwa University, Taiwan

CP-7

Characteristics of Indium Zinc Tin Oxide Thin Film Prepared on Flexible Substrates Using the Hetero-Target Sputtering System, **D.H. KIM**, Y.S. RIM, H.-W. CHOI, K.-H. KIM, Kyungwon University, Korea

CP-8

Effect of Thermal Acceleration on the Degradation of Gallium doped Zinc Oxide Thin Film, **J. KANG**, M. LEE, H. CHOI, W. SEO, Korea Institute of Ceramic Engineering and Technology, Korea, D.Y. LEE, Daelim College of Technology, Korea

CP-9

Linear and Nonlinear Optical Properties of CdSe/PMMA Nanocomposite Filters with Mechanical Property Improvement, G. CHEN, University of Arkansas, Y.A. WANG, Ocean NanoTech, LLC., M. XIAO, **M. ZOU**, University of Arkansas

CP-10

Influence of Sputtering Gas on the Properties of Reactively Sputtered Tungsten Oxide Films, A.K. CHAWLA, S. SINGHAL, **R. CHANDRA**, H.O. GUPTA, Indian Institute of Technology Roorkee, India

CP-11

Modulation of Luminescence Emission Spectra of N-Doped Ga_2O_3 Nanowires by Thermal Evaporation, H.-C. SHIH, **L.-W. CHANG**, National Tsing Hua University, Taiwan, M.W. HUANG, C.-F. LI, National Chung Hsing University, Taiwan, J.-W. YEH, National Tsing Hua University, Taiwan

CP-13

Characterization of Anomalous Luminescence Properties from Self-Ordered Porous Anodic Alumina with Oxalic Acid Electrolytes, **Y.-R. CHEN**, C.-H. FANG, National United University, Taiwan, J.-C. WANG, T.-E. NEE, Chang Gung University, Taiwan

CP-14

Enhancement of Light Reflectance and Thermal Stability in Ag-Mg Alloy Contacts on p-Type GaN, **Y.H. SONG**, G.H. JUNG, J.H. SON, J.-L. LEE, Pohang University of Science and Technology (POSTECH), Korea

CP-15

Electrochromic Tungsten-Titanium Oxide Films Deposited by Co-Sputtering using a Pulsed Sputtering Deposition System, **K.-W. WENG**, Mingdao University, Taiwan, S. HAN, National Taichung Institute of Technology, Taiwan, Y.-C. CHEN, National Chung Hsing University, Taiwan

CP-16

Deposition and Post-Deposition Treatment for Obtaining Sco on Si and Quartz Substrate, J.L. DESCHANVRES, **A. KHAN**, H. ROUSSEL, C. JIMENEZ, INP Grenoble-Minatec, France, B. SERVET, Thales Research & Technology, France, M. MODREANU, Tyndall National Institute, Ireland

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Town & Country - Session DP

Symposium D Poster Session
5:00 – 7:00 pm

DP-1

Silver-Carbon Nanoparticles Produced by High-Current Pulsed Arc, **F. MAYA**, M. MIKI-YOSHIDA, Centro de Investigación en Materiales Avanzados (CIMAV), México, S. MUHL, O. PEÑA, Universidad Nacional Autónoma de México

DP-2

Optical and Electron Field Emission Properties of Silver Doped Diamond Like Carbon Films Deposited by RF Reactive Sputtering Technique, **SK.F. AHMED**, M.W. MOON, K.R. LEE, Korea Institute of Science and Technology, Korea

DP-3

Temperature Programmed Desorption and Spectroscopic Studies of Surface Functionalized Nanodiamond Particles Prepared for Bio-Applications, **S.-J. CAI**, Y.-C. CHIU, V. YEH, C.P. CHEN, C.-L. CHENG, National Dong Hwa University, Taiwan

DP-4

Preparation and Evaluation of the Porous Graphite/Carbon Composites for Lithium-ion Battery with High Rate Charging, **M.L. LEE**, National Tsing Hua University, Taiwan, J.M. CHEN, Industrial Technology Research Institute, Taiwan, J.-W. YEH, H.-C. SHIH, National Tsing Hua University, Taiwan

DP-5

Wetting Behavior of a Droplet on Dual Rough Surfaces Coated with Hydrophobic and Hydrophilic DLC Films, **T. CHA**, J.W. YI, M.W. MOON, Korea Institute of Science and Technology, Korea, H.Y. KIM, Seoul National University, Korea, K.R. LEE, Korea Institute of Science and Technology, Korea

DP-6

Structure-Property Relationships of Galvanic Nickel Coatings Codeposited with Nano Diamond Particles, **O. SHENDEROVA**, International Technology Center, D.L. SCHULZ, R.A. SAILER, North Datota State University, G.E. MCGUIRE, W. MECOUCH, International Technology Center

DP-7

Metal-Containing Diamond Like Carbon (DLC:Me) and AlN (AlN:Me) Metallo-Dielectric Nanocomposites, G.M. MATENOGLOU, H. ZOUMBOS, University of Ioannina, Greece, A. LOTSARI, Aristotle University of Thessaloniki, Greece, CH.E. LEKKA, D.F. ANAGNOSTOPOULOS, University of Ioannina, Greece, PH. KOMNINOU, Aristotle University of Thessaloniki, Greece, G.A. EVANGELAKIS, **P. PATSALAS**, University of Ioannina, Greece

DP-9

Effect of Oxygen Plasma Treatment on Bonding States for Columnar Structured a-CN_x Thin Films Prepared by a Reactive Sputtering, **M. AONO**, S. KIKUCHI, N. KITAZAWA, Y. WATANABE, National Defense Academy, Japan

DP-10

DLC Coated Spinal Disks with Predictable Long Term Adhesion, **R. HAUERT**, C.V. FALUB, Empa, Switzerland, G. THORWARTH, Swiss Federal Institute for Materials Testing and Research (EMPA), B. WEISSE, U. MÜLLER, Empa, Switzerland, M. PARLINSKA-WOJTAN, Swiss Federal Institute for Materials Testing and Research (EMPA), C. VOISARD, Synthes GmbH, Switzerland, M. TOBLER, IonBond AG, Switzerland

DP-11

Structural and Optical Properties of Ultrananocrystalline Diamond / InGaAs/GaAs Quantum Dot Structures, **C. POPOV**, A. GUSHTEROV, L. LINGYS, C. SIPPEL, J.P. REITHMAIER, University of Kassel, Germany

DP-13

Deposition of Amorphous Carbon-Silver Composites, O. GARCIA-ZARCO, Z. MONTIEL, **S.E. RODIL**, Universidad Nacional Autónoma de México, M. CAMACHO-LÓPEZ, Universidad Autónoma del Estado de México

DP-14

Enhancement of Deposition Rate and Droplet Reduction in T-Shape Filtered Arc Deposition System for DLC Preparation, **M. KAMIYA**, T. YANAGITA, H. TANOUE, S. OKE, Y. SUDA, H. TAKIKAWA, Toyohashi University of Technology, Japan, M. TAKI, Y. HASEGAWA, Onward Ceramic Coating Co., Ltd., Japan, T. ISHIKAWA, Hitachi Tool Engineering, Ltd., Japan, H. YASUI, Industrial Research Institute of Ishikawa, Japan

DP-15

Ashing of DLC Film by Oxygen Plasma Beam Converted from Filtered Carbon-Cathodic-Arc, **H. TANOUE**, M. KAMIYA, S. OKE, Y. SUDA, H. TAKIKAWA, Toyohashi University of Technology, Japan, M. TAKI, Y. HASEGAWA, Onward Ceramic Coating Co., Ltd., Japan, T. ISHIKAWA, Hitachi Tool Engineering, Ltd., Japan, H. YASUI, Industrial Research Institute of Ishikawa, Japan

DP-16

Characterization of High sp^3 Diamond-Like Carbon Films Synthesized by Cathodic Arc Activated Deposition Process, **W.-C. LIN**, D.-Y. WANG, Mingdao University, Taiwan

DP-17

On Mechanism of Self-Arrangement of Nanosized Diamond Particles Under Sintering, **O.O. BOCHECHKA**, G.S. OLEYNIK, A.V. KOTKO, National Academy of Sciences, Ukraine

DP-19

A New Globular Nanocarbon Material and Production Fire Method Therof, V.G. SUSCHEV, **V.A. MARCHUKOV**, V. Y. DOLMATOV, FSUP SCTB Technolog, Russia

DP-20

New Approaches to Industrial Production of Detonation Nanodiamonds, **V. Y. DOLMATOV**, V.A. MARCHUKOV, M.V. VERETENNIKOVA, FSUP SCTB Technolog, Russia

DP-24

Development of Si-Doped Hydrogenated Amorphous Diamond-Like Carbon (a-C:H:Si) for Tribological Applications in Humid Environment, **H.-R. STOCK**, B. HILKER, Stiftung Institut fuer Werkstofftechnik, Germany

DP-25

Annealing Effect on the Structural, Mechanical and Electrical Properties of Titanium-Doped Diamond-Like Carbon Thin Films, **Y.-H. LIN**, H.-D. LIN, C.-K. LIU, National Tsing Hua University, Taiwan, Y.-C. CHEN, National Chung Hsing University, Taiwan, Y.-S. CHANG, H.-C. SHIH, National Tsing Hua University, Taiwan

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: Town & Country - Session EP

Symposium E Poster Session
5:00 – 7:00 pm

EP-1

The Oliver and Pharr Method for Coatings and Physical Scratch Test Analysis for Layered Materials, **M. FUCHS**, N. SCHWARZER, Saxonian Institute of Surface Mechanics, Germany

EP-2

The Stucture and Properties of Nano-Crystalline ZrTiN Thin Films : Effect of Ratio of Ti/Zr, **Y.-W. LIN**, J.H. HUANG, G.P. YU, National Tsing Hua University, Taiwan

EP-3

Effect of Surface Polishing on the Abrasion Performance of a Chromium Carbide-Based Coating Deposited by VPS, **Z. MARCANO**, Universidad Central de Venezuela, J. LESAGE, University of Lille, France, E.S. PUCHI-CABRERA, M.H. STAIA, Universidad Central de Venezuela

EP-5

A Study of the Reciprocating Sliding Wear Performance of Plasma Surface Treated Titanium Alloy, **G. CASSAR**, University of Sheffield, United Kingdom, J.C.A. BATISTA-WILSON, S. BANFIELD, Tecvac Ltd, J. HOUSDEN, Tecvac Ltd., A. LEYLAND, A. MATTHEWS, University of Sheffield, United Kingdom

EP-6

In-Situ Raman Tribo-Spectrometry Technique for High Temperature Sliding Contacts, **J. BULTMAN**, UDRI/Air Force Research Laboratory, C. MURATORE, Air Force Research Laboratory/UTC, Inc., A. SAFRIET, UDRI/Air Force Research Laboratory, A.A. VOEVODIN, Air Force Research Laboratory

EP-9

Solid Particle Erosion Performance of HVOF WC-Co and WC-Co-Cr Coatings Deposited onto SAE 1045 Steel, **Y.Y. SANTANA**, J.G. LA BARBERA-SOSA, A. BENCOMO, E.S. PUCHI-CABRERA, M.H. STAIA, Universidad Central de Venezuela

EP-12

Impact Properties of TiN, TiAlN and TiSiN PVD Coatings at Ambient and Elevated Temperatures, **K.-D. BOUZAKIS**, M. PAPPA, S. GERARDIS, G. KATIRTZOGLOU, S. MAKRIMALLAKIS, G. SKORDARIS, Aristoteles University of Thessaloniki, Greece, R. M'SAOUBI, Seco Tools AB

EP-13

Production of a Multicomponential Layers on a 99.8% Purity Iron by the Two State Boro-Nitriding Process: Microstructural and Mechanical Characterization, **I. CAMPOS**, O.A. GÓMEZ-VARGAS, Instituto Politécnico Nacional, México, U. FIGUEROA-LÓPEZ, Tecnológico de Monterrey, México, M. ORTIZ-DOMÍNGUEZ, Instituto Politécnico Nacional, México

EP-14

Effect of Spraying Distance on the Microstructure and Mechanical Properties of a Colmonoy 88 Alloy deposited by HVOF Thermal Spraying, **J.G. LA BARBERA-SOSA**, Y.Y. SANTANA, Universidad Central de Venezuela, N. CUADRADO, J. CARO, CTM Centre Tecnològic, Spain, P.O. RENAULT, E. LE BOURHIS, Université de Poitiers, France, M.H. STAIA, E.S. PUCHI-CABRERA, Universidad Central de Venezuela

EP-15

Effect of Damage Accumulation in Co-Depletion Layer on the Fatigue Debonding Behavior of Diamond Coated WC-Co, **S. KAMIYA**, A. UEDA, Nagoya Institute of Technology, Japan, H. HANYU, OSG Corporation, Japan, J.C. MADALENO, J. GRACIO, University of Aveiro, Portugal

EP-16

Effect of Microstructure on the Mechanical Properties of In-Situ Deposited Carbon Monolayer on the Si(100) at High Temperature Under Ultra High Vacuum, C.K. CHUNG, **S.T. HUNG**, C.W. LAI, National Cheng Kung University, Taiwan

EP-17

Effects of Si Addition on the Mechanical Properties and Cutting Performance of Nanocrystalline Cr-Si-C-N Coatings Prepared by a Hybrid Coating System, **M.C. KANG**, J.H. JEON, K.H. KIM, J.S. KIM, Pusan National University, Korea

EP-19

Mechanical and Tribological Properties of Graded Ti(BN-MoS_2) Based PVD Coatings, **I. EFEOGLU**, A. ÇELIK, A. ALSARAN, I. KAYMAZ, F. YETIM, Ataturk University, Turkey

EP-20

Effect of the Target Shuttering on the Characteristics of the Ta-Si-N Thin Films by Reactive Magnetron Co-sputtering, C.K. CHUNG, **T.S. CHEN**, N.W. CHANG, S.T. HUNG, National Cheng Kung University, Taiwan

EP-21

Mechanical Properties Characterization of Multilayered Nano Thin Films by Atomistic Simulations, **J.-C. HUANG**, Tungnan University, Taiwan

EP-22

Phase Analysis of Alumina Coating by X-Ray Diffraction (XRD), Transmission Electron Microscope (TEM), Secondary Electron Microscopy (SEM) and Micro Indentation, **W. ENGELHART**, Walter AG, Germany, W. DREHER, NMI Natural and Medical Sciences Institute, Germany, V. SCHIER, Walter AG, Germany, O. EIBL, Eberhard Karls University Tübingen, Germany

EP-23

Galling and Wear Characteristics of Some Commercial PVD Coatings as Evaluated by Tribological Testing, **J. ERICSSON**, M. OLSSON, Dalarna University, Sweden

EP-24

Tribological Properties of Cr_2N Ceramic Films with Tungsten Dopants, **C.S. WU**, National Kaoshiung University, Taiwan, C.W. CHU, University of I-Shou, Taiwan

EP-25

Microabrasion Wear Testing of PEO Coatings on 2024 Aluminium Alloy, A. PILKINGTON, H.X. CHENG, **A. YEROKHIN**, A. MATTHEWS, University of Sheffield, United Kingdom

EP-26

Influence of Nitrogen Ion Implantation on Hard Coating Layer to Improve Adhesion Strength Using Combined Surface Modification, **G.C. JEONG**, Korea Polytechnic University, Korea, Y.H. SOHN, University of Central Florida, S. KWUN, Korea University, Korea

EP-27

Ti(C,O,N)-Based Coatings for Biomedical Applications: Influence of Composition and Structure om the Mechanical/Tribological and Biological Behaviour, N. JORDÃO, S. RIBEIRO, Universidade do Minho, Portugal, **J.C. SANCHEZ-LOPEZ**, M.D. ABAD, Instituto de Ciencia de Materiales de Sevilla, Spain, F. VAZ, M. HENRIQUES, R. OLIVEIRA, Universidade do Minho, Portugal, R. ESCOBAR-GALINDO, Instituto de Ciencia de Materiales de Madrid (ICMM -CSIC), Spain, S. CARVALHO, Universidade do Minho, Portugal

EP-28

Tribological Behaviour of PT and PtN_x Coatings Deposited by Magnetron Sputtering, **M. FLORES**, E. RODRIGUEZ, J. GARCIA, Universidad de Guadalajara, Mexico

Thursday Afternoon Poster Sessions

Characterization: Linking Synthesis Properties and Microstructure
Room: Town & Country - Session FP

Symposium F Poster Session
5:00 – 7:00 pm

FP-1

DC Electric Field Induced Second Harmonic Generation Studies of Low Dielectric Constant SiOC(-H) Thin Films, R. NAVAMATHAVAN, Chonbuk National University, Korea, C. Y. KIM, H. S. LEE, J.-K. WOO, **C.K. CHOI**, Cheju National University, Korea, S.H. SEO, H.Y. CHANG, Korea Advanced Institute of Science and Technology, Korea

FP-2

Light Out-Coupling Properties of Organic Light-Emitting Diodes with the Ba-doped Alq3 Layer and Degradation Mechanism of the Devices Based on its Electronic Structure, **J.T. LIM**, G.Y. YEOM, Sungkyunkwan University, Taiwan

FP-3

Effect of P Additions on the Thermal Stability and Electrical Characteristics of NiSi, **H.-F. HSU**, C.-L. TSAI, H.-Y. CHAN, T.-H. CHEN, National Chung Hsing University, Taiwan

FP-4

Performance of Various Duplex Systems Based on DLC and Salt Bath Nitriding Processes of 316L Stainless Steel, **L. GIL**, S. LISCANO, UNEXPO, Venezuela, C. GRUESCU, D. CHICOT, University of Science and Technology of Lille, France, E.S. PUCHI-CABRERA, M.H. STAIA, Universidad Central de Venezuela

FP-6

Microstructure and Nanostructure of PVD ZrN-Cu Thin Films, M. AUDRONIS, O. JIMENEZ, A. LEYLAND, A. MATTHEWS, The University of Sheffield, United Kingdom, **C. TSOTSOS**, University of Cyrpus

FP-7

Bias Efects on the Wear Behavior of AISI 304 Stainless Steel Arc-Deposited CrTiAlN Multilayer Coatings, C.-H. HSU, **K.-L. CHEN**, Z.H. LIN, Tatung University, Taiwan, C.-K. LIN, Feng Chia University, Taiwan

FP-8

Microstructure Analysis of TiO_2 Thin Film Modified by Metal Plasma Ion Implantation for Enhanced Photosensitivity, D.-Y. WANG, Mingdao University, Taiwan, **C.-C. YEN**, National Chung Hsing University, Taiwan, M.-H. SHIH, Mingdao University, Taiwan, L.S. CHANG, H.-C. SHIH, National Chung Hsing University, Taiwan

FP-9

Analysis of ZrN/Zr Thin Film on Flexible Polyethylene Terephthalate (PET) Substrate Deposited by Unbalanced Magnetron Sputtering, **H.-M. CHU**, J.H. HUANG, G.P. YU, National Tsing Hua University, Taiwan

FP-10

Dynamic Deformation and Durability Evaluation of Various Lubricant Coated Magnetic Disks, **W. KUROSAKA**, K. OSHIMOTO, S. MIYAKE, Nippon Institute of Technology, Japan

FP-11

Interface Evolution of Annealed SiO_2 and Ta/SiO_2 Encapsulated Cu Films, **A.P. WARREN**, T. SUN, University of Central Florida, K. BARMAK, Carnegie Mellon University, M.F. TONEY, Stanford Synchrotron Radiation Laboratory, K.R. COFFEY, University of Central Florida

FP-14

Control of Hard Protective ZrN Coatings by Modulated IR Radiometry, J. GIBKES, Ruhr-University Bochum, Germany, **F. VAZ**, A.C. FERNANDES, F. MACEDO, Minho University, Portugal, K. PUCHONG, J. PELZL, B.K. BEIN, Ruhr-University Bochum, Germany

Applications, Manufacturing, and Equipment
Room: Town & Country - Session GP

Symposium G Poster Session
5:00 – 7:00 pm

GP-1

Nanocomposite Coatings and Triple Coatings on High Performance Tools with Dedicated Edge Preparation, **A. LÜMKEMANN**, M. MORSTEIN, T. CSELLE, O. CODDET, PLATIT AG, Switzerland, V. HAJEK, M. JILEK, Pivot a.s., Czech Republic, P. KARVANKOVA, PLATIT AG, Switzerland

GP-2

Deposition of SiO_2 Thin Films with Dielectric Barrier Discharge at Atmospheric Pressure PECVD using PDMS/O_2/He/Ar, **Y.S. KIM**, J.H. LEE, G.Y. YEOM, Sungkyunkwan University, Korea

GP-3

Cutting Performance of Cr-Al-N and Cr-Al-Mo-N Coated End-Mill Deposited by Hybrid Coating, **H.S. TAK**, Pusan National University, Korea, S.H. KWON, KAIST, Korea, K.H. KIM, M.C. KANG, Pusan National University, Korea

GP-4

Effect of Ferromagnetic Module on Microcrystalline Silicon Thin Films Deposited by Internal-ICP, **H.-C. LEE**, M.H. JEON, K.N. KIM, J.H. LIM, G.Y. YEOM, Sungkyunkwan University, Korea

GP-5

Line-Type Internal Linear Inductively Coupled Plasma Source for Large Area Roll-to-Roll Plasma Processing, **J.H. LIM**, K.N. KIM, G.H. GWEON, G.Y. YEOM, SungKyunKwan University, Korea

GP-6

Through-Thickness Microstructural Characterization of the Plasma Electrolytic Oxidized Titanium Oxide Fabricated on Metal Titanium, **P.-J. CHU**, A. YEROKHIN, A. LEYLAND, A. MATTHEWS, University of Sheffield, United Kingdom, J.-L. HE, Feng Chia University, Taiwan

GP-7

Electron Temperature Cooling Down with Multi-Step Ionizations in an Electron Beam Generated Plasma, H.Y. CHANG, **S.H. CHAE**, Korea Advanced Institute of Science and Technology (KAIST), Korea

GP-8

Structure and Properties of Low Temperature Plasma Carburized Austenitic Stainless Steels, R.M. SOUZA, University of São Paulo, Brazil, M. IGNAT, SIMAP Grenoble INP, France, C.E. PINEDO, **A.P. TSCHIPTSCHIN**, University of São Paulo, Brazil

GP-10

Manganese Oxide Thin Films Electrodeposited Using a Potentiodynamic Method– Effects of Potential Sweep Rates on the Material Characteristics and the Pseudocapacitive Performance, M.T. LEE, J.K. CHANG, Y.T. HSIEH, W.T. TSAI, National Cheng Kung University, Taiwan, **C.-K. LIN**, Feng Chia University, Taiwan

GP-11

SiO_x Protective and Barrier Coatings by Atmospheric Pressure Plasma Jet Deposition, **P. SCOPECE**, I. KULYK, R. SULCIS, F. MARINELLO, A. PATELLI, CIVEN Association Venice, Italy

GP-12

Multi-Layer Coatings of Aluminum for Corrosion Resistance and Electromagnetic Shielding, J.-H. CHEN, Y.-J. FANG, H.-T. HSU, **T.-J. YANG**, Feng Chia University, Taiwan

GP-13

Characterization and Photocatalytic Activity of Composite V-TiO_2/ITO Thin-Film Electrode, C.Y. CHANG, Mingdao University, Taiwan, **M.Y. CHANG**, National Chung-Hsing University, Taiwan, Y.H. HSIEH, Mingdao University, Taiwan, Y.S. WANG, National Dong Hwa University, Taiwan

GP-14

Plasma Processing Efficiency in Pulse Plasma System, **M. ZLATANOVIC**, I. POPOVIC, Faculty of Electrical Engineering, Serbia

GP-15

Effects of Pressure on Physical Properties of Amorphous Carbon Film, **K. KAYAMA**, M. WATANABE, Keio University, Japan, H. KODAMA, Kanagawa Academy of Science and Technology, Japan, T. SUZUKI, Keio University, Japan

GP-16

Control of Plasma Non-Uniformity in Large Area / Very High Frequency Capacitive Discharges, **S.K. AHN**, B.K. NA, H.Y. CHANG, Korea Advanced Institute of Science and Technology, Korea

GP-17

Photoelectrocatalytic Degradation of Sodium Oxalate by TiO_2/Ti Thin-Film Electrode, T.C. CHENG, Mingdao University, Taiwan, Y.H. HSIEN, National Chung-Hsing University, Taiwan, K.S. YAO, Y.Y. CHEN, **Y.C. YEN**, C.Y. CHANG, Mingdao University, Taiwan

GP-18

Effect of Additives and pH in H_2O_2-Based Slurry on Cu-CMP Corrosion Behavior, **C.-C. HUNG**, W.-H. LEE, S.-C. CHANG, National Cheng Kung University, Taiwan, Y.-D. JUANG, Y.-L. WANG, National University of Tainan, Taiwan

GP-19

Effect of Plasma Nitriding Pre-Treatment on D2 Steel Surface Deformation Under Nitride Based Multilayer AIP Coating, **T. OKUDE**, D. YASUNAGA, K. YAMAMOTO, Kobe Steel Ltd., Japan, T. KASHI, K. IMAI, Koshuha All Metal Service CO., Ltd., Japan, T. TAKAZAWA, J. YOSHIDA, Nippon Koshuha Co,. Ltd., Japan

GP-21

Characterization of Cuprous Oxide Films by Using Atmospheric Pressure Nitrogen Plasma Torch, H.-Y. CHEN, **M.-W. TSAI**, C.-H. TSAI, National Kaohsiung University of Applied Sciences, Taiwan

GP-22

Mechanical Properties Evaluation of Chromized Tungsten Carbide-Cobalt Hardmetals, **J.-L. LI**, J.-W. LEE, Y.-T. LIN, C.-J. WONG, Tungnan University, Taiwan

GP-24

Synthesis of SiO_x Films on Polycarbonate Substrates under Atmospheric Pressure, **M. NOBORISAKA**, Keio University, Japan, H. KODAMA, Kanagawa Academy of Science and Technology, Japan, T. SUZUKI, Keio University, Japan

GP-26

Low Temperature Treatments for Improving Sputtered ZrO_x Dielectric by High Pressure O_2 and O_3 Passivation, **S.-C. CHEN**, T.-C. CHANG, H.-H. SU, C.-C. HUANG, P.-C. YANG, H.-C. HUANG, D.-S. GAN, N.-J. HO, National Sun Yat-Sen University, Taiwan

GP-28

Effect of Low-Temperature Supercritical Fluid Technology Treatment on the Performance of ZnO TFTs, **M.C. CHEN**, T.-C. CHANG, National Sun Yat-Sen University, Taiwan, G.-W. JHANG, Y.-H. TAI, National Chiao Tung University, Taiwan

GP-29

Surface Modification with Borane Clusters: Superior Protection of Metal Surfaces Against Corrosion, **T. BASE**, M.G.S. LONDESBOROUGH, J. BOULD, Institute of Inorganic Chemistry of the Academy of Sciences of the Czech Republic

New Horizons in Coatings and Thin Films
Room: Town & Country - Session HP

Symposium H Poster Session
5:00 – 7:00 pm

HP-1

Impact of Strain Engineering on Nanoscale Strained Si NMOSFETs with a Silicon-Carbon Alloy Stressor, **W.-C. WANG**, National Chung Hsing University, Taiwan, C.-C. LEE, Taiwan Semiconductor Manufacturing Company, Taiwan, J. HUANG, S.-T. CHANG, National Chung Hsing University, Taiwan

HP-2

Growth and Characterization of ZnO Nanoflowers, **J.H. PARK**, S. PRIKHODKO, M. POZUELO, University of California - Los Angeles, S.D. SITZMAN, Oxford Instruments America, S. KODAMBAKA, University of California - Los Angeles

HP-4

Reliability Study of Through-Silicon via (TSV) Copper Filled Interconnects, **A. KAMTO**, The University of Alabama, Y. LIU, L. SCHAPER, University of Arkansas, S.L. BURKETT, The University of Alabama

HP-5

Properties of Pure and Silver Doped Ti_3SiC_2 Films Deposited by HiPIMS, R. BANDORF, M. SCHMIDT, H. GERDES, Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany, **G. MARK**, MELEC GmbH, Germany

HP-6

Effects of Alloying Elements(Zr,Hf) on the Nanopore and Nanotube Formation of Ti-30(Nb,Ta) Alloys, **H.-C. CHOE**, Y.-M. KO, Chosun University, Korea, W.A. BRANTLEY, Ohio State University

HP-8

Nano-Structural Titanium Dental Implants Synthesized by Sand-blasting Etching and Plasma Etching Techniques, Y.-Y. CHANG, **Y.-T. HSU**, Y.-C. YANG, H.-I. KAO, C.-P. LAI, Mingdao University, Taiwan

HP-10

Effect of Seed-Layer Films on the Growth of ZnO Nanowires via Hydrothermal Technique, **S.-N. BAI**, Chienkuo Technology University, Taiwan

HP-13

Resistive Switching Characteristics and Mechanism of $SrZrO_3$ Thin Films, **M.-H. LIN**, M.-C. WU, S.-W. JAN, Y.-H. HUANG, National Chiao Tung University, Taiwan, C.-H. LIN, Winbond Electronics Corporation, Taiwan, T.-Y. TSENG, National Chiao Tung University, Taiwan

HP-14

Surface Morphology Changes of Anodized Ti-x(Nb,Ta) Binary Alloys for Dental Implant, **K. LEE**, H.-C. CHOE, Y.-M. KO, Chosun University, Korea, W.A. BRANTLEY, Ohio State University

HP-15

Nonvolatile Memory Effect of W Nanocrystals Thin Film Under Various Nitride-Base Plasma Treatments, **S.-C. CHEN**, T.-C. CHANG, National Sun Yat-Sen University, Taiwan, W.-R. CHEN, National Chiao Tung University, Taiwan, Y.-C. LO, K.-T. WU, National Tsing Hua University, Taiwan, S.M. SZE, National Chiao Tung University, Taiwan, J. CHEN, I.H. LIAO, ProMOS Technologies, F.-S. HUANG, National Chiao Tung University, Taiwan

HP-16

Influence of Annealing Temperature on Formation of Mo Nanocrystal Memory in Oxygen Incorporated Mo and Si Thin Film, **C.-C. LIN**, National Chiao Tung University, Taiwan, T.-C. CHANG, National Sun Yat-Sen University, Taiwan, C.-H. TU, National Chiao Tung University, Taiwan, S.-C. CHEN, J.-Y. LIN, National Yunlin University of Science and Technology, Taiwan, S.M. SZE, C.-W. HU, T.-Y. TSENG, National Chiao Tung University, Taiwan

HP-17

Charge Stored Effects of Ni-O-Si and Ni-Si-N Nanocrystals Thin Film Using a Low Temperature Fabrication, **W.-R. CHEN**, National Chiao Tung University, Taiwan, T.-C. CHANG, National Sun Yat-Sen University, Taiwan, C.-Y. CHANG, National Chiao Tung University, Taiwan

HP-18

Correlations Between Microstructure and Properties of High Purity Electrodeposited Nickel Coatings, A. GODON, **J. CREUS**, X. FEAUGAS, E. CONFORTO, P. GIRAULT, Université de la Rochelle, France, L. PICHON, Université de Poitiers, France, C. SAVALL, Université de la Rochelle, France

HP-19

Biocompatibility and Anti-Microbial Properties of TiO_x Thin Films, S.E. RODIL, H. ARZATE, Universidad Nacional Autónoma de México, **A ALMAGUER-FLORES**, Universidad Nacional Autónoma de México, Mexico

HP-20

Field Emission Properties of Zinc Oxide Nanorods Grown on ZnO/Si Substrates by Using Plasma Treatment, **I.-C. YAO**, T.-Y. TSENG, P. LIN, National Chiao Tung University, Taiwan

HP-21

Comparison of the Ti-Si-O Composites Nanocrystals Synthesized via Different Methods for Nonvolatile Memory Applications, **L.-W. FENG**, National Chiao Tung University, Taiwan, T.-C. CHANG, National Sun Yat-Sen University, Taiwan, P.-S. WANG, National Chiao Tung University, Taiwan, C.-F. WENG, M.-C. CHEN, D.-S. GAN, N.-J. HO, H.-J. HUANG, National Sun Yat-Sen University, Taiwan, C.-Y. CHANG, National Chiao Tung University, Taiwan

HP-24

Fabrication of Dye-Sensitized Solar Cells Based on ZnO Nanowires, M.F. HOSSAIN, S. BISWAS, M. SHAHJAHAN, **T. ARAKAWA**, T. TAKAHASHI, University of Toyama, Japan

HP-25

Comparative Study of Dye and CdS Sensitized Grätzel Solar Cells, **M.F. HOSSAIN**, S. BISWAS, M. SHAHJAHAN, T. TAKAHASHI, University of Toyama, Japan

HP-27

The Role of Metal Plasma Ion Implantation in Anatase-Titanium Dioxide: Correlation Between Photoreactivity and Implantation Mechanism, **M.-H. SHIH**, Mingdao University, Taiwan, C.-C. YEN, National Chung Hsing University, Taiwan, D.-Y. WANG, Mingdao University, Taiwan, L.S. CHANG, H.-C. SHIH, National Chung Hsing University, Taiwan

HP-29

The Properties of Transparent Semiconductor $Zn_{1-x}Ti_xO$ Thin Films Prepared by Sol-Gel Process, **C.-Y. TSAY**, Feng Chia University, Taiwan, H.-C. CHENG, Industrial Technology Research Institute, Taiwan, C.-Y. CHEN, K.-J. YANG, C.-K. LIN, Feng Chia University, Taiwan

HP-30

Silver Phthalocyanine Films for Photovoltaic Applications, **A. MAHAJAN**, DAV College, India, H. GUPTA, R.K. BEDI, Guru Nanak Dev University, India

HP-34

Effects of the Anodizing Processon the Corrosion Behaviors of Ti-xHf Alloys, **Y.-H. JEONG**, H.-C. CHOE, Y.-M. KO, H. AHN, Chosun University, Korea

HP-35

The Studies of Flexible a-Si:H, Y.-T. CHOU, **C.-Y. SU**, P.-T. LIU, S.-Y. TSAI, National Chiao Tung University, Taiwan, I.-H. PENG, National Tsing-Hua University, Taiwan

HP-36

Characterization of Electrically Conducting Polyaniline Nanofiber/polyimide Nanocomposites, **A. HOPKINS**, The Aerospace Corporation

Surface Engineering for Thermal Management
Room: Town & Country - Session TSP

TSP Poster Session
5:00 – 7:00 pm

TSP-1

Thermal Conductivity of Polymer-Ceramic-Metallic Nanolaminates, **A.R. WAITE**, Air Force Research Lab/UTC, Inc./University of Dayton, J.G. JONES, Air Force Research Lab, A.A. VOEVODIN, Air Force Research Lab/University of Dayton, C. MURATORE, Air Force Research Lab/UTC, Inc., A.M. URBAS, Air Force Research Lab, J.O. ENLOW, Air Force Research Lab/UES, T.J. BUNNING, Air Force Research Lab

TSP-2

Surfaces Incorporating Phase Change Materials for Controlled Storage and Release of Thermal Energy, **C. MURATORE**, Air Force Research Laboratory, S. AOUADI, Southern Illinois University Carbondale, A.A. VOEVODIN, Air Force Research Lab/University of Dayton

TSP-3

Anti-Bacterial TiN_x-Ag Coatings on Titanium Dental Implants, Y.-Y. CHANG, **C.-C. HSUER**, P.-J. LIN, H.-T. HSU, D.-Y. WANG, Mingdao University, Taiwan

TSP-4

A Study of the Anti-Microbial Properties of TiN/Ag Nanocomposite Coatings, **P.J. KELLY**, H. LI, K.A. WHITEHEAD, J. VERRAN, Manchester Metropolitan University, United Kingdom, R.D. ARNELL, University of Central Lancashire, United Kingdom

TSP-5

Carbon Nanotube - MoS2 Nanocomposites as Self-Lubricating Coatings, **B. SIROTA**, X. ZHANG, B. LUSTER, A. CHURCH, Southern Illinois University Carbondale, C. MURATORE, Air Force Research Lab/UTC, Inc., A.A. VOEVODIN, Air Force Research Lab/University of Dayton, P. KOHLI, S. TALAPATRA, S. AOUADI, Southern Illinois University Carbondale

TSP-7

Molecular Dynamics Atom-by-Atom Simulations of Chemical Vapor Deposition of SiNH, J. HOUSKA, **J.E. KLEMBERG-SAPIEHA**, L. MARTINU, Ecole Polytechnique de Montreal, Canada

TSP-8

Corrosion Resistance of Stainless Steel Bipolar Plates for Fuel Cell by High Temperature Nitrogen Implantation, **D.H. KWON**, K.K. KIM, J.S. KIM, Pusan National University, Korea, S.M. MOON, Korea Institute of Machinery & Materials, Korea, J.S. LEE, Korea Atomic Energy Research Institute, Korea, M.C. KANG, Pusan National University

TSP-9

Antibacterial Properties and Tribology of a-C:Ag Coatings Deposited by Pulsed Cathodic Filter Arc, **J. ENDRINO**, Instituto de Ciencia de Materiales de Madrid, Spain, M. ALLEN, Ohio State University, J.C. SANCHEZ-LOPEZ, Instituto de Ciencia de Materiales de Sevilla, Spain, R. ESCOBAR GALINDO, Instituto de Ciencia de Materiales de Madrid, Spain, A. ANDERS, Lawrence Berkeley National Laboratory, J.H. HORTON, T.M. HORTON, SUNY Upstate Medical University, J.M. ALBELLA, Instituto de Ciencia de Materiales de Madrid, Spain

TSP-11

Bactericidal Effect of TiO_2 Particle with Magnetic Core on Fish Pathogens, T.C. CHENG, K.S. YAO, Mingdao University, Taiwan, C.I. CHANG, Fisheries Research Institute, Taiwan, **H.C. HSU**, C.J. HWANG, D.Y. WANG, C.Y. CHANG, Mingdao University, Taiwan

TSP-12

Simultaneous Oxidation and Hydroxyapatite Coating on Titanium and Enhancement of Bioactivity of Osteoblast-Like Cells, S.K. MOON, Yonsei University College of Dentistry, Korea, B.Y. KIM, Incheon National University, Korea, K.Y. KIM, MST Technology Company, Korea, K. KIM, D.H. LEE, **M.H. HONG**, Y.K. LEE, Yonsei University College of Dentistry, Korea

TSP-13

Preparation and Properties of Branched Polymers as Postoperative Tissue Adhesion Barriers, S.-R. HSIEH, Taichung Veterans General Hospital, Taiwan, **C.-J. CHANG**, T.-D. WAY, T.-W. HUNG, Feng Chia University, Taiwan

TSP-14

Coating of Electrically Conductive CNT/PTFE Composite Film on the Metal Bipolar Plate for PEMFC, **Y. SHOW**, K. TAKAHASHI, Tokai University, Japan

TSP-20

Modeling Film Growth in Reactive Sputtering Process, **S. FADDEEVA**, J. OSEGUERA, S. MARTÍNEZ, F. CASTILLO, ITESM-CEM, Mexico

TSP-21

Influence of a Lateral Electric Field on Nucleation, Growth, and Conductivity of Gold Films on Sapphire, **M.S. BYRNE**, R.J. LAD, University of Maine

TSP-24

Growth Characteristics and Sintering Behavior of YSZ Thin Film Prepared by E-Beam Evaporation at Various Pressures, **H.-H. HUANG**, Cheng Shiu University, Taiwan, M.-C. HUANG, M.-H. CHEN, C.-F. YANG, National Kaohsiung First University of Science and Technology, Taiwan, C.-Y. HSU, Cheng Shiu University, Taiwan

NOTES

Friday Morning, May 1, 2009

	Coatings for Use at High Temperature **Room: Royal Palm 1-3 - Session A3-2** **Thermal Barrier Coatings** **Moderators:** A. Bolcavage, Rolls-Royce Corporation, R. Mevrel, ONERA, K. Murphy, Howmet Castings	**Hard Coatings and Vapor Deposition Technology** **Room: Golden West - Session B6-3** **Hard and Multifunctional Nano-Structured Coatings** **Moderators:** M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Laboratories, U.S. Army ARDEC, R. Sanjines, EPFL
8:00 am	**A3-2-3** Moisture-Induced Spallation of Thermal Barrier Coatings, **M. RUDOLPHI**, D. RENUSCH, M. SCHÜTZE, DECHEMA e.V., Germany	**B6-3-1 Invited** Progress in the Development of Adaptive Nitride-Based Coatings for High Temperature Tribological Coatings, **S. AOUADI**, B. LUSTER, P. KOHLI, Southern Illinois University Carbondale, C. MURATORE, Air Force Research Laboratory/UTC, Inc., A.A. VOEVODIN, Air Force Research Laboratory
8:20 am	**A3-2-4** Thermal Barrier Coatings Adherence and Spallation : Interfacial Indentation Resistance and Cyclic Oxidation Behaviour Under Thermal Gradient, **J. SNIEZEWSKI**, V. VIDAL, Y. LE MAOULT, P. LOURS, Université de Toulouse, France	Invited talk continued.
8:40 am	**A3-2-5** Correlation of Mechanical Properties and Electrochemical Impedance Spectroscopy Analysis of Thermal Barrier Coatings, J. GÓMEZ-GARCÍA, A. RICO, **C.J. MÚNEZ**, P. POZA, V. UTRILLA, Universidad Rey Juan Carlos, Spain	**B6-3-3** Influence of the Chemical Composition on Tribological Properties of Nitride-Based Nanocomposite Coatings, P. DESSARZIN, ETH Zurich and PLATIT AG, Switzerland, P. KARVANKOVA, **M. MORSTEIN**, PLATIT AG, Switzerland, N.M. RENEVIER, University of Central Lancashire, United Kingdom, N.D. SPENCER, ETH Zurich, Switzerland
9:00 am	**A3-2-6** The Response of Zirconia Layers under External Thermal and Mechanical Solicitations: Microstructural Evolution and Mechanical Stability, B. BENALI, A.M. HUNTZ, **M. ANDRIEUX**, University Paris Sud 11, LEMHE-ICMMO, France, F. JOMARD, Groupe d' Etude de la Matière Condensée (GEMaC), France, M. IGNAT, SIMAP Grenoble INP, France	**B6-3-4** Development and Properties of Advanced Nitride Coatings for Tooling Applications, **M. LECHTHALER**, F. NEFF, E. PLESIUTSCHING, OC Oerlikon Balzers AG, Liechtenstein, R. FRANZ, University of Leoben, Austria
9:20 am	**A3-2-7 Invited** Understanding and Modeling of Multicomponent Interdiffusion for Life Prediction and Life Extension of Thermal Barrier Coatings, **Y. SOHN**, University of Central Florida	**B6-3-5** Influence of B on Structural, Mechanical and Tribological Properties of Arc Evaporated Al-Cr-N Thin Films, **C. TRITREMMEL**, P.H. MAYRHOFER, University of Leoben, Austria, M. LECHTHALER, OC oerlikon Balzers AG, Liechtenstein, C. POLZER, PLANSEE Composite Materials GmbH, Austria, C. MITTERER, University of Leoben, Austria
9:40 am	Invited talk continued.	**B6-3-6 Invited** From Understanding the Growth Mechanism to the Design and Fabrication of High-Performance Functional Coating Architectures, **L. MARTINU**, Ecole Polytechnique de Montreal, Canada, A. AMASSIAN, Cornell University, E. BOUSSER, S. HASSANI, J. HOUSKA, R. VERNHES, J.E. KLEMBERG-SAPIEHA, Ecole Polytechnique de Montreal, Canada
10:00 am	**A3-2-9** Comparison of the Oxidation Behavior of Beta and Gamma - Gamma Prime NiPtAl Coatings, J.A. HAYNES, **B.A. PINT**, Oak Ridge National Laboratory, Y. ZHANG, Tennessee Technological University, I.G. WRIGHT, Oak Ridge National Laboratory	Invited talk continued.
10:20 am	**A3-2-10** Effect of Exposure Conditions on the Oxidation of MCrAlY-Bondcoats and Lifetime of Thermal Barrier Coatings, **J. TOSCANO**, M. SUBANOVIC, E. WESSEL, D. NAUMENKO, L. SINGHEISER, J. QUADAKKERS, Research Center Juelich, Germany	**B6-3-9** Combinatorial Approach to the Growth of α-$(Al,Cr)_2O_3$ Solid Solution Strengthened Thin Films by Reactive r.f. Magnetron Sputtering, **M. STUEBER**, D. DIECHLE, H. LEISTE, S. ULRICH, Forschungszentrum Karlsruhe, Germany, V. SCHIER, Walter AG, Germany
10:40 am		**B6-3-10** Dense Nanostructured Oxide Coatings Made By Plasma Spray and Conventional Consolidation Processes, **M. GELL**, E.H. JORDAN, J. WANG, C. MUOTO, University of Connecticut
11:00 am		**B6-1-9** Nano-Structured CrN/AlN Superlattice Coatings Synthesized by Pulsed Closed Field Unbalanced Magnetron Sputtering, J. LIN, **B. MISHRA**, Colorado School of Mines, M. PINKAS, Nuclear Research Center, Israel, J.J. MOORE, Colorado School of Mines, W.D. SPROUL, Reactive Sputtering, Inc.
	Thank You & Farewell Party Trellises Courtyard near pool 12:00 – 1:30 pm	Award Nomination Submission Deadlines: RF Bunshah and Graduate Student October 1, 2009

Friday Morning, May 1, 2009

	Optical Thin Films **Room: Royal Palm 4-6 - Session C3** **Optical Characterization of Thin Films** **Moderators:** U. Beck, BAM Berlin, T. Tiwald, J.A. Woollam Co., Inc.	**Tribology and Mechanical Behavior of Coatings and Thin Films** **Room: California - Session E3-2** **Tribology of Nanostructured and Amorphous Films** **Moderators:** J. Fontaine, Ecole Centrale de Lyon, T.W. Scharf, The University of North Texas
8:00 am	**C3-1 Invited** Spectroscopic Ellipsometry for Characterization of Thin Films and Surfaces: Harnessing Materials for Energy, **M. SCHUBERT**, University of Nebraska-Lincoln	**E3-2-1** Characterization and Modeling of Self-Lubrication in Nanocrystalline Nickel, **C.C. BATTAILE**, S. PRASAD, J.R. MICHAEL, Sandia National Laboratories
8:20 am	Invited talk continued.	**E3-2-2** Nanocomposite Nickel Coatings with Silicon Carbide Reinforcement: Friction and Wear Behaviour at 298 and 493 K, **M. SHAFIEI**, A.T. ALPAS, University of Windsor, Canada
8:40 am	**C3-3** Effect of Laser Fluence on ZnCdS Thin Films, S. SINGHAL, A.K. CHAWLA, **R. CHANDRA**, H.O. GUPTA, Indian Institute of Technology Roorkee, India	**E3-2-3 Invited** Nanoscale Deformation Mechanism of Nanocomposite Thin Films, **J. DE HOSSON**, C. CHEN, Y. PEI, K. SHAHA, University of Groningen, Netherlands
9:00 am	**C3-4** Effect of Rapid Thermal Annealing on the Electrical and Optical Behaviors of Cu2O-Ag Nanocomposite Thin Films, **C.-C. TSENG**, National Chung Hsing University, Taiwan, J.H. HSIEH, Mingchi University of Technology, Taiwan, W. WU, National Chung Hsing University, Taiwan	Invited talk continued.
9:20 am	**C3-5 Invited** Laser Damage Thresholds of Optical Coatings, **D. RISTAU**, Laser Zentrum Hannover e.V., Germany	**E3-2-5** Tribological Investigation of New Low Wear Coating Systems on Ti6Al4V, **K.J. KUBIAK**, Ecole Centrale de Lyon, France and University of Leeds, United Kingdom, T.G. MATHIA, Ecole Centrale de Lyon, France, B.G. WENDLER, W. PAWLAK, Technical University of Lodz, Poland
9:40 am	Invited talk continued.	**E3-2-6** Wear of C/CrC Coating Against Alumina at Room Temperature, **Z. ZHOU**, I. ROSS, L. MA, W. RAINFORTH, University of Sheffield, United Kingdom, P.EH. HOVSEPIAN, Sheffield Hallam University, United Kingdom
10:00 am	**C3-7** Numerical Ellipsometry: Analysis of Thin Metal Layers Using n-k Plane Methods with Multiple Incidence Angles, **F.K. URBAN**, D. BARTON, Florida International University, T. TIWALD, J.A. Woollam Co., Inc.	**E3-2-7** Constitution, Microstructure, and Tribological Properties of Nanocrystalline Reactive Magnetron Sputtered V-Al-C-N Hard Coatings, **C. ZIEBERT**, M. STUEBER, H. LEISTE, S. ULRICH, Forschungszentrum Karlsruhe, Germany
10:20 am	**C3-9** Investigation of Scattering Mechanisms in Transparent Conductive Ga-Doped ZnO Films with Thicknesses of Less than 100 nm, T. YAMADA, A. MIYAKE, H. MAKINO, N. YAMAMOTO, **T. YAMAMOTO**, Kochi University of Technology, Japan	**E3-2-8** Surface Morphology and Tribological Properties of Multilayer TiAlN Coatings Deposited by Reactive Magnetron Sputtering, M. WANG, T. TOIHARA, M. SAKURAI, OSG Corporation, Japan, **W. KUROSAKA**, S. MIYAKE, Nippon Institute of Technology, Japan
10:40 am	**C3-10** Optical and Photoluminescence Studies of Gold Nanoparticles - Embedded ZnO Thin Films, A. PATRA, V. DAMODARA DAS, S. KASIVISWANATHAN, Indian Institute of Technology Madras, India, **S. KUMAR**, Guru Nanak Dev University, India	**E3-2-10** CrN-Ag Nanocomposite Coatings: High Temperature Tribological Properties, **C.P. MULLIGAN**, Benet Laboratories, U.S. Army ARDEC, T.A. BLANCHET, D. GALL, Rensselaer Polytechnic Institute
11:00 am	**C3-11** Anisotropic Optical Properties of Sculptured Thin Films Grown by Glancing Angle Deposition, **D. SCHMIDT**, University of Nebraska-Lincoln, B. BOOSO, University of Dayton, T. HOFMANN, University of Nebraska-Lincoln, A. SARANGAN, University of Dayton, E. SCHUBERT, M. SCHUBERT, University of Nebraska-Lincoln	
	Thank You & Farewell Party Trellises Courtyard near pool 12:00 – 1:30 pm	Award Nomination Submission Deadlines: RF Bunshah and Graduate Student October 1, 2009

Friday Morning, May 1, 2009

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G2-2
Coatings for Automotive and Aerospace Applications
Moderators: G. Dadheech, General Motors,
H. Rudigier, OC Oerlikon Balzers AG

8:00 am	**G2-2-1** TiN Multilayer Systems for Compressor Airfoil Sand Erosion Protection, **A. FEUERSTEIN**, A. KLEYMAN, Praxair Surface Technologies, Inc.	
8:20 am	**G2-2-2** Lubricated PVD Coatings for Automotive Applications, K. BOBZIN, N. BAGCIVAN, N. GOEBBELS, **K. YILMAZ**, RWTH Aachen University, Germany, B.-R. HOEHN, K. MICHAELIS, M. HOCHMANN, University of Munich, Germany	
8:40 am	**G2-2-3** Multiphase Wear and Erosion Resistant Coatings for Aerospace Applications, **J. NAINAPARAMPIL**, A.K. RAI, R. BHATTACHARYA, UES Inc.	
9:00 am	**G2-2-4** Investigations on the Interfacial Strength of Chromium Adhesion Layers in DLC Coating Systems for High Load Applications, **J. SCHAUFLER**, K. DURST, University Erlangen Nürnberg,, Germany, R. MERTENS, Oerlikon Balzers AG, Liechtenstein, M. GÖKEN, University Erlangen Nürnberg, Germany	
9:20 am	**G2-2-5 Invited** Innovative Surface Technologies for Advanced Automotive Applications: From Super-Hard and –Low Friction Coatings to Super-Fast Surface Treatments, **A. ERDEMIR**, O.L. ERYILMAZ, G. KARTAL, Argonne National Laboratory, K. KAZMANLI, S. TIMUR, M. URGEN, Istanbul Technical University, Turkey	
9:40 am	Invited talk continued.	
	Thank You & Farewell Party Trellises Courtyard near pool 12:00 – 1:30 pm	Award Nomination Submission Deadlines: RF Bunshah and Graduate Student October 1, 2009

NOTES

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B2-1

Arc and E-Beam Coatings and Technologies
Moderator: V.I. Gorokhovsky, Arcomac Surface Engineering, LLC, J. Vetter, Sulzer Metaplas GmbH

10:00am **B2-1-1 Metallic Film Deposition using a Vacuum Arc Plasma Source with a Refractory Anode**, *I. Beilis,* ***R.L. Boxman*** *(boxman@eng.tau.ac.il)*, Tel Aviv University, Israel **INVITED**

A plasma source has been developed utilizing the Hot Refractory Anode Vacuum Arc (HRAVA) mode. These arcs are initiated as cathode spot arcs whose plasma jets initially deposit cathode material on a refractory anode. The anode is heated by the arc, and a dense plasma plume is formed by re-evaporation of cathode material from the anode. The steady-state HRAVA mode is reached when the anode is sufficiently hot that there is no net deposition of cathodic material on it, and a plasma plume expands radially. Thin films of cathodic material are formed on substrates arranged radially around the electrode axis. Macroparticle-free films form on substrates having a direct line of sight to the anode, but not to the cathode. HRAVAs with current I=150-340 A, Cu, Ti, and Cr cathodes, graphite, Mo and W anodes and 5 10 mm inter-electrode gaps were investigated theoretically and experimentally. The time to reach steady state with a graphite anode decreased from ~90 s to 60 s when I inc reased from 175 to 340 A. The anode surface temperature linearly increased with I: on a graphite anode from 1950 K to 2200 K when I increased from 175 to 340A, and on a W anode from 2250 to 2500 K when I increased from 150 to 250 A. The temperature distribution on the asymmetric anodes with inclined front surfaces was asymmetric, and their maximum temperature was higher than for symmetric ones by about 50–100 K. The difference between the maximum and minimum temperatures on the anode surface increased as the inter-electrode gap decreased. The deposition rate was determined by measuring the deposited film thickness by profilometry -- in 300 A arcs with Cu cathodes, at distances of L=110 and 80 mm from the arc axis, 2.0 and 3.6 μm/min respectively. The HRAVA Cr deposition rate at L=80 mm for I=200 to 300 A was 0.72 to 1.4 μm/min, and for Ti at L=100 mm, 0.88 to 1.8 μm/min. HRAVA thickness distribution was axially symmetric within 10% and the mass deposition rate was ~400 mg/min and was almost MP-free. The steady-state HRAVA deposition rate was 2.3 μm/min, compared to the Filtered Vacuum Arc Deposition (FVAD) rate which was 0.25 μm/min. Furthermore, the area deposited by the HRAVA source was 100 cm^2 (8 cm from the electrodes axis), while the area deposited by the FVAD source was 30 cm^2 (70 cm from the cathode). The cathode utilization efficiency using HRAVA was 40 times greater than with FVAD.

10:40am **B2-1-3 Investigation of Electrochromic Properties of Nickel Oxide Films Prepared by Electrom Beam Evaporation**, ***D.R. Sahu*** *(sahu@mail.ncku.edu.tw), T.-J. Wu, J.-L. Huang*, National Cheng Kung University, Taiwan

Electrochromic nickel oxide films were deposited by electron beam evaporation method. Films were charcterized and anlysed using XRD, SEM, AFM, ESCA Cyclic voltamettry and UV-Vis spectrophotometer. The vairation in the electrochromic properties of NiOx films as a functions of electron beam power along with thickness are studied. XRD anlaysis indicted that different electron beam power led to the deposition of film with different orientation with different electrochromic behhaviour. The optimum performances of the film are attributed with highest ratio of (111) plane orientation. The thinner film possesses better efficiency than the thicker film. However performance of the thinner film is deteriorated after subjection to 800 cycling tests. The details electrochromic properties of the films will be discussed during presentation.

11:00am **B2-1-4 Surface Modification of Iron Containing Aluminum Alloys by Treating with Copper Plasma Produced with Cathodic Arc**, ***B. Corlu****, M. Urgen (urgen@itu.edu.tr)*, Istanbul Technical University, Turkey

In this study, we describe a novel approach for modification of surfaces by the help of cathodic arc plasma and its bias voltage dependent interaction with the substrates. As an example, alloying and surface modificaiton of aluminum with cathodic arc plasma of copper is given. Surfaces of aluminum alloys with iron contents of 0.2 and 1% was modified with cathodic arc physical vapor deposition (CA-PVD) using copper cathodes. The effect of successively applied bias voltages of -150V and -1000 V was investigated on the resulting microstructural surface features of the alloys. Surface modification processes were performed below and above 500° C. In the experiments conducted below 500° C, the modified surface predominantly consisted of Al_2Cu intermetallics, accompanied with a small amount of α-aluminum solid solution. However, in experiments conducted above 500° C melting and resolidification of the copper deposited substrate surface took place corresponding to -1000V and -150V bias voltages respectively. A eutectic-like microstructure, containing α-aluminum, Al_2Cu phases, and iron containing intermetallic quasicrystal structures were obtained.

11:20am **B2-1-5 Structure and Mechanical Properties of Multilayer Nanocrystalline TiAl/TiAlYN Coatings, Produced by Reactive Cathodic-Arc-Evaporation**, ***V.S. Goltvyanytsya*** *(vladtnt@gmail.com), S.K. Goltvyanytsya,* Company Real Ltd., Ukraine*, A.V. Demchyshyn, L.D. Kulak, M.V. Bulanova, I.D. Gornaya,* Institute of Problems in Material Science, NANU, Ukraine*, V.I. Gorokhovsky*, Arcomac Surface Engineering, LLC

One of the effective trends of multifunctional coatings improvement is connected with development of multilayer composition layer technology with nano thickness of each layer on the basis of refractory metal compounds. By varying of technological parameters of coating condensation it is possible to influence on their properties. Multilayer combined deposits with alternate layers provide optimal combination of wearability, strength and fracture strength.

In this study influence of sublayers on structure properties of multilayer deposits TiAlN/TiAlYN (150-300 layers) with fixed total thickness 10-15 μm were examined. Single-layer coatings TiAlN had microhardness of 40 Gpa. Multilayer systems were produced via vacuum-arc atomization of TiAl and TiAlY cathodes in nitrogen atmosphere with further coatings condensation on steel substrates. For creation of system with different layer thickness the time of one layer deposition varied from 10 to 30 sec. Total deposition time of coatings was the same and equal to about 60 min. Thickness of sublayers was less than 100 nm. Microstructure, elemental and phase composition were studied by means of scanning microscopy, Auger-electron spectroscopy, X-ray microanalysis and X-ray structure analysis. Microhardness of vacuum-arc condensates was measured by means of systems PMT-3M and "Micron-Gamma". Formed multilayer TiAlN/TiAlYN deposits had enhanced hardness and oxidation resistance in comparison with monolayer TiAlN owing to creation of protection amorphous Al_2O_3-Y_2O_3 layer, which prevented their further oxidation was established.

Improved mechanical properties of multilayer coatings (TiAlN/TiAlYN) in comparison with homogeneous deposits, which were formed on the basis of mononotrides of transition metals, can be widely adopted as protective heat resistant and anti-wear coatings.

11:40am **B2-1-6 Optical Reflective Performance of Copper Alloy Thin Films with Ti-Zr and Al-Zr Additions**, ***C.-Y. Su*** *(cysu@ntut.edu.tw),* National Taipei University of Technology, Taiwan*, C.-Y. Tsay,* Feng Chia University, Taiwan*, C.-W. Chang,* National Taipei University of Technology, Taiwan*, C.-H. Hsu,* Tatung University, Taiwan*, C.-K. Lin*, Feng Chia University, Taiwan

Copper alloy with additions of less than 3 wt.% of Ti-Zr or Al-Zr are prepared by vacuum arc remelter (VAR). After rolling, heat treating, and cutting, homemade targets with a diameter of 2" are available for DC sputtering onto silicon and/or glass substrates. After deposition, the chemical compositions of the thin films are examined and compared with those of the original targets. The optical reflective performances of the as-deposited films and those after a durability test at 50 oC with 85% humidity for 24 hours are investigated. Experimental results show that Cu-Ti-Zr thin films are superior in reflectivity than those of the Cu-Al-Zr films. Cu thin film with Ti-Zr additions exhibits a reflectivity around 70%, obviously better than those of the Cu-Al-Zr films (less than 40%). An average decrease of ~10% in reflectivity can be noticed after durability test. The optical reflective performance of copper alloy thin films with respect to composition, microstructure, and surface morphology will be addressed in the present study.

CVD Coatings and Technologies

Moderator: M. Pons, SIMAP, Grenoble-INP, CNRS, H. Holzschuh, Walter AG

10:00am **B3-1-1 The Influence of CVD Process Parameters on the Texture of α-Al_2O_3 Coatings**, ***S. Ruppi*** *(sakari.ruppi@secotools.com)*, Seco Tools

Even though textured CVD α-Al_2O_3 layers have been overwhelmingly dealt with in patent literature there are only a few published research studies on this subject[1,2,3]. Even though some preferred growth textures in laboratory scale CVD α-Al_2O_3 coatings have been observed already in the 1980's[4] it has not been possible control the growth direction of CVD α-Al_2O_3. However, advances in the nucleation and deposition technology have enabled controlled deposition of strongly textured α-Al_2O_3 coatings[1,2] and in a recent study[2] it was shown that α-Al_2O_3 could be obtained with very strong textures and that the texture of α-Al_2O_3 could be controlled by the process data. This study[2] also indicated a substantial influence of the growth texture on the wear properties of coated tools.

In the present paper the influence of the process parameters on the texture and growth of α-Al_2O_3 was investigated. The experimental α-Al_2O_3 coatings were deposited both as multi- and single layers and the nucleation surfaces were kept identical for all experimental α-Al_2O_3 layers. Coatings were studied by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Texture was determined by X-ray diffraction using CrK_a and CuK_a radiation. Further, microtexture was investigated using electron back-scatter diffraction (EBSD).

The effects of conventional CVD precursors and total pressure on the growth and texture of @alpah@-Al_2O_3 will be discussed. The experimental results that will be presented clearly showed that growth rate and microstructure of α-Al_2O_3 could strongly be manipulated by the process data. Most importantly, the experiments confirmed that the growth texture could simply be determined by process parameters.

[1]S. Ruppi, Int. J. Refract. Met. Hard Mater. 23(2005) 306-316.

[2]S.Ruppi, Surf. Coat. Technol. 202 (2008) 4257.

[3]Osada, E. Nakamura, H. Homma, T. Hayahi, T. Oshika, Int. J. Refract. Met. Hard Mater. 224 (2006) 387.

[4]J.M. Kim, C.S. Park, J.C. Chun, Thin Solid Films. 97(1982) 536.

10:20am **B3-1-2 Microstructural Investigation of CVD Alumina Coatings Grown on Single Crystals of α-Al_2O_3**, ***S. Canovic*** *(sead.canovic@chalmers.se),* Chalmers University of Technology, Sweden*, B. Ljungberg, C. Björmander,* Sandvik Tooling, Sweden*, M. Halvarsson*, Chalmers University of Technology, Sweden

Multilayer coatings of TiC, TiN and Al_2O_3 are often formed on cemented carbide cutting tools by conventional chemical vapour deposition (CVD) in order to increase the wear resistance of the tools. In this work, CVD multilayer alumina coatings separated by thin layers of TiX (X=C or N) are investigated. There are several reports indicating that the interfacial structures are important for the characteristics of the TiX/alumina coatings. The multilayer coatings are deposited on α-Al_2O_3 single crystals with given top surfaces in order to avoid the influence of the diffusion of tungsten and cobalt from a cemented carbide substrate. Additionally, the use of these substrates makes it possible to study TiX/alumina interfaces where the nucleation surface is well defined and known beforehand. Previously, we have investigated TiX/alumina multilayer coatings deposited on single crystals of α-Al_2O_3 with 2 different top surfaces (c and r) and we observed some interesting differences. In this work the sample matrix is extended and includes CVD multilayer alumina coatings, separated by thin layers of TiC, deposited on α-Al_2O_3 single crystals with 4 given top surfaces (c, r, a, m). The main part of this work was carried out by transmission electron microscopy (TEM) using a Phillips CM 200 FEG working at 200 kV. Cross-section TEM thin foils were prepared using a combined FIB/SEM (focused ion beam/scanning electron microscope) instrument, whereby electron transparency was achieved throughout the coating thickness. The detailed microstructure of the different coatings will be described in this work.

10:40am **B3-1-3 Residual Stress Evolution in CVD Multilayer Coatings on Cutting Tools at High Temperature**, ***M. Klaus,*** Technische Universität Berlin, Germany*, Ch. Genzel (genzel@helmholtz-berlin.de),* Helmholtz-Centre Berlin for Materials and Energy, Germany*, H. Holzschuh*, Walter AG, Germany

Cutting tools are usually coated by CVD methods to protect them from abrasive wear and to increase the lifetime of the products. To prevent crack propagation through the coatings, the films are not uniform but consist of stacks of alternating sublayers with different thickness, chemical structure and crystallographic texture. In case of Al_2O_3/TiCN mulilayer structures deposited on WC substrates, the as-grown coatings exhibit tensile residual stresses which is due to the differences in the thermal expansion coefficients. To generate beneficial compressive stresses in the coatings, which contribute significantly to the excellent mechanical properties, subsequent mechanical surface treatment such as grit blasting is applied. In previous work we showed by means of X-ray diffraction methods that the residual stresses introduced into the multilayer coatings by blasting are not uniform but occur in form of more or less steep intra- and interlayer stress gradients wit hin or between the individual sublayers and/or the substrate. In the present work we extend our investigations to residual stress analysis at high temperatures which occur under service conditions. The main goal of this study is to analyze the residual stress evolution in the individual sublayers and the substrate and to quantify the ratio between extrinsic (thermal) and intrinsic (blasting induced) stresses as a function of temperature.

11:00am **B3-1-4 Surface Treatments for Enhancing the Adhesion of Nanocrystalline Diamond Coatings to Tungsten Carbide Micro End Mills**, ***P.J. Heaney*** *(trackingfast@hotmail.com), C.D. Torres,* University of Wisconsin - Madison*, M.A. Hamilton,* University of Pennsylvania*, A.V. Sumant,* Argonne National Laboratory*, K. Sridharan,* University of Wisconsin - Madison*, R.W. Carpick,* University of Pennsylvania*, F.E. Pfefferkorn*, University of Wisconsin - Madison

Diamond has outstanding material properties such as high hardness, high thermal conductivity, chemical inertness, and extremely low friction and wear under particular conditions. Applying a diamond coating to cutting tool surfaces can significantly increase the tribological performance and hence the operational life-span of the tool. Coating micro end mills with diamond is a challenging task, due to the small size (5-300 μm diameter) and complicated shapes. We have been able to coat micro end mills with continuous diamond films as thin as 60 nm. Micro end milling tests under severe conditions (dry cutting of adhesive material) have demonstrated that the diamond coatings significantly increase the machining performance (reduced forces, adhesion, wear, friction) of the tools. The main failure mode of the tool is delamination of the coating, due to poor adhesion between the coating and the tungsten carbide (WC) tool. Coating adhesion must be improved to increase the ove rall tool life and performance of micro end milling.

Cobalt (Co) in the WC adversely effects diamond growth and coating adhesion, requiring a substrate preparation technique to prepare tools. The preparation must result in a surface that allows proper diamond nucleation, low Co content, prevent Co from diffusing to the surface during diamond deposition, and not weaken the tool or introduce residual stresses. The methods that we have studied include depositing a tungsten interlayer, treating the surface with low energy (< 5keV) carbon ion implantation, and chemical etching with a hydrofluoric acid solution. After surface preparation, the tools were characterized using scanning electron microscopy, atomic force microscopy, and white light interferometry to measure changes in morphology and roughness, x-ray photoelectron spectroscopy (XPS), Raman, and near-edge x-ray absorption fine structure spectroscopy (NEXAFS) to observe chemical and bonding changes of the substrat e and resulting diamond coatings, and machining tests to check the integrity of the tool. The tools were then coated with diamond and evaluated while dry machining aluminum. Coating adhesion while machining was measured by time before delamination, severity of delamination, and change in machining metrics.

Use of the Center for Nanoscale Materials was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under Contract No. DE-AC02-06CH11357.

11:20am **B3-1-5 Synthesis, Properties and Cutting Performance of the $(Al,Cr)_2O_3$ Coating by CVD**, ***A. Osada*** *(aosada@mmc.co.jp)*, Mitsubishi Materials Corporation, Japan **INVITED**

Al_2O_3 coatings have been one of the most important coatings for the cutting tool and are mostly produced by CVD (Chemical Vapor Deposition) process. PVD Al_2O_3 coatings had been developed for a long time and introduced to the market a few years ago. Recently there are some reports about cutting performance of $(Al,Cr)_2O_3$ and Cr_2O_3 coatings by cathodic arc evaporation, however no reports in case using CVD process. In this work

$(Al,Cr)_2O_3$ and Cr_2O_3 coatings were deposited using a hot-wall CVD equipment, with $AlCl_3$ - Cr-chloride - CO_2 - HCl - H_2 gas mixture. The growth textures and morphologies of the coatings were analyzed by XRD, SEM and TEM. The effects of the Cr content on these properties will be discussed. The cutting test results of the coatings will be presented.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D2-1

Diamond and Diamond-Like Carbon Materials
Moderator: O. Shenderova, International Technology Center, R. Hauert, Empa

10:00am **D2-1-1 Preparation and Comparison of a-C:H Coatings using Reactive Sputter Techniques**, ***M. Keunecke*** *(martin.keunecke@ist.fraunhofer.de), K. Weigel, K. Bewilogua,* Fraunhofer Institute for Surface Engineering and Thin Films, Germany*, R. Cremer,* CemeCon A.G., Germany*, H.-G. Fuss*, Cemecon AG, Germany

Amorphous hydrogenated carbon (a-C:H) coatings are widely used in several industrial applications. These coatings commonly will be prepared by plasma activated chemical vapor deposition (PACVD). The mainly used method to prepare a-C:H coating in industrial scale bases on a glow discharge in a hydrocarbon gas like acetylene or methane using a substrate electrode powered with medium frequency (m.f. - some 10 to 300 kHz). Some aims of further development are adhesion improvement, increase of hardness and high coating quality on complex geometries. A relatively new and promising technique to fulfill these requirements is the deposition of a-C:H coatings by a reactive d.c. magnetron sputter deposition from a graphite target with acetylene as reactive gas. An advancement of this technique is the deposition in a pulsed magnetron sputter process. Using these three mentioned techniques a-C:H coatings were prepared in the same deposition machine a Cemecon CC 800/9. For adhesion i mprovement different interlayer systems, e.g. CrN were applied. The effect of different substrate bias voltages (d.c. and d.c.pulse) was investigated. Applying magnetron sputter technique in the d.c. pulse mode, micro hardness values up to 40 GPa could be reached. Beside the hardness other mechanical properties like resistance against abrasive wear and adhesion were measured and compared. Cross sectional SEM images showed the growth structure of the coatings. The coating process capability will be demonstrated with images of coated mills.

10:20am **D2-1-2 Effect of Boron Incorporation on the Structure and Electrical Properties of Diamond-Like Carbon Films Deposited by Femtosecond and Nanosecond Pulsed Laser Ablation**, *A. Sikora,* Université Jean Monnet, France*, O. Bourgeois, J.L. Garden,* CNRS et Université de Grenoble, France*, J.N. Rouzaud,* Ecole Normale Supérieure, France*, J.C. Sanchez-Lopez, T.C. Rojas,* Instituto de Ciencia de Materiales de Sevilla, Spain*, A.S. Loir, F. Garrelie,* ***C. Donnet*** *(Christophe.Donnet@univ-st-etienne.fr)*, Université Jean Monnet, France

The influence of the incorporation of boron in Diamond-Like Carbon (DLC) films on the microstructure of the coatings has been investigated. The a-C:B films have been deposited at room temperature in high vacuum conditions, by ablating graphite targets either with a femtosecond pulsed laser (800 nm, 150 fs, femto-DLC), or with a nanosecond pulsed laser (248 nm, 20 ns, nano-DLC). Doping with boron within the range 2-8 % has been performed by ablating alternatively graphite and boron targets. The film structure and composition have been highlighted by coupling Atomic Force Microscopy, Scanning Electron Microscopy, Electron Energy Loss Spectroscopy and High Resolution Transmission Electron Microscopy. Using the B K edge, EELS characterization reveals the boron effect on the carbon bonding. Moreover, the plasmon energy reveals a tendency of graphitization associated to the boron doping. The boron particles synthesized by femtosecond PLD have been characterized by HRTEM and reveals that the particles are amorphous or crystallized. The nanostructure of the boron doped nano-DLC and the boron doped femto-DLC are thus compared. In particular, the incorporation of boron in the DLC matrix is highlighted, depending on the laser used during deposition. Electrical measurements will show that some of these films have potentialities to be used in low temperature thermometry (77-300 K range), considering their conductivity and temperature coefficient of resistance (TCR).

10:40am **D2-1-3 Retrospective Lifetime Prediction of Failed and Explanted DLC Coated Hip Joint Balls**, ***G. Täger*** *(georg.taeger@uni-essen.de), L.E. Podleska,* University Essen, Germany*, C.V. Falub, G. Thorwarth, M. Stiefel, R. Hauert*, Empa, Switzerland **INVITED**

Background

particle induced osteolysis is still a maior concern in total hip arthroplasty. Numerous efforts are made to improve tribogy and reduce wear induced particles which cause subsequent inflammation and implant loosening. One of these modifications is thin film coating of the bearing surfaces using diamond like carbon coatings.

Materials&methods

In a consecutive trial 101 DLC-coated femoral heads (DLC-group) and another 101 femoral heads consisting of Al_2O_3, both articulating against polyethylene cups, were implanted. Patients were comparable for age, gender, activity index and indications. Surgery followed a standard protocol. There were no further differences regarding design, cementless fixation or any other surgical issue.

Results

Survivorship analysis for aseptic loosening 8.5 years following implantation resulted in a significant difference between both groups with a 54% survival for DLC/PE compared to 88% for Al_2O_3/PE bearings ($p < 0.001$). SEM showed delamination of the carbon layer which caused excessive debris of polyethylene and in some cases even of the metallic substrate of the heads XPS depth profiling and cross sectional SEM analysis performed on the explanted DLC coated TiAlV hip joint balls revealed that the coating consists of several DLC-Si layers with an adhesion promoting Si interlayer on top of the TiAlV substrate. Using an adhesion determination technique a delamination speed of about 50 micrometer per year was estimated for the DLC coated TiAlV hip joints, in agreement with the size of the ob-served delaminated spots. High-resolution SEM monitoring of the Focused Ion Beam (FIB) transversal cuts in the vicinity of the delaminated spots revealed that the delamination of the DLC coatings is correlated with the in-vivo corrosion of the under laying adhesion promoting Si interlayer.

Conclusion

Despite the promising effects of thin film coatings, detailed investigation on the surfaces characteristics have to be performed prior to clinical use

11:20am **D2-1-5 Properties of Nanostructured Surfaces of DLC Thin Films Prepared by Pulsed-DC PECVD**, ***C. Corbella*** *(corbella@ub.edu), S. Portal, M. Rubio-Roy, E. Bertran, M.C. Polo, E. Pascual, J.L. Andújar*, University of Barcelona, Spain

Diamond-like carbon (DLC) thin films, which show attractive mechanical properties for their performance as hard and tribological coatings, have been prepared at room temperature by pulsed-DC plasma-enhanced chemical vapour deposition (PECVD). Previous to the deposition, substrate nanostructuring has been performed in order to tailor the surface properties of DLC. For that, spherical silica submicroparticles of between 300 and 500 nm diameter were synthesized by sol-gel method and deposited on silicon wafers by Langmuir Blodgett. After that, the samples were annealed to promote chemical bonds between the spheres and the substrate, and finally, DLC films with thickness equivalent to particle size were deposited. A systematic study of the surface properties has been performed: wettability (contact angle), friction coefficient (nanotribometer) and abrasive wear rate (Calotest) were evaluated to optimize substrate nanostructure and deposition parameters. Such surface modification is suitable for applications requiring protective coatings with tunable hydrophobicity.

11:40am **D2-1-6 Topography Development of Toluene Based a-C:H Coatings Deposited by RF-PECVD**, ***C. Hormann*** *(christoph.hormann@iwm.fraunhofer.de), S. Meier*, Fraunhofer Institute of Mechanics of Materials IWM, Germany

PECVD deposition using toluene as hydrocarbon precursor gas has proven to be an efficient method to deposit amorphous hydrogenated carbon (a-C:H) films for tribological applications. These coatings feature an unusual surface topography compared to films deposited from conventional precursors like methane or acetylene. Mound-like structures on the nano- and micrometer scale develop early during film growth and show a distinct dependence on the initial substrate structure and the progress of deposition. We investigated this growth both experimentally and using continuum simulation models and present results which show that taking into account non-local self shadowing effects of the surface topography during deposition we can reproduce the experimentally observed behaviour in simulation.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E2-1

Mechanical Properties and Adhesion
Moderator: R. Chromik, McGill University, L. Davies, Caterpillar, J. Michler, Empa

10:00am **E2-1-1 Fracture and Failure in Polymer – Metal Systems for Flexible Electronics**, ***D.F. Bahr*** *(dbahr@wsu.edu), J.D. Yeager,* Washington State University*, M.S. Kennedy,* Clemson University*, N.R. Moody*, Sandia National Laboratories **INVITED**

The impact of adhesion on the reliability of small systems that utilize metals on polymers, both on the micro- and meso-scale, can directly determine the reliability of these devices in service. In this paper the toughness of the gold – polyimide and gold – polymethyl methacrylate interface will be measured experimentally, and the results of the toughness will be correlated to sample preparation, the presence of an adhesion promoting layer, and the results of reliability testing. These systems are applicable to many modern electronic technologies incorporating flexible substrates in order to increase effectiveness and reliability over a wide range of conditions. Microelectronic devices which utilize polymers coated with patterned thin metal films are already being developed for such uses as electronic paper, computer displays, and smart textiles. The properties of flexible electronics also make them a viable option for small, implantable electrodes for recording bra in signals. They are also possible candidates for power generation in vibration harvesting applications. The need for quantitative methods arose from the desire to predict film adhesion under a variety of externally applied stresses and also directly compare different film systems. These quantitative measuring techniques can be subdivided into two different groups. The first group uses compressive stresses to induce fracture along an interface and then estimates the adhesion energy by modeling the delamination morphology the stressed overlayer technique; while the second group measures the crack growth rate as a function of applied load. This paper will demonstrate the toughness of polymer – metal system interfaces is related to both the type and presence of adhesion promoting layers (Cr, Ti-W, SiO_2, and Ta) and alterations in surface chemistry through plasma treatments. Both methods show improvements, with plasma treating causing an improvement of the toughness of the gold – Kapton interface by 50%, and adhesion promoting layers improving the toughness of the gold – PMMA system a similar amount. Initial results on a piezoelectric polymer (PVDF) will also be presented. @paragra[j@The authors wish to acknowledge the financial support of the National Institute of Mental Health (NIMH 60263 & NIMH 71830) and the Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

10:40am **E2-1-3 Softening and Hardening Behavior of Nano-Columnar a-C:H Coating via Electron Beam Irradiation**, ***T. Aizawa*** *(aizawa@asiaseed.org),* AsiaSEEd-Institute, Japan*, E. Iwamura,* Arakawa Chemical Co. Ltd., Japan*, T. Uematsu*, Tokyo Metropolitan Industrial Research Institute, Japan

Three dimensionally grown a-C:H coated films are chemically modified to have fine columnar nano-structure via electron beam irradiation. With increasing the duration time via this electron beam irradiation, densification takes place in the initially deposited a-C:H films irrespective of the sputtering pressure and the hydrogen content in a-C:H film. When starting from the deposited a-C:H film by higher sputtering pressure and hydrogen content, graphitization advances in the inter-columns together with densification. In this case, both hardness and Young's modulus decrease with the duration time in the electron beam irradiation. That is, the initial a-C:H deposited film makes softening via electron beam irradiation. On the other hand, when starting from the deposited a-C:H film by lower sputtering pressure and hydrogen content, both hardness and Young's modulus increase with the duration time; hardening of the initial a-C:H films takes place via electron beam irradiation. In this paper, this softening and hardening behavior is first experimentally described by using the nano-indentation and field-emission type in-lens SEM. Mechanical properties are precisely measured by this nano-indentation test with aid of composite theory. Nano-structure by nano-columnar alignment via electron beam irradiation is observed by in-lens SEM to define the geometric configuration of nano-columns. In addition, XRR (X-ray reflection method) as well as atomic force microscopy is used to measure the density and stiffness of nano-columnar structure and to trace their change during the duration time in the electron beam irradiation.

11:00am **E2-1-4 Mechanical Behavior and Stability of Self Standing Films and Film on Substrate Systems**, ***M. Ignat*** *(michel.ignat@inpg.fr),* SIMAP Grenoble INP, France*, C. Seguineau,* Novamems France*, A. Chouaf*, ENSEM Casablanca Maroc, France

The essential elements in different technological devices, range nowadays at a submicron scale. At this scale the mechanical properties of thin films, as their response to external solicitations, differ from their bulk counterpart, essentially because their microstructure. As a consequence, there is a permanent lack of information on films behaviour, because microstructure derives from obtention conditions, and these conditions may differ from laboratory, to another.

We present and discuss results coming out from in-situ micro tensile experiments, performed on two sort of "thin" samples: electroplated Copper deposited on a polymer substrate, and self standing thin films of gold and aluminum.

The insitu experiments are performed with a dedicated device and the samples are obtained by lithography as wet and/or dry etching.For the self standing samples (thinner than oneμm) the results are discussed in terms of deduced mechanical parameters, and related to the microstructure.

For the electroplated Copper on polymers, different patterns of the thin Copper electroplated depositions were studied. In this case, we point out the degradation of the film when applying to the system cyclic solicitations; as the effect of singularities, associated to the geometry of the Copper patterns on the substrate.

11:20am **E2-1-5 Procedures and Equations for the Next Generation of Surface Testers Solving the Problem of Pile-Up, Sink-In and Making Area-Function-Calibration Obsolete**, ***N. Schwarzer*** *(n.schwarzer@siomec.de)*, Saxonian Institute of Surface Mechanics, Germany

In the presentation procedures and formulae for a new and more general surface tester concept will be given and discussed. The concept is based on the idea that the next generation of surface testers will provide the means to use all degrees of freedom of movement a probe on a sample surface could perform. Thus, in addition to the ordinary normal stiffness also lateral, tilting and twisting stiffness will be measured and used in the subsequent parameter determination of the investigated materials. It will be demonstrated that such a concept would not only completely solve classical problems like "pile-up" and "sink-in" it would also supersede the need of area function calibration for the indenter tips and allow direct measurement of local intrinsic and residual stresses, anisotropy and many other things, too.

[1]G. M. Pharr, A. Bolshakov: J. Mater. Res., Vol. 17, No. 10, Oct 2002

11:40am **E2-1-6 Interfacial Fracture of Thin Hard Films on Compliant Substrates**, ***N.R. Moody*** *(nrmoody@sandia.gov),* Sandia National Laboratories*, M.S. Kennedy,* Clemson University*, M.J. Cordill,* Erich Schmid Institute*, D.P. Adams, J.A. Emerson,* Sandia National Laboratories*, D.F. Bahr,* Washington State University*, E.D. Reedy*, Sandia National Laboratories

Deformation and fracture of thin films on compliant substrates are key factors constraining the performance of emerging flexible substrate devices. However, the effects of substrate compliance on interfacial film fracture are not well defined. We are therefore studying these effects for sputter deposited thin hard tungsten films on substrates that span two orders of magnitude in compliance. Following film deposition, high compressive film stresses triggered spontaneous delamination and buckling in films on the PMMA substrates with buckle height-to-width ratios much greater than elastic theory predicts due to substrate yielding. As a consequence, interfacial fracture energies calculated using elastic buckle theory are not an accurate representation of the energy for crack growth. We therefore used finite element analysis with a cohesive zone model to simulate interfacial crack growth. When substrate yielding was included in the analysis, calculated energy release rates w ere substantially higher than those calculated using an elastic film-rigid substrate approach. Moreover, calculated height-to-width ratios matched observations. In this presentation we will show that combining experimental measurements for film failure and simulations with cohesive zone models provides a means to accurately describe and predict device performance.

This work is supported by Sandia National Laboratories, a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G3

Atmospheric Plasma, Hollow Cathode, and Hybrid Technologies

Moderator: V.H. Baggio-Scheid, General-Command of Aerospace Technology, A. Leyland, University of Sheffield

10:00am **G3-1 Synthesis of Amorphous Carbon Films using Large-scale Atmospheric Pressure Plasma CVD Equipment**, ***H. Kodama*** *(eco_kodama@newkast.or.jp),* Kanagawa Academy of Science and Technology, Japan*, T. Horiuchi, M. Kumagai,* Kanagawa Industrial Technology Center, Japan*, T. Suzuki*, Keio University, Japan **INVITED**

In order to expand application of amorphous carbon films, it is necessary to realize low-cost and high-speed synthesis to large area. Atmospheric pressure plasma CVD technique using dielectric barrier discharge method is an ideal and suitable method because equipment configuration is simple and also it is easy to enlarge deposition area. We started this study to synthesize high gas-barrier amorphous carbon film for food package at low-cost. Films with hyper-barrier (O_2 transmission: <0.01 cc/m^2/24h/atm) were successfully synthesized by prototype equipment (treatment width: 100 mm) we originally set up. From this result, we found out that it is possible to synthesize functional amorphous carbon films even under atmospheric pressure. Therefore, we constructed two types of large-scale CVD equipment. Treatment width of both equipment, one is a roll-to-roll type and the other is a flat-panel type, is 450 mm. In this presentation, we will introduce structure and properties of the films synthesized by these equipments.

10:40am **G3-3 Atmospheric Pressure Plasma Coatings to Improve the Tribological Behaviour of Rubbers Against Metal Counter Bodies**, ***B. Verheyde*** *(bert.verheyde@vito.be),* VITO-MAT, Belgium*, A. Vanhulsel, D. Havermans,* VITO Belgium*, M. Wangenheim*, IDS Germany

In industries like aeronautics, automotive and pneumatics there is a great need for tailoring and extending the performance and functionalities of rubber-like materials used in sliding and sealing systems. An important driving factor for new developments in the field of tribology are environmental considerations concerning the effects and consumption of greases and lubricants. Therefore there is a steady demand for materials and surface coatings with solid lubricant ability and dry friction capability.

To improve the tribological properties (friction coefficient, wear, noise, lubricant,…) of elastomer parts moving against a metal counter body, an atmospheric pressure plasma treatment of the surface could be an efficient process.

The PLASMASPOT$^{(r)}$is a DBD plasma torch system working at atmospheric pressure and developed at VITO. It can be used for chemical activation or surface modification of different kind of materials as well as for coating by adding a polymerizable precursor to the plasma afterglow.

We applied siloxane based coatings on soft elastomer substrates starting from hybrid organic-inorganic precursors. They were characterized with FTIR, XPS and SEM-EDX. Furthermore different tribological testing against metal counter bodies was performed to have an idea of the improvement of the frictional behaviour and the wear resistance of these coatings. Although soon after deposition cracks appear in the coating, a significant lowering of the initial friction coefficient can be maintained during a long sliding distance.

11:00am **G3-4 Fatigue Life Determination of Plasma Nitrided Medical Grade CoCrMo Alloy**, ***Ö. Bayrak*** *(ozgubayrak@atauni.edu.tr), F. Yetim, A. Alsaran, A. Çelik*, Ataturk University, Turkey

In this paper, the fatigue behavior of plasma nitrided medical grade forged CoCrMo alloy was studied. Since metallic biomaterials are used for implant applications where high and/or cyclic stresses along with corrosive effects of human body are of concern, enhancing mechanical and surface properties of implant alloys is crucial. Plasma nitriding is known as a surface treatment which may improve mechanical properties of applied metallic materials. Information concerning fatigue properties of CoCrMo alloy is quite insufficient and effects of surface treatments on this alloy was not covered. Therefore, investigation of possible effects of plasma nitriding on the CoCrMo alloy was found to be beneficial. Plasma nitriding was implemented at three different temperatures as 600°C, 700°C and 800°C for time intervals of 1, 2 and 4 hours. S-N and S-N-P curves of untreated and nitrided specimens were obtained via axial tension compression fatigue tests. It was found that plasma nitriding treatment reduces the fatigue resistance of forged CoCrMo alloy by the ratios of 7% to 23% depending on the phase structure and hardness of the modified layer which are determined by the treatment parameters.

11:20am **G3-5 Mass and Energy Spectrometry of Plasmas at Atmospheric Pressure**, ***J.A. Rees*** *(arees@hiden.co.uk), D.L. Seymour, T.D. Whitmore*, Hiden Analytical Ltd., United Kingdom **INVITED**

Electrical plasmas operating at atmospheric pressure are now in widespread use for materials processing and in related areas such as surface characterisation. Highly functional surfaces can be deposited on a variety of materials, including textiles, plastics, and polymers. Other processes include medical applications such as the sterilisation of surgical instruments and localised treatment of biological tissues. For surface analysis of a wide range of materials, plasma treatment at atmospheric pressure combined with mass analysis is increasingly used to supplement vacuum SIMS. In all these areas, a variety of plasma sources are in use, including RF 'needle' sources, corona discharges, glow discharge jets, and dielectric barrier discharges. Plasmas are also being incorporated in catalytic conversion systems to allow lower operating temperatures.

It is essential when improving any plasma processing system to have information available on the nature and energies of the active species produced in the discharge, ideally measured at the point where they impact on the target substrate, and of any interaction products. Such measurements can be made using either a quadrupole mass spectrometer to sample neutral species via a capillary inlet tube, or a molecular beam instrument, incorporating an electrostatic energy analyser, to examine positive and negative ions as well as neutral species. Typical data for a number of different plasma sources will be described and correlated with measured properties of surfaces exposed to the plasmas. Preliminary data for a micro-reactor combining catalysis with a surface barrier discharge will also be outlined.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B2-2

Arc and E-Beam Coatings and Technologies
Moderator: V.I. Gorokhovsky, Arcomac Surface Engineering, LLC, J. Vetter, Sulzer Metaplas GmbH

1:30pm **B2-2-1 Trends and Applications of Cathodic Arc Evaporation**, ***O. Zimmer*** *(otmar.zimmer@iws.fraunhofer.de),* Fraunhofer Institute for Materiasl and Beam Technology, Germany, *V. Weihnacht, H.-J. Scheibe*, Fraunhofer Institute for Materials and Beam Technology, Germany **INVITED**

Cathodic vacuum arc evaporation (CVAE) is in industrial use since about 30 years. The main fields of applications are the cost effective deposition of hard protective coatings for tools and components as well as decorative coatings for faucets or plastic parts for example. Other fields are coatings with highest demands on film structure and mechanical properties for applications in micro technology. The different applications require different growth conditions to get the coating quality and to realize a sufficient productivity. These requirements can only be full filled by using optimized arc sources and advanced plasma cleaning processes. High rate processes for machine elements and high precision processes for micro applications demand different source concepts. Recent developments in evaporator technology enable the high rate deposition of advanced nano composite hard coatings for high end cutting applications. Also possible is the deposition of thicker films due to innovations in film structure. Cylindrical or rectangular cathodes with new concepts of weak and stronger magnetic fields are used to achieve highest plasma activation and low particle quantity. The using of pulsed arc sources is the preferable way for the deposition of hard amorphous carbon coatings (ta-C) in an industrial environment. At least plasma filtering is an additional tool to adjust film properties and opens the door for a lot of new applications.

In the paper current developments in the area of arc evaporators, plasma filters, pulsed processes and film properties are discussed concerning technical aspects and applications.

2:10pm **B2-2-3 Zirconium and Aluminum Arc Behavior and Ion Current Generation During Metal and Oxide Deposition**, ***I. Zukerman*** *(idozukerman@gmail.com),* Tel Aviv University / NRC-Negev, Israel, *V.N. Zhitomirsky,* Tel Aviv University, Isreal, *A. Raveh,* NRC-Negev, Israel, *R.L. Boxman,* Tel Aviv University, Israel, *S.K. Kim*, University of Ulsan, Korea

The operation of a vacuum arc plasma gun with Zr and Al truncated cone cathodes was studied. The arc operation and the cathode spot motion was studied in vacuum and in a 0.2-1 Pa oxygen background. The process parameters (arc current and voltage, and background gas pressure) as well as the ion current at the substrate, were continuously recorded during arcing. The influence of the deposition parameters arc current (25-175 A), axial magnetic field strength (0-10 mT), and oxygen pressure, on the arc behavior, electrode poisoning, ion flux and deposition rate was studied. It was found that the number of the cathode spots on the truncated cone-shaped cathodes in a confining magnetic field increased with the arc current Ia. However, with increasing Ia the spots tended to operate on the cathode conical periphery rather than on the cathode working base. Also, the ion current, and thus coating deposition rate increased as the arc current increased to 75 and 100 A for Al and Zr cathodes, respectively, and then sharply decreased. The deposition rate in the centers of the plasma beams estimated from the saturation ion current was 1.2 and 2.8 μm/min for Al (Ia=75 A) and Zr (100 A), respectively. During operation in oxygen, the arc behavior and the ion current changed with time. For the Zr arc at oxygen pressure $\geq$ 0.5 Pa, the ion current at the substrate decreased with time and the arc voltage increased, probably, due to the electrode poisoning.

2:30pm **B2-2-4 Phase Formation in Oxide Layers Synthesized by Pulsed Arc Evaporation**, *M. Doebeli,* Ion Beam Physics, Paul Scherrer Institut and ETH Zuerich, Switzerland, *A. Dommann,* Centre Suisse d'Electronique et de Microtechnique Neuchâtel, Switzerland, *J. Herrán,* Centre Suisse d'Electronique et de Microtechnique Neuchâtel, Switzerlandand CEIT and Tecnun San Sebastian, Spain, *E. Kalchbrenner,* OC Oerlikon Balzers AG, Liechtenstein, *A. Neels,* University of Neuchâtel, Switzerland, ***J. Ramm*** *(juergen.ramm@oerlikon.com), H. Rudigier,* OC Oerlikon Balzers AG, Liechtenstein, *J. Thomas,* IFW Dresden, Germany, *B. Widrig*, OC Oerlikon Balzers AG, Liechtenstein

Pulsed arc evaporation (P3eTM) has been proven to synthesize a number of metallic oxides in a stable and reliable industrial deposition process. In previous reports, it was also shown, that this process allows the deposition of oxides which consist mainly of high temperature phases, e.g. the corundum-type structure for the solid solutions of Al-Cr-O. The formation process of high temperature phases in the Al-Cr-O material system has been investigated in more detail for the metallic interface consisting of Cr and Al-Cr. Investigations by transmission electron microscopy indicate a quasi-epitaxial interface between the Cr and the Al-Cr layers and an abrupt and sharp interface to the oxide layer. According to the analysis, the corundum-type structure in the Al-Cr-O layer is stabilized already in the vicinity of the interface. The crystallite size of the corundum-type structure, however, can be tailored by the process parameters and the oxygen partial pressure during the ar c evaporation process. X-ray diffraction analysis in combination with electron diffraction suggests oxide layers with a single phase besides minor metallic constituents in form of droplets. The analysis results are compared with the ternary Al-Cr-O phase diagram and possible phase formation mechanism are discussed.

2:50pm **B2-2-5 Influence of Bias Pulsing on Structure and Properties of Arc-Evaporated $(Al_{1-x}Cr_x)_2O_3$ Hard Coatings**, ***M. Pohler*** *(markus.pohler@unileoben.ac.at), G.A. Fontalvo, R. Franz,* University of Leoben, Austria, *D. Kurapov,* OC Oerlikon Balzers AG, Liechtenstein, *C. Polzer,* PLANSEE Composite Materials GmbH, Austria, *C. Mitterer*, University of Leoben, Austria

Thermodynamically stable α-Al_2O_3 coatings produced by chemical vapor deposition are widely used on tools for high performance cutting operations due to their outstanding chemical inertness and high temperature resistance. During the last years, crystalline α-Al_2O_3 was successfully synthesized by physical vapor deposition techniques which allow a reduction of deposition temperature from ~1000 °C to ~600°C. Recent studies using cathodic arc evaporation showed that the addition of Cr is beneficial for the synthesis of a crystalline α-phase. These α-$(Al_{1-x}Cr_x)_2O_3$ coatings are deposited at temperatures below 600°C using pulsed bias voltage. The aim of this work was to investigate the effect of varying the duty cycle of the pulsed bias on structure and properties of $(Al_{1-x}Cr_x)_2O_3$ coatings. All coatings were deposited in an industrial scale cathodic arc evapor ation system using powder metallurgically produced targets with the composition $Al_{50}Cr_{50}$ and $Al_{70}Cr_{30}$. Synthesis was done in pure oxygen atmosphere at a substrate temperature of 550°C. An increase of the duty cycle of the symmetric bipolar pulsed bias from 70 % to 90 % increased the crystallinity of the coatings, as analyzed by X-ray diffraction. The grain size of, e.g., $(Al_{0.5}Cr_{0.5})_2O_3$ increased from ~60 nm to ~110 nm. This results in an increase of the hardness from ~20 to ~33 Gpa, as measured by nanoindentation. In summary, the variation of the duty cycle presents a feasible way to improve the properties of $(Al_{1-x}Cr_x)_2O_3$ coatings.

3:10pm **B2-2-6 Deposition of ZrO_2/Al_2O_3 Thin Films by Cathodic Arc Plasma Deposition**, ***S.K. Kim*** *(skim@ulsan.ac.kr), V.V. Le,* University of Ulsan, Korea, *J.W. Lee,* KAIST, Korea, *V.N. Zhitomirsky, R.L. Boxman*, Tel Aviv University, Israel

Thin films of ZrO_2/Al_2O_3 thin films were deposited on SKD 11 tool steel substrate using Zr and Al cathodes by a cathodic arc plasma deposition system. The substrates were mounted on a rotating holder which alternatively exposed them to plasma from the two cathodes. The influence of the Zr and Al cathode arc currents and the substrate bias on the mechanical and the structural properties of the films were investigated. The hardness of the film decreased with the increase of Zr cathode current from 60 A to 80 A. The hardness of the film increased with the increase of bias voltage up to -200 V and then decreased with further increase of the negative bias. The film structure was elucidated by HRTEM microscopy.

3:30pm **B2-2-8 Hollow Cathode Plasma Assisted AlTiSiN-Thin Film Deposition by Vacuum Arc P+-Technology for Advanced Wear Protection of Tools**, ***M. Holzherr*** *(martin.holzherr@vtd.de), T. Schmidt, M. Falz, K.-D. Steinborn*, VTD Vakuumtechnik Dresden GmbH, Germany

In the past the hollow cathode plasma assisted AlTiN- and AlCrN-thin film depositions had been carried out by means of simultaneous operation with vacuum arc evaporation sources. As a matter of fact, it could be verified that the additional hollow cathode plasma (P+-technology) results in advanced thin film properties such as higher hardness, lower droplet density and improved wear resistance properties. Hence, the main application is wear protection of tools under extremely hard cutting conditions. This means high cutting speed and feed with dry or low lubricant machining. Although by the addition of silicon to AlTiN- or AlCrN-standard vacuum arc coatings a phase separation and the forming of nanocomposites TiAlN/Si_3N_4 - or AlCrN/Si_3N_4 -coatings with super hardness and excellent temperature stability were reported, the goal of the presented application of the newly developed P+-technology is to further improve the deposition conditions as well as to optimise the AlTiSiN- thin film properties.

Examples are the reduction of the deposition temperature and the increase of brilliance of the coatings. Lower deposition temperatures enlarge the range of tool substrates suitable for coating and yields to an economical reduction of process times. In this contribution a comparison of AlTiSiN-thin film coatings deposited by standard vacuum arc sources and the P+-process will be presented. AlTiSi-target materials with three different Si-at %-contents (5, 10 and 15 at %) had been used for the thin film depositions. The experiments were arranged in a medium sized industrial batch coater DREVA 600 whereas this system is provided with two hollow cathode plasma sources and up to twelve magnetic steered vacuum arc sources. The effects of modified Si- contents in AlTiSi-target materials and the simultaneous operation of the two hollow cathodes on plasma parameters during the vacuum arc coating process were examined by the use of optical emission spectroscopy (O E S-) method and langmuir-probe. The influence of the measured plasma parameters on selected AlTiSiN-thin film properties will be demonstrated in the standard as well as in the P+-process. The results of the oxidation behaviour and hardness modifications after annealing experiments as well as drill test will be presented.

3:50pm **B2-2-9 Synthesis and Mechanical Properties of CrN_x Coatings Deposited by Arc Ion Plating**, ***M. Zhang*** *(m.zhang@live.com), K.H. Kim,* Pusan National University, Korea*, G. Lin,* School of Physics and Optoelectronic Technology, China*, C. Dong*, School of Material Science and Engineering, China

Compositional gradient CrN_x coatings were fabricated using arc ion plating by varying N_2 flow rate from 0 to 120 SCCM. The effects of negative substrate bias and deposition temperature on the coating growth and properties were systematically investigated with XRD, GDOES, SEM, TEM, Nanoindention, stress, adhesion and wear tests. The results showed that gradient CrN_x coatings mainly crystallized in fcc rocket-salt NaCl structure. Along the thickness direction of gradient CrN_x coatings, the content of ceramic phase increases and that of metal phase decreases gradually. By increasing substrate bias, the microstructure of the coatings evolved from an apparent columnar structure to equiaxed one. The coatings deposited at 150°C completed this evolution at a bias of -100V; while -200V in the case of 300 °C. The maximum hardness of 33GPa was obtained from the coatings deposited at a bias of -50V with a residual stress of -3.1GPa. It was also found that the adhesion strength and the wear resistance of gradient CrN_x coatings were better than that of homogeneous CrN coatings.

4:10pm **B2-2-10 Characterization of Worn Ti-Si Cathodes used for Reactive Cathodic Arc Evaporation**, ***J. Zhu*** *(zhu@ifm.liu.se), A. Eriksson, N. Ghafoor,* Linköping University, sweden*, M. Johansson,* SECO Tools AB, Sweden*, F. Giuliani,* Linköping University, Sweden*, J. Sjölén,* SECO Tools AB, Sweden*, L. Hultmann, J. Rosén, M. Odén*, Linköping University, Sweden

Cathodic Arc Evaporation (CAE) is a common PVD technique to deposit hard coatings. As the source material for the coatings, the cathode is one of the key factors for this technique. It is widely acknowledged that a compound layer, which influences the plasma composition and charge state but also the erosion rate, forms on the cathode surface when used in a reactive gas. Up to now, there are only a limited numbers of reports on this layer. We have used SEM, XRD, FIB and TEM-EDX/EELS, to investigate the evolving microstructure and composition of the surface compound layer of the worn Ti1-xSix cathodes (x=0, 0.1, 0.2), which are employed to produce commercial TiSiN hard coatings. Virgin $Ti_{1-x}Si_x$ cathodes, powder metallurgical fabricated, contain a hexagonal solid solution between Ti and Si. The cathode containing 20% Si has a two-phase structure with hexagonally shaped Ti5Si3 grains surrounded by fine Ti/Ti_5Si_3 eutectic microstructure. After arcing at a current of 60A and in a pure N2 atmosphere in an industrial-scale CAE system, the cathode is covered with a 4-10 µm thick compound layer on the surface. This compound layer consists of two distinctly different microstructures: (i) nanocrystalline equiaxed grains which are either Ti or Si rich, and on top of these (ii) columnar grains where the grain boundaries are depleted of Si. The nitrogen present in this layer and its role in forming new compounds is discussed. The arc erosion is observed to occur preferentially from Ti_5Si_3 grains over the eutectic, which results in a higher roughness of the cathode surface. The influence of the preferential erosion on the chemical composition in the plasma is also addressed.

4:30pm **B2-2-11 Structure and Composition of Arc Deposited $(Ti_{1-x}Si_x)(C_{1-y}N_y)_z$ Coatings**, ***A. Eriksson*** *(ander@ifm.liu.se), J. Zhu, M. Odén,* Linköping University, Sweden*, M. Johansson, J. Sjölén,* SECO Tools AB, Sweden*, L. Hultman, J. Rosén*, Linköping University, Sweden

The Ti-Si-C-N system is a promising candidate for wear resistant coatings. An increased understanding of the material as well as of the arc evaporation process is of importance for future applications and process development. Coatings in the quaternary Ti-Si-C-N system have been deposited using industrial scale cathodic arc evaporation. Two strategies were followed: (i) reactive arcing from conventional Ti-Si targets in mixtures of gaseous CH_4 and N_2, and (ii) reactive arcing from ternary Ti-Si-C targets, including Ti_3SiC_2 (Maxthal 312™) compound target, in N_2-atmosphere at various pressures. The as-deposited coatings resulting from the two methods are compared with respect to composition, microstructure and mechanical properties as determined by XRD, SEM, TEM, ERDA and nano-indentation. When arcing from ternary cathodes, nanocrystalline coatings were obtained, with Si and C concentrations of up to 12 and 27 at%, respectively. Microstructure and composition were found to be highly dependent on N_2-pressure. For the binary target approach, similar phase structures were obtained, but with generally lower level of C and N incorporation.

Hard Coatings and Vapor Deposition Technology
Room: Royal Palm 1-3 - Session B3-2

CVD Coatings and Technologies
Moderator: M. Pons, SIMAP, Grenoble-INP, CNRS, H. Holzschuh, Walter AG

1:30pm **B3-2-1 Mechanical Behavior of PECVD Silicon Oxide, Oxynitride and Nitride: A Comparative Micro-Cantilever Deflection Study**, ***K. Matoy*** *(kurt.matoy@k-ai.at),* KAI Kompetenzzentrum automobil- und Industrie-Elektronik GmbH, Austria*, R. Pippan,* Erich Schmid Institute Leoben, Austria*, G. Dehm,* University of Leoben, Austria*, H. Schönherr, T. Detzel*, Infineon Technologies AG, Austria

The mechanical behavior of plasma enhanced chemical vapour deposited (PECVD) silicon oxide, oxynitride and nitride films was investigated. Hardness, Young's modulus, fracture stress and fracture toughness were determined by nanoindentation and the micro-cantilever deflection technique. The micro-cantilever experiments revealed linear elastic behavior of all PECVD materials. Up to failure bending strength of 4.2GPa for silicon oxide, 6.5GPa for silicon oxynitride and 7.9GPa for silicon nitride were observed. The fracture toughness values obtained from the bending experiments scale between 0.7MPam^1/2 and 1.6MPam^1/2 depending on the chemical composition. Furthermore, a correlation between the fracture properties and the refractive index was observed, which may offer the possibility to estimate the mechanical properties by the refractive index.

1:50pm **B3-2-2 Properties and Tribological Behaviour of CVD TiBN-Coated Cutting Tools**, ***D. Stiens*** *(dirk.stiens@walter-tools.com),* Walter AG, Germany*, I. Dreiling,* University of Tuebingen, Germany*, H. Holzschuh,* Walter AG, Germany*, T. Chasse*, University of Tuebingen, Germany

CVD Titanium Boronitride coatings show interesting results in cutting tests. To understand the wear behaviour Pin on Disk tribological model tests of the coatings have been performed at ambient and elevated temperature against 100Cr6 steel and Aluminium as counter materials. Coatings with different Boron contents have been studied. The samples from cutting tests and tribological experiments were subsequently investigated by Laser Raman Spectroscopy in order to find products of tribochemical reactions. Different Titanium Oxide Phases which originate from coating oxidation have been detected. Also Iron borate phases which originate from reactions with the friction partner were found. The influence of laser heating has been systematically studied by variation of the Laser power. To estimate the local temperature samples have been tempered at different temperatures in air. The oxidation products induced by tempering were investigated by Raman at low Laser power and compared to those obtained by Laser-induced oxidation.

2:10pm **B3-2-3 Experimental Thermodynamics for the Evaluation of ALD Growth Processes**, ***E. Blanquet*** *(elisabeth.blanquet@simap.grenoble-inp.fr), P Violet,* SIMAP, Grenoble-INP, CNRS, France*, D. Monnier,* STMicroelectronics, France*, I. Nuta,* SIMAP, Grenoble-INP, CNRS, France*, C. Chatillon*, SIMAP, Grenoble-INP, CNRS **INVITED**

Atomic Layer Deposition (ALD) is applied in many advanced technologies that require control of film properties in the nanometer or sub-nanometer scale. ALD finds use mainly in semiconductor applications but also in solar energy, catalysis, micro-systems and biological applications. ALD films can be used to improve oxidation resistance, corrosion resistance, to improve electrical or optical properties … Atomic layer deposition (ALD) , which is based on the sequential self-limiting surface reactions from generally two gaseous precursors, provides an ideal technique for depositing ultrathin and conformal films.

To develop and optimize an ALD process for a new material requires knowledge of the reactions mechanisms. Most of the studies on ALD modeling have been dedicated on the two precursors surface reactions. However, the most commonly use of organometallic molecule precursors with the occurrence of thermal decomposition requires an understanding of the gas-p hase chemical reactions. Information on physical and chemical behavior of this kind of precursor is scarce and namely species that were transported to the ALD deposition chamber are generally unknown. Mass spectrometer coupled with a Knudsen effusion cell has already proved to be a powerful tool for studying the stability of gaseous molecules, thermodynamics of condensed phases and, more generally vaporization processes. The identification and quantification of the actual species present in the gaseous phase can be determined using a specific system composed of a Knudsen effusion cell coupled with a mass spectrometer. This presentation describes the application of such approach for TaN and ZrO_2 ALD deposition processes.

2:50pm **B3-2-5 Effect of Air Exposure on Cu Diffusion Barrier Properties of TaN Thin Films Grown by Atomic Layer Deposition**, ***O.H. Kim*** *(okim@che.ufl.edu), D.J. Kim, K.C. Kim, H.M. Ajmera, T.J. Anderson, J. Koller, L. McElwee-White, D.P. Norton*, University of Florida

The effect of exposing TaN diffusion barriers to air was studied by characterizing the interfacial reactions of the Cu/TaN/Si stack with and without air exposure as a function of annealing temperature. TaN thin films were grown on Si (100) substrate by atomic layer deposition (ALD) using $(Et_2N)_3Ta=NBu^t$ [tert-butylimido tris(dimethylamido) tantalum, TBTDET] and NH_3 as precursors. TBTDET exposure time and growth temperature were optimized to give self-limiting adsorption. Cu thin films were subsequently deposited on TaN/Si by chemical vapor deposition (CVD) using $(tmvs)Cu^I(hfac)$ as a single source precursor. To investigate the effect of exposure of the TaN barrier film to air before deposition of the copper layer in the stacks, the TaN/Si samples were either directly transferred to the Cu-CVD chamber under vacuum or first exposed to room temperature air before loading into the chamber. An interfacial layer of TaO_xN_y was observ ed between Cu and TaN diffusion barrer in the air exposed samples. As a diffusion barrier test, the Cu/TaN/Si structures were thermally annealed under nitrogen at elevated temperature for 30 min. The barrier quality of 4 nm TaN films was evaluated by observing the breakdown temperature of annealed Cu/TaN/Si stacks using X-ray diffraction, sheet resistivity measurements, and etch-pit tests. Interestingly, it was found that the breakdown temperatures of air exposed films are higher than those vacuum transferred. The TaO_xN_y interfacial layer appears to have a beneficial role in preventing Cu transport.

3:10pm **B3-2-6 DLI-CVD of M-TiO_2 (M = Ag, Cu) Antibacterial Thin Films**, *J. Mungkalasiri,* Cirimat - Cea, France*, L. Bedel, F. Emieux,* CEA, France*, J. Doré, F. Renaud,* Nosoco.tec, France*,* ***F. Maury*** *(francis.maury@ensiacet.fr)*, CNRS ENSIACET, France

M-TiO_2 (M = Ag ; Cu) nanocomposite layers were grown by pulsed direct liquid injection chemical vapor deposition (DLI-CVD) on stainless steel with the goal to produce bactericidal surfaces. Liquid solutions of titanium tetra-isopropoxide (TTIP), silver pivalate (AgPiv) and copper tetramethyl heptadionate (Cu(tmhd)2) in appropriate solvents were used as Ti , Ag and Cu molecular sources, respectively. The deposition temperature was typically 683 K and the total pressure was 800 Pa. Monodisperse Ag nanoparticles (5-10 nm) are embedded in an anatase matrix. A bactericidal behavior determined by the JIS Z 2801 standard test was found for Ag-TiO_2 films with a silver content lower than 1 at.%. An increase of the Ag content of the films increases the numbers of metal particles rather than their size. By contrast, when Cu is used as antibacterial agent, it is incorporated as metal particles with a large size distribution ranging from 50 to 200 nm depending on the copper content of the films. The influence of the growth conditions on the structural features and the antibacterial properties of the thin films is reported and discussed.

3:30pm **B3-2-7 Evaluation of the Tungsten Diphenylhydrazido Complex $Cl_4(CH_3CN)W(NNPh_2)$ as a Precursor for CVD of WN_xC_y**, ***D.J. Kim*** *(dojunida@ufl.edu), O.H. Kim, T.J. Anderson, J. Koller, L. McElwee-White, L.C. Leu, D.P. Norton*, University of Florida

The tungsten diphenylhydrazido complex $Cl_4(CH_3CN)W(NNPh_2)$ (1) was used to deposit tungsten carbonitride (WN_xC_y) by metal-organic chemical vapor deposition (MOCVD) in the temperature range 300 to 700 °C. The effect of deposition temperature on the film microstructure, surface morphology, chemical composition, bonding states, growth rate, electrical resistivity, lattice parameter, and grain size was studied. The microstructure of films deposited using 1 was amorphous below 500°C and polycrystalline above this temperature. Between 500 and 700°C the lattice parameter varied from 4.15 to 4.20 Å, while the average grain size increased from 25 to 55 Å. XPS results for films deposited using 1 indicate that W is primarily bonded to N and C for films deposited over the entire deposition temperature range. The growth rates of films deposited using 1 varied from 1.0 to 25.4 Å/min and showed Arrhenius behavior in the rang e 300 to 450°C with an apparent activation energy of 0.49 eV. The barrier quality of Cu/WN_xC_y/Si stacks was evaluated using XRD patterns and cross-sectional TEM images along with EDS. The integrity of both Cu/WN_xC_y and WN_xC_y/Si interfaces was retained after annealing at 500 °C for 30 min. XRD spectra revealed no Cu_3Si peaks and EDS qualitative analysis showed a sharp decrease in the Cu K_α peak at the Cu/WN_xC_y interface. Hence, WN_xC_y films deposited at 400°C are viable materials to serve as a Cu diffusion barrier. The properties of films deposited with 1 are compared to those deposited from tungsten piperidylhydrazido complex $Cl_4(CH_2CN)W(N\text{-}pip)$ and tungsten imido complexes $Cl_4(CH_3CN)W(NR)$ (R = Ph, ^{i}Pr, and allyl)[1] to provide insight into the effect of incorporating hydrazido and imido ligands on film properties.

[1] O. J. Bchir, K. M. Green, H. M. Ajmera, E. A. Zapp, T. J. Anderson, B. C. Brooks, L. L. Reitfort, D. H. Powell, K. A. Abboud, L. McElwee-White, J. Am. Chem. Soc. 127 (2005) 7825-7833.

3:50pm **B3-2-8 Silicon Carbide CVD for Polycrystalline Growth: Thermodynamic Evaluation vs. Film Morphology**, *J. Trevarthen,* SIMAP, Grenoble-INP, CNRS, France*,* ***A. Claudel****, R. Martin,* ACERDE, France*, G. Chichignoud, M. Morais,* SIMAP, Grenoble-INP, CNRS, France*, E. Blanquet (elisabeth.blanquet@simap.grenoble-inp.fr),* SIMAP, Grenoble-INP, CNRS, Framce*, M. Pons*, SIMAP, Grenoble-INP, CNRS, France

Polycrystalline silicon carbide (SiC) is widely used in a range of commercial applications due to its resistance to extreme conditions. This resistance is improved by increasing the crystallinity and purity of the silicon carbide.

High temperature Chemical Vapour Deposition as a process allows greater control over a wider range of reaction parameters (temperature, pressure, ratio of injected gaseous precursors) when compared to other techniques such as Physical Vapor Transport. The variation of these parameters allows control over properties of the final material such as its morphology, crystalline quality and composition. As such it is preferable for the growth of pure, highly crystalline SiC.

Thermodynamic calculations have been performed on a CVD system employing H_2 and methyltrichlorosilane (MTS) for a temperature range of 1200-1600°C, a pressure range of 150-250 mbar and H_2/MTS ratios of 5-150. The corresponding data was u sed to calculate the theoretical supersaturation of the gaseous system at equilibrium and the composition of the solid deposit also at equilibrium, which were correlated to experimental data obtained for the HT-CVD process within these ranges of reaction parameters. Characterization techniques such as FEG-SEM, EBSD, XRD, profilometry were used to evaluate film roughness and film morphology, especially grain size and orientations.

4:10pm **B3-2-9 Adhesion and Homogeneity of a-C:H:Si Films Deposited in a Modified Plasma Nitriding System for Industrial Application**, ***C. Forsich*** *(c.forsich@fh-wels.at), D. Heim,* University of Applied Sciences, Austria*, T. Mueller*, Rubig GmbH & Co KG Anlagentechnik, Austria

Si doped diamond-like carbon coatings (a-C:H:Si) have attracted significant attention owing to their desirable properties, e.g. chemical inertness, good corrosion resistance, low friction coefficient and reduced wear. These properties make a-C:H:Si films interesting for industrial application on machine parts and tools. a-C:H:Si deposition on plasma nitrided (with compound layer) and on oxidized steel substrates which were prior nitrided was carried out in a commercial available DC plasma nitriding apparatus in one run. This technique has the potential to upscale the a-C:H:Si process to dimensions up to several meters whereas parameters are used comparable to plasma nitriding, such as pressure, gas flows and temperature. In contrast to PVD (physical vapour deposition) it is not possible to deposit metallic interlayers consisting of Cr and Ti. Coatings with beneficial tribological properties were obtained resulting in a friction coefficient down to 0.05. Rockwell C tests exhibited excellent coating adhesion with pre-treatment prior deposition. However, in some cases scratch tests resulted in critical loads up to 10-15 N. Several interfaces and different gradient films were deposited in order to further improve the adhesion behaviour. The homogeneity of the a-C:H:Si films deposited on complex geometric substrates strongly depends on the pressure and gas mixture used in the process. Therefore, parameter variations were systematically carried out. The process gases H_2, Ar, C_2H_2, CH_4 as well as HMDSO (hexamethyldisiloxane) and TMS (tetramethylsilane) were regulated by mass flow controllers and fed into the vacuum chamber. The determination of the composition of the a-C:H:Si films and the interlayers was carried out using GDOES (glow discharge optical emission spectroscopy). The hardness was evaluated by a nano-indenter and the topography of the layers

was characterized by means of SEM (scanning electron microscopy) whereas t h e tribological properties were assessed with a ball-on-disc test.

4:30pm **B3-2-10 The Effect of Substrate Temperature of High Energy Photon Treatment on Oxygen Doped Silicon Carbide**, ***C.-C. Huang*** *(jjhuanga@csu.edu.tw), J.L. Huang, , H.H. Lu*, National Chen-Yi University, Taiwan

Changes in the configuration and properties of a commercial oxygen doped silicon carbide (SiCO) are examined as a function of photon treatment substrate temperature. The treatment results in a chemical configuration rearrangement in which the Si-O stretching and Si-C bending increase. A direct consequence of the configuration and structural evolution is a change in the electrical properties. A correlation is made between configuration and structure changes in the treatment and the resulting electrical properties. Fourier Transform Infrared spectroscopy (FTIR) was used to quantify the increased Si-O and Si-C and reduced Si-H as a result of increased treated substrate temperature. As a consequence, chemical and configuration changes affect the electrical properties of SiCO films after treatment. The dielectric constant and leakage current density were determined by the metal insulator silicon (MIS) method. High substrate temperature was shown to greatly improve the dielec tric constant and leakage current. A high substrate temperature contributes to the formation of a low polarity structure of O1/2-Si(CH_3) in the treatment process, which makes the dielectric constant recover. It also helps eliminate the dangling bond to reduce the leakage current density of the SiCO films. The treatment time does not affect the final configuration of SiCO films. As treatment time increases, dielectric constant and leakage current increases and becomes saturated.

4:50pm **B3-2-11 Molecular Beam Mass Spectrometry and Modelling of CH_4-CO_2 Plasmas in Relation with Polycrystalline and Nano-Smooth Diamond Deposition**, *T. Gries,* ***L. Vandenbulcke*** *(vanden@cnrs-orleans.fr), S. de Persis, C. Met,* ICARE-CNRS, France*, O. Aubry,* GREMI-Polytech/Université d'Orléans, France*, J.L. Delfau*, ICARE-CNRS, France

CH_4 - CO_2 plasmas have been studied by emission spectroscopy, microwave interferometry, Langmuir probing and molecular beam mass spectrometry (MBMS). MBMS allowed us studying the variations of the concentration of both stable species (H_2, CH_4, CO, CO_2, C_2H_2, …, C_6H_6, C_8H_6) and radicals (H, OH, CH_3, C_3H_3, C_3H_5, C_6H_5…) as a function of the inlet composition, total flow rate, power density injected in the plasma and distance from the substrate. The other techniques have been used to deduce the variations of the plasma parameters like the gas kinetic temperature, the electron density and the electron temperature. A modelling of the plasma kinetics is based on a combustion mechanism which incorporates more than 150 species and 850 reactions and a specific dissociation mechanism including about 50 species. The calculation results are obtained from a CRESLAF version of the CHEMKIN II package which has been slightly modified for incorporating the electron dissociation mechanism and conditions of constant plasma parameters in a plasma column flowing inside a tubular reactor. This 2D-model takes into account the coupled hydrodynamics of the gaseous species, gas phase chemistry and surface recombination at the reactor wall. The results are compared to the MBMS experimental results of the gaseous composition in the plasma as a function of various parameters. These experimental and modelling studies are used for correlating the influence of the relative concentration of important gaseous species in the plasma (especially radicals) to the deposition domain, the growth rate, the structure (polycrystalline or nano-smooth) and the diamond quality of the deposits. The influence of the concentrations of both hydrocarbon radicals and H and OH species in the plasma is especially evidenced together with their experimental concentration gradients in front of the growing surface. The growth of nano-smooth diamond films, with roughness in the 7-30 nm range, relatively to polycrystalline ones is explained and the variation of their intrinsic properties correlated.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D2-2

Diamond and Diamond-Like Carbon Materials

Moderator: O. Shenderova, International Technology Center, R. Hauert, Empa

1:30pm **D2-2-1 Raman Analysis of DLC Coated Engine Components with Complex Shape: Understanding of Wear Mechanisms**, ***O. Jarry***, Sorevi*, C. Jaoul (jaoul@ensil.unilim.fr), P. Tristant, M. Colas, T. Merle-Mejean, C. Dublanche-Tixier*, University of Limoges, CNRS, SPCTS, France

DLC films such as amorphous hydrogenated carbon films (a-C:H) are presented as one of the best solutions to reduce friction in internal combustion engines and increase the lifetime of the mechanical parts, e.g. the valve train components. Many authors have already carried out tribological tests on flat samples including Raman analysis of the wear tracks. However very few studies dealt with tests realized in the real engine conditions, although DLC coatings are very sensitive to the working conditions. The originality of this work was to perform Raman analysis directly on complex-shaped mechanical parts after being used in real engines.

DLC films were deposited by r.f. PACVD in an industrial scale reactor. Adhesion of the films was improved by the interlayer system between the steel sample and the DLC film. First, an overall characterization of the DLC films was carried out. Structure of the films was compared using Raman spectroscopy. Then tribological tests were conducted in unlubricated and lubricated conditions. Raman analysis of the wear tracks was also realized to check the stability of the DLC structure. Finally, Raman spectroscopy was performed directly on real parts with complex shapes. Mechanical parts showing different wear morphology were analyzed. A particular attention was paid to interactions with the Raman signal of the interlayer system. Different kinds of wear mechanisms were identified using this Raman analysis. Unusual high wear rate of the DLC layer were explained by the graphitization of the DLC structure. The knowledge of the various wear mechanisms allowed us to improve the wear resistance of the parts.

1:50pm **D2-2-2 Hydrogen-Free Hard Amorphous Carbon Coatings: Properties by Cathodic Vacuum Arc Evaporation and Magnetron Sputter Deposition**, *M. Stueber (michael.stueber@imf.fzk.de),* Forschungszentrum Karlsruhe, Germany*,* ***J. Vetter***, Sulzer Metaplas GmbH, Germany*, H. Leiste, S. Ulrich, C. Ziebert,* Forschungszentrum Karlsruhe, Germany*, J. Mueller, G. Erkens*, Sulzer Metaplas GmbH, Germany

Basic research and industrial development of diamond like carbon coatings (DLC) have been carried out since decades with regard to their exceptional properties. Different types of DLC coatings (usually hydrogenated amorphous carbon coatings, a-C:H:Me, a-C:H:X) deposited by reactive processes in carbon gas atmospheres have, therefore, been adapted in a large number of industrial applications. Significant progress in the PVD deposition of hard, hydrogen-free amorphous carbon coatings is achieved by applying both tailored magnetron sputtering and improved cathodic vacuum arc processes. Coatings of high surface quality, reduced droplet contamination, low intrinsic stress values, excellent adhesion and hardness values up to 60 GPa with a thickness up to several microns are deposited on silicon and cemented carbide substrates with a new Metaplas MZR304RF hybrid PVD deposition equipment. Results on the laboratory scale deposition experiments, on the thin film microstructure an d properties will be presented. Constitution analyses of the coatings, in example by Raman spectroscopy, and various mechanical characterization methods including nanoindentation, will be described. Scale-up measures and the technical potential of the films, for example in tooling applications and components, will be discussed.

2:10pm **D2-2-3 Non-Destructive Characterization of Carbon Films**, ***A. Ferrari*** *(acf26@hermes.cam.ac.uk)*, University of Cambridge, United Kingdom **INVITED**

The availability of reliable characterization tools for carbon films down to a few atomic layers' thickness is one of the most decisive factors for technology development and production. In particular, non-destructive techniques are preferred. I will review the use of x-ray reflectivity, surface acoustic waves, and Raman spectroscopy to characterize carbon films in terms of density, thickness, layering, elastic constants, roughness, structure, and chemical composition. Raman spectroscopy, in particular, allows the assessment of most of the materials properties, even if indirectly. The measurement of thermal conductivity of carbon films will also be discussed.

2:50pm **D2-2-5 Characterisation of a Plasma Beam Source During Duplex Treatment of High Speed Steel for Depositing DLC Coatings**, ***M. Fenker*** *(fenker@fem-online.de), M. Balzer, N. Bermayer,* Forschungsinstitut Edelmetalle & Metallchemie, Germany*, M. Rinke, M. Stüber, S. Ulrich, J. Ye*, Forschungszentrum Karlsruhe GmbH, Germany

Plasma characterisation methods have been utilised to study a duplex process, consisting of plasma nitriding of high speed steel (HSS) and subsequently depositing a DLC (a-C:H) coating. Both treatments are performed by using a plasma beam source. For the plasma nitriding a gas mixture of N_2/H_2 was used. The nitriding duration was varied and the substrate temperatures ranged from 400 to 500 °C. The subsequent DLC deposition was performed at 100 °C with acetylene as precursor gas. The formation of a white layer could be avoided by suitable adjustment of the process parameters, resulting in well-adherent DLC coatings for nitriding depths ≥ 20 µm. Plasma characterisation methods – like optical emission spectroscopy, mass spectroscopy, Langmuir probe, electrical double probe, retarding field analyser and Faraday cup measurements – have been conducted to understand the influence of the plasma parameters on the nitriding behaviour and t he DLC film growth. The hardness of the a-C:H coatings was in the range of 1200 – 3000 HV, decreasing with increasing acetylene flow due to a decrease of the kinetic energy of the C_xH_y molecules impinging on the growing film surface. Raman spectroscopy showed a shifted G-peak at 1530 cm^{-1} and a shifted D-peak. The friction coefficients in dry pin-on-disk tests with an Al_2O_3 counterpart and a load of 10 N were in the range of 0.05 and 0.10. In conclusion, it could be shown that the duplex treatment with a plasma beam source has been optimised by correlating the plasma parameters with the properties of the coated parts.

3:10pm **D2-2-6 Correlation of Plasma Properties and Microstructures of DLC Films Deposited by Laser Induced High Current Pulsed Arc**, *J.-B. Wu (wujinbao@itri.org.tw),* ***C.-Y. Chen***, Industrial Technology Research Institute, Taiwan*, M.-Y. Li,* National Nano Device Laboratories, Taiwan*, M.-S. Leu, A.-K. Li*, Industrial Technology Research Institute, Taiwan

Hydrogen free diamond-like carbon (DLC) coatings were deposited on Si(100) and stainless steel substrates by laser induced high current pulsed arc (LIHCPA) system with different deposition partial pressure. The microstructures and hardness behavior of DLC films were identified by the instrumental analyses such as scanning electron microscopy, X-ray photoelectron spectroscopy and nano-indentation. In order to investigate the correlations between the microstructures of films and plasma characteristics, a quadrupole plasma analyzer was used to identify the positive ion energy distribution (IED). Meanwhile, the pulse arc current of 4 kA was applied to the carbon target to deposit the DLC films with high density and hardness. It was found that the deposition partial pressures affected not only the hardness and sp^3 content of the films but also the carbon ion energy. The results clearly showed that when the deposition partial pressure increased from 3×10^{-3} Pa to 1 Pa, the hardness decreased from 52 GPa to 27 GPa, and also the sp^3 fraction of the DLC films measured by XPS would increase from 47 % to 61 % accordingly. Moreover, by taking deposition partial pressure of 3×10^{-3} Pa into consideration, the results revealed that the plasma species generated by the carbon target were verified as C^+, C^{2+}, C_2^+ and C_3^+ among which that the C^+ ion energy can achieve the energy of 50 eV.

3:30pm **D2-2-7 Amorphous Carbon Deposited By Sputtering And Plasma Enhanced Chemical Vapor Deposition Without Vacuum Pumping During Deposition**, *G.A. Viana,* ***F.C. Marques*** *(marques@ifi.unicamp.br)*, University of Campinas, Brazil

Amorphous carbon film films have been deposited by a large variety of deposition method. All of them use a flow of certain gas for the deposition of the films. Plasma enhanced chemical vapor deposition (PECVD), for instance, usually uses a flow of methane into the chamber and pumped with a vacuum pump during deposition. This gas is then disposable to the atmosphere. For films deposited by sputtering, usually an argon gas flow is adopted for sputtering a graphite target. In this case a large amount of argon is also released to the atmosphere. In this work we propose the deposition of amorphous carbon by sputtering and PECVD without vacuum pumping during deposition. For that purpose the deposition chamber is prior pumped down to about 10-8 Torr in order to clean the chamber walls, avoiding the releasing of adsorved contaminating gases during deposition. The chamber is then closed and argon (for sputtering) or methane (for PECVD) is introduced into the chamber until a cert ain pressure. The films are then deposited without further supply of argon or methane. Graphitic-like amorphous carbon was obtained by sputtering and diamond- and polymeric-like carbon films were obtained by PECVD. Visible and infrared transmission spectroscopy, nanohardness, stress and Raman measurements reveal that the properties of the films are similar to those reported for films deposited by the conventional procedure. Ones conclude that this technique can be used to reduced gas waste and thus contributing to a sound environment.

3:50pm **D2-2-8 Thermal Annealing Effect on Tribological, Electrical, and Corrosion Properties of DLC Coatings**, *L. Wang, Z.J. Peng,* University of Windsor, Canada*, J. Housden, E. Spain,* Tecvac Ltd.*,* ***X. Nie*** *(xnie@uwindsor.ca)*, University of Windsor, Canada

Diamond-like Carbon (DLC) coatings usually have high hardness, chemical inertness and high electrical resistivity. These properties make them attractive in a wide range of applications such as engineering tools, dies, electronic devices and even medical and food packaging field. Especially, due to their remarkable tribological properties (low friction coefficient and wear rate), DLC coatings have become one of the important coatings applied as protective layer on engineering tools. However, DLC coatings are likely graphitized at high working temperatures. Many research efforts have been made to investigate the effect of annealing temperature on coating structure, mechanical and tribological properties. In this study, besides the mechanical and tribological properties, electrical conductivity and corrosion protective properties of annealed DLC coatings were investigated. The annealing treatments were performed in air for 1 hour at the temperatures ranged from 200 to 600 °C. Nanomechanical test instrument, pin-on-disc tribometer, potentiodynamic electrochemical tests and interfacial contact resistance tests were used to study the mechanical, tribological, corrosion protective and electrical conductivity properties of DLC coatings before and after annealing. The investigation results were discussed in terms of feasibility of DLC coatings for various applications.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E2-2

Mechanical Properties and Adhesion
Moderator: R. Chromik, McGill University, L. Davies, Caterpillar, J. Michler, Empa

1:30pm **E2-2-1 Hardness and Elastic Modulus of DLC Thin Films by Depth-Sensing Indentation Modeling**, *F. Roudet,* ***D. Chicot*** *(didier.chicot@univ-lille1.fr),* University of Science and Technology of Lille, France*, L. Gil,* UNEXPO, Venezuela*, M.H. Staia, E.S. Puchi-Cabrera*, Universidad Central de Venezuela

In biomedical applications, Diamond-Like Carbon (DLC) films are widely used due to their good tribological and tribo-corrosion behavior, as well as their remarkable biomedical properties. To improve their applicability, it is necessary characterize their mechanical properties in the best possible way, especially hardness and elastic modulus, which are required in wear analysis. One of the most adequate techniques to determine the mechanical properties of thin films, is depth-sensing indentation (DSI) since these can be analyzed both in nano and micro-indentation depth regimes. However, it is widely recognized that the hardness measurement of a film is affected by the presence of the substrate when the indentation depth is higher than approximately 10% of the film thickness. On the contrary, for the determination of the elastic modulus of a film the substrate interferes with the measurement as soon as the indentation depth exceeds about 1% of the film thickness. Thus, in both cases, analytical models are often required to separate the contributions of the substrate and film from the global indentation measurement, even in nano-indentation measurements. In this work, the application of several models (Jönsson and Hogmark (1984), Chicot and Lesage (1995), Korsunsky et al. (1998) and Puchi-Cabrera (2002)) for the determination of the film hardness using both Martens hardness and contact hardness deduced from the Oliver and Pharr analysis is proposed. For the determination of the elastic modulus, a recent model developed by one of the authors is applied, which takes into account the frame compliance of the instrument. Such a model considers two asymptotic trends, i.e. the film trend for low applied loads and the substrate trend for highest ones, described by means of a simple parametric relationship. Finally, the loading curve of DSI test is interpreted in terms of "instantaneous Martens hardness" since no dwell-time is performed at each increment of load.

1:50pm **E2-2-2 Parametric Study on the Behavior of a Film/Substrate System to Limit Crack Propagation by the Finite Element Method**, ***N.K. Fukumasu*** *(newton.fukumasu@usp.br), R.M. Souza,* University of São Paulo, Brazil*, M. Ignat*, SIMAP Grenoble INP, France

Coated systems under high tensile loads tend to present cracks that nucleate at the film surface and propagate perpendicular to the film/substrate interface. From experimental tests, it is possible to observe that some

substrate materials have the capacity to prevent the propagation of film cracks into the substrate and along the film/substrate interface, due to high plastic deformation at the crack tip. In this work, this behavior was investigated by a parametric finite element analysis on some of the mechanical properties of the substrate and the thin film (Young's modulus, yield stress and fracture toughness). Systems were composed of an elastic thin film onto a perfectly plastic substrate and the numerical analyses considered that both were perfectly bonded. Tensile loads were applied perpendicular to the crack path and crack propagation was included in the analysis. Results allowed correlating some of the parameters that were studied with the ability of the substrate to limit the tendency for crack propagation along the film/substrate interface.

2:10pm **E2-2-3 Nano-Impact – a Reliable Tool to Predict Coating Performance**, ***B. Beake*** *(ben@micromaterials.co.uk)*, Micro Materials Ltd, United Kingdom

An advanced nanomechanical test technique, nano-impact testing, can simulate the interrupted contact (and cyclic loading) conditions occurring in severe contact applications and evaluate the fatigue fracture resistance of coated components. Essentially a fast repetitive nanoindentation technique, nano-impact was developed to test coating properties at high strain rates and to investigate surface fatigue and fracture due to repetitive contact with the aim of using results to optimise the coating properties for improved durability. A commercial nanoindentation system [NanoTest system, Micro Materials Ltd] is utilised to perform high cycle and low cycle fatigue testing by nano-impact. In contrast to conventional impact testing at the macro-scale, or instrumented micro-impact, nano-impact testing offers some distinct potential advantages for testing thin coatings, in throughput, automation, surface sensitivity. The traditional wear test method of periodically stopping the test to remove the sample and ex-situ assessment of any degradation limits mechanistic study. There is a growing realisation that the next generation of wear test instruments should be developed to enable the wear process to be studied in situ reflecting a shift in focus towards designing coatings for wear protection based on mechanistic understanding. Counter-intuitively, the nano-scale impact test can be more severe than instrumented micro-impact testing. The contact strains are higher with the smaller and sharper indenters and higher contact pressures are possible, which can result in more accelerated wear behaviour. Shorter tests become feasible, with the possibility of performing repetitive tests across samples and obtaining a more complete statistical picture of the impact response. Clear correlations are observed with actual performance testing. In particular the rapid, automated, laboratory nano-impact tests correlate well with the longer, more laborious and expensive tests of (1) cutting tool life, ranking coating performance in terms of tool life in end milling and simulating the evolution of the tool wear (2) coating wear in high performance engine applications. As its usage grows, the laboratory nano-impact test will become a successful way to speed up the pace of new coating development for interrupted contact applications. The principles and applications of the nano-impact technique are reviewed in this presentation.

2:30pm **E2-2-4 Adhesion of Diamond Like Carbon (DLC) Coatings on Metalic Biomedical Implants**, ***C.V. Falub*** *(Claudiu.Falub@empa.ch), R. Hauert, G. Thorwath, U. Müller, M. Parlinska-Wojtan, C. Affolter, P. Schmutz, J. Michler,* Empa, Switzerland*, M. Tobler,* IonBond AG*, C. Voisard*, Synthes GmbH

Owing to their excellent mechanical properties, e.g. high hardness, low friction coefficient, low wear rate, diamond like carbon (DLC) coatings are excellent candidates for surface engineering, such as protective films for metalic biomedical implants. However, interface chemistry and relatively high intrinsic stresses (~ GPa) can determine unpredictable in-vivo delamination of these layers, which may lead to the total failure of the implant. The speed of delamination, which in fact influences the lifetime of the coated implant, can range from ~ mm/day to below µm/year. While fast delamination of the order of hundreds of µm/day can easily be detected, very slow delaminations of the order of a few µm/year are very difficult to observe. Nevertheless, several delaminating microscopic spots growing with a speed of a few tens of µm/year may cause the failure of the coated implant after several years. Therefore, in order to estimate the lifetime of a coated implant in the human body it is extremely important to have a reliable quantitative method for determining the speed of coating delamination. Although qualitative studies of the mechanisms involved in DLC coated implant failure exist, to our knowledge no quantitative analysis of coating adhesion lifetime on biomedical implants has ever been done and that is the aim of this work. The discussion will tackle both fundamental physical and chemical aspects at the DLC/implant interface, but also fracture mechanics analysis using finite element modelling (FEM). In-vivo stability of different interlayers will be discussed in conjunction with electrochemistry experiments as well as mechanical failure tests.

2:50pm **E2-2-5 A Micro-FEM Modelling Based Fracture Mechanics Approach to Wear Resistance Assessment of Thin Hard Coatings**, ***K. Holmberg*** *(kenneth.holmberg@vtt.fi)*, VTT Technical Research Centre of Finland **INVITED**

Thin hard coating has been implemented successfully in several industrial applications. However, there even larger use is still limited by surface fracture, wear and the difficulty of lifetime prediction and control. A new approach is presented that is based on finite element modelling of the coated surface on micro level, simulation of critical stresses and strains and calculation of fracture toughness of the surface. TiN, MoS_2 and DLC coatings on steel substrate with and without bond layers have been investigated in a sliding ball against plane contact geometry both empirically and by modelling. The influence of residual stresses on the surface cracking is shown and the crack growth both vertically through the coating as well as horizontally as interface cracking between the coating and the substrate resulting in coating delamination is analysed. A parametric analysis of the influencing parameters has been carried out and gives guidelines for improved control of the cracking and wear process.

3:30pm **E2-2-7 Yield Maps for a Bilayer Film System (Coating/Seed Layer) on a Substrate Using the Finite Element Method**, *A. Chatterjee, A.A. Polycarpou (polycarp@illinois.edu),* ***P. Bellon***, University of Illinois at Urbana-Champaign

The finite element method was used to study plastic yielding in a bilayer (e.g., coating and seed layer) on a substrate system when subjected to ramped load scratch. Prevention of delamination is inherently related to prevention of yield onset at the seed layer/ substrate interface (I2). The objective is to systematically analyze the effect of film thickness ratio (t1/t2), film hardness ratio (H1/H2) and friction coefficient µ on the onset of yield at I2. Yield zone maps were constructed where the yield positions were depicted and contact pressures (PCont) required for onset of yield at those specific locations were found. Onset of yield at I2 occurs when $0.5< t1/t2<5$. For $t1/t2>5$, yield onset occurs in the coating at $\mu = 0.1$ and on the surface at $\mu = 0.5$. For $t1/t2<0.5$, yield onset occurs at the interface between coating/seed layer (I1) for $\mu = 0.1$ and at the surface as well as I1 for $\mu = 0.5$. PCont required for yield at I2 is independent of H1/H2 and is minimum c ompared to PCont at other locations. PCont increases with H1/H2 when yield onset occurs at the surface, in the film or at I1. Competition between increase in shear stress at the surface/film with the stress discontinuities at interfaces I1 and I2 is responsible for yield onset at different zones. When $t1/t2 = 20$ and $H1/H2 = 4$, PCont required for yield onset is the highest for both $\mu = 0.1$ and 0.5. The possibility of delamination is therefore, minimum when the film parameters are close to the above specified ratios. Thus, for both low and high friction applications, the yield zone map is a useful tool for designing a bilayer film/substrate system in order to prevent delamination.

3:50pm **E2-2-8 Mechanical and Tribological Properties of Thermally Oxidized Ti6Al4V-TiC Composite**, ***N. Dalili***, *A. Edrisy (edrisy@uwindsor.ca),* University of Windsor, Canada*, J. Lo*, CANMET-MTL; Natural Resources, Canada

In this study TiAl6V4V-10% TiC composites (TMC), fabricated by metal injection moulding (MIM) process as an ex-situ fabrication method, were thermally oxidized (TO) in order to enhance their sliding behaviour under high contact pressure. The composite samples were thermally oxidized at temperature range of 600-800°C for 20 min, 20 h and 65 h. Optimum oxidation condition was determined at 800°C for the duration of 20 min. The TO-treated surfaces at the optimum oxidation condition were characterized by a hard TiO_2 oxide compound (rutile) with the thickness of 3±0.5 µm and a 30±1 µm hardened diffused layer (α-Ti). TEM results showed that the oxide scale consisted of ultrafine grains in the range of 0.1-0.5 µm. The compound layer comprised a thin Al-rich layer as a result of outward diffusion of Al during oxidation. Using a ball on disc tribometer, it was shown that the TO-treated TMC samples had superior sliding wear resistance when compared to that of the untreated ones. The high wear rates of TMC at high loads attributed to the three-body abrasion of TiC fragments and material transfer of Ti matrix to the counterface. Single pass scratch tests under progressive Hertzian pressure of 5.3-144 GPa were performed on TMC and TO-treated TMC surfaces. The wear and scratch paths were investigated using analytical microscopy techniques including focused ion beam (FIB), scanning electron microscopy (SEM) and optical surface profilometery. The results from the scratch tests showed fracture and fragmentation of TiC particles along with severely deformed TiAl6V4V matrix under high contact pressures for TMC samples. However, the main surface damage for TO-treated samples was formation of half-circle type cracks in the oxide film.

4:10pm **E2-2-9 Effect of Aging on Adhesion of Black Anodic Coatings for Space Applications**, ***Y. Goueffon*** *(yann.goueffon@isae.fr),* CNES, France*, C. Mabru,* Universite de Toulouse, ISAE, France*, L. Arurault,* Universite de Toulouse, CIRIMAT-Institut Carnot, France*, C. Tonon,* Astrium, France*, P. Guigue*, CNES, France

Due to the space vacuum, thermal regulation of spacecrafts is passively managed by radiative exchanges between its external surfaces and the environment. Black inorganic anodized aluminium alloys are often used for their thermo-optical properties ($\alpha s \geq 0.93$; $\varepsilon n \geq 0.9$). However, many cases of flaking have been observed after thermal cycles carried out to simulate the space environment. In orbit, such particles could contaminate instruments of the satellite. The aim of this study is to evaluate the influence of the ageing on such films adhesion.

The process used is a sulphuric anodizing, followed by an inorganic colouring and a sealing. It has been shown that the porosity and the thickness of the anodic film have a major influence on the mechanical behaviour during adhesion measurement performed by scratch tests and 4-point bending tests.

Black anodized samples with different porosities have been submitted to thermal cycles. The influence of the pressure and temperature on the adhesion has been observed. Two main mechanisms have been highlighted: the difference of thermal expansion coefficient between film and substrate and the dehydration of the film. Finite element modelling illustrated the mechanical state at the interface between the substrate and its cracked or non cracked anodic film during thermal loads (process and cycling). Particularly, the presence of cracks can either be detrimental or beneficial on the interface loading depending on the mechanical properties of the film which change with the porosity.

4:30pm **E2-2-10 Effect of Hydrogen on the Mechanical and Structural Properties of SiC-Derived Carbon Films**, *D.-S. Lim (dslim@korea.ac.kr),* ***H.-J. Choi***, Korea University, Korea*, M.J. McNallan,* University of Illinois at Chicago*, Y.H. Sohn*, University of Central Florida

SiC-derived carbon films have been produced by etching silicon carbide in mixed gas environment containing chlorine and hydrogen[1-2]. Hydrogen plays a crucial role in the SiC derived carbon films. But the detailed role of hydrogen on the carbon formation and its structure and properties is still not well understood. In this study, the effect of hydrogen content on the structure and mechanical properties of carbon films modified from sintered SiC by mixtures of chlorine–hydrogen gas at 1000 °C for 20 hours was investigated. Based on the structural analysis of carbon films produced, conversion from silicon carbide proceeded faster with increasing chlorine content at a high temperature. The rate of carbon formation determined by SEM observation was decreased with increasing hydrogen content. The hardness and elastic modulus of the carbon films tended to decrease with increasing hydrogen content. By varying the concentration of hydrogen, the ratio between the crystalline graphite and amorphous carbon was changed. The different bonding types and crystallinity of the carbon films directly correlated with their mechanical properties. The possible mechanism for the lowering mechanical properties has been discussed based on the analysis of HREM and Raman spectroscopy observation.

[1]A. Nikitin and Y. Gogotsi, "Nanostructureed Carbide-Derived Carbon," Encyclopidia of nanoscience and nanotechnology, Vol. 7 p 553-573 (2004),

[2] Hyun-Ju Choi, Jeon-Kook Lee, and Dae-Soon Lim, "Tribology of Carbon Layers Fabricated from SiC under the Different H2/Cl2 Gas Mixtures," Accepted for Publication in Journal of Ceramic Processing Research (2008).

4:50pm **E2-2-11 Tensile Testing of Amorphous Diamond-Like Carbon and Crystalline Diamond Coatings**, *J. Schaufler,* ***K. Durst*** *(karsten.durst@ww.uni-erlangen.de), K. Kellermann, S. Rosiwall, M. Göken*, University Erlangen Nürnberg, Germany

The research efforts on carbon coatings have strongly increased in the last decade. Many investigations, especially in the field of improvements in the deposition processes and the tribological behavior of the coatings under high loading conditions have been done. But up till now, a clear and broad understanding of the basic deformation and failure mechanism under elementary loading conditions has not been achieved. Of great importance is the interaction between the coating and the substrates with regard to the occurring failure mechanism. In this work the failure and delamination behavior of diamond-like carbon coatings and crystalline diamond coatings, both deposited to a thickness of 2 µm on steel substrates, were investigated under tensile loading. In both systems a chromium-chromiumcarbide transition layer was used to enhance the adhesion between the coating and the steel substrate. The two coating systems were deposited with two different deposition processes (DL C system: combined PVD-PECVD, diamond system: Hot filament CVD), therefore the width of the adhesion layers varies between 500 nm (DLC system) and 20 µm (diamond system). The coated specimens were deformed under tensile loading conditions with a micro tensile testing equipment. In-situ tensile tests in a SEM allow the analysis of the damage evolution in the coated systems. By comparison with the stress strain behavior of the uncoated steel substrates, the mechanical stresses within the coatings are derived allowing a better understanding of the failure behavior of the coated systems The investigated coating systems show a complete different failure behavior under tensile loading. In the DLC coating cracks occur after strain values of around 1.7%. These brittle cracks grow perpendicular through the adhesion layer to the surface of the steel substrate. Even after strains of more than 20%, the DLC coating still adheres to the substrate. Furthermore, after high deformation of th e specimens a formation of shear bands in the adhesion layer were observed with a TEM. In contrast, the crystalline diamond coating showed a spallation after strain values of 1% and large regions of the coating delaminated at the interface chromiumcarbide-diamond.

5:10pm **E2-2-12 The Mechanical Strength of Micro-to-Nanoscale Porous Ag Coatings**, *A. Jankowski (alan.jankowski@ttu.edu),* ***H. Ahmed***, Texas Tech University

The use of porous metal coatings is ever increasing in renewable-energy system applications as solar cells and hydrogen fuel cells. In particular, the scale of porosity in metal coatings is particularly important to their catalytic performance. Potentially just as important is the mechanical stability of the porous coating in these devices. A series of rate-dependent deformation tests are now conducted to better understand operative deformation mechanisms in the evaluation of strength as the porous support structure changes across multiple length scales, i.e. from the micro-to-nano. Tensile testing is used to evaluate commercially available, free-standing silver membranes with constituent micron-to-submicron porosity. Scratch testing of porous silver-coated substrates permits evaluation of nanoscale porous structures. Preliminary findings indicate that the strain-rate sensitivity of tensile tested specimens is found to increase as length scale decreases. The trends are similar to those experimental results reported for bulk nanocrystalline metals. Underlying structural features that can contribute to this mechanical behavior include pore size, filament or strut size, and the grain size within. These features of length scale are evaluated, and then assessed for the scratch hardness behavior of nano-porous silver coatings.

This work is supported through the J.W. Wright Endowment in Mechanical Engineering at Texas Tech University.

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G5/H5

Coatings for Renewable Energy Systems
Moderator: L. Bardos, Uppsala University, S. Roy, Newcastle University

1:30pm **G5/H5-1 Electrochemical Energy Conversion: Coatings for Fuel Cells, Electrolysers and Batteries**, ***E. Roberts*** *(Edward.roberts@manchester.ac.uk)*, Unversity of Manchester, United Kingdom **INVITED**

Electrochemical energy conversion devices include state of the art energy storage systems such as lithium-ion batteries, electrolysers such as those used in the chlor-alkali industry, and fuel cells which could offer a clean alternative to the internal combustion engine for transport. The environment in electrochemical energy conversion devices is typically highly corrosive. Strongly oxidising species are normally present in the electrolytes and high positive potentials can occur at the electrodes. Corrosion of materials in electrochemical devices often leads to a rapid deterioration in performance, severely limiting the lifetime. At the same time, electrode materials are required to provide high conductivity, low contact resistance and in some cases electrocatalytic activity. In addition, material costs can make a significant difference to the commercial viability of these devices. This is particularly true for fuel cell systems, where material costs will need to be significantly reduced compared to the current state of the art if this technology is to make a significant impact. Coated materials may provide a practical solution, where a low cost substrate is coated with thin layer of a material which offers the required surface properties.

In this paper, the nature of electrochemical energy conversion devices will be reviewed, and the desired properties of materials, particularly the plate materials, will be identified. Methods for characterising material performance and performance targets will be considered in this context. The types of coatings that have been studied will be discussed and there performance will be evaluated. Coatings used in this context include conducting polymers, amorphous and diamond-like carbon, as well as ultra-thin noble metal coatings. Finally, the future directions and opportunities in this field will be considered.

2:10pm **G5/H5-3 Thin-Film Based Superconducting Wires for Electric Power Applications**, ***V. Selvamanickam*** *(selva@uh.edu)*, University of Houston **INVITED**

High Temperature Superconductors (HTS) have been developed over the last 20 years for electric power applications. The challenge was in fabricating oxide ceramic material in form of flexible wires in continuous lengths of more than a kilometer with a high current carrying capability. This goal was recently met by SuperPower Inc and a prototype power transmission cable made with nearly 10,000 meters of HTS wire was installed and energized in the electric power grid. The enabling technology for this achievement is ion beam assisted deposition (IBAD) which provides the ability to grow nearly single-crystalline films on thin tapes of polycrystalline substrates such as nickel superalloys. A high degree of biaxial texture is developed in IBAD MgO within a thickness of 10 nm. This texture is epitaxially transferred to a superconducting film via one or two intermediate layers. Metal Organic Chemical Vapor Deposition (MOCVD) is used to epitaxially grow high quality $REBa_2Cu_3O_7$ (RE = rare earth) superconducting films with a current density over 6 MA/cm^2. Multiple buffer layers based on bixbyite, rock salt, and perovskite crystal structures as well as amorphous layers all with a total thickness less than 200 nm are deposited between the flexible metal substrate and the superconducting layer for the purpose of diffusion barrier, nucleation promoter, and lattice match. In addition to near-single crystalline growth of the superconductor, high performance is also achieved through nano-scale defect structure for pinning of fluxons. The ability to grow near-single crystalline films on inexpensive polycrystalline substrates has been the key to success with HTS and now this technique is being explored for other energy applications too.

2:50pm **G5/H5-5 Deposition and Characterisation of NiO_x Films by Reactive Sputtering for Application in Dye Sensitized Solar Cells**, ***M. Awais***, *M. Rahman, D. MacElroy,* University College Dublin, Ireland, *N. Coburn, D. Dini, J.G. Vos,* Dublin City University, Ireland, *D.P. Dowling (denis.dowling@ucd.ie)*, University College Dublin, Ireland

NiO_x due to its high band gap (3.8 eV) and p-type nature has considerable potential for use as an electrode in a tandem dye-sensitized solar cells (DSSc)[1]. However to-date the efficiency of sol gel deposited NiO_x semiconductors in DSSc has been found to be relatively poor[2], which limits the effectiveness of the tandem cells. One of the possible ways to improve this efficiency is to deposit the coating by the reactive sputtering method. This is because the uniformity, reproducibility, and mechanical durability of the sputtered deposited film is much higher compared to sol-gel or other wet chemical deposition techniques. In this study, NiO_x films were deposited using the unbalanced magnetron sputtering technique. The coating was deposited onto both silicon wafer and indium tin oxide glass (ITO) substrates. The influence of deposition parameters such as pressure, nickel target current and substrate bias voltage were correlated with coating properties, including surface roughness, thickness, crystallographic structure and surface energy. These evaluations were carried out using optical profilometry, spectroscopic ellipsometry, XRD and water contact angle measurements. The NiO_x coatings were sensitized with Ru-complex dye containing appropriate anchoring moieties (carboxylic group). The dye adsorption was investigated in transmission mode on the ITO using UV-Vis spectroscopy in the range 200 – 600 nm. Dye adsorption was enhanced on nickel oxide surfaces exhibiting higher surface energy values. For example for 70 nm thick NiO_x coatings, it was found that increasing surface roughness (Ra) from 4.0 to 4.4 nm, with corresponding increase in surface energy from 37.1 to 57.6 mN/mm, there was a 12 % increase in the level of light absorption.

[1]E. L. Miller, R. E. Rocheleau, Journal of Electrochemical Society 144 (1997) 1995.

[2] J. He, H. Lindström, A. Hagfeldt, S. Lindquist, Journal of Physical Chemistry B 103 (1999) 8940.

3:10pm **G5/H5-6 Renewable Energy Systems - What Coatings Do We Need?**, ***H. Bernhoff*** *(Hans.Bernhoff@angstrom.uu.se), M. Bergkvist,*, Uppsala University, Sweden **INVITED**

Rapid developments in different renewable energy systems bring about an increased need for functional and specific-purpose coatings. Three main categories of renewable energy systems can be recognized : (1) water power systems including wave power, marine current, tidal systems, and conventional hydro power installations (2) wind power systems, and (3) solar and solar-thermal systems. Furthermore, several additional thermal systems utilizing geothermal energy, ocean thermal energy, recycling of heat or cooling energy have additional demands on coatings. Important driving factors for new coatings and coating applications in these systems are their power efficiencies, lifetimes and environmental considerations concerning greases, lubricants and solid lubricants, to improve properties like friction, wear, noise, etc. The terminological requirements apply in systems with movable parts. For example there are often elastomer parts moving against a metal counter body requiring surface modification of materials and wear resistant coatings at long sliding distances. Polymeric and composite coatings could be used for this purpose. A special category of corrosion resistant and wear resistant coatings is required in systems for offshore systems such as wind and wave-power installation. Submerged component will also need environmental friendly antifouling systems. Furthermore hydrophobic coatings on wind-turbine blades can be applied to reduce forming of ice and aggregation of contaminants which will affect aerodynamic performance. In this paper several renewable energy systems will be described and the most important parts requiring coating applications with specific parameters will be discussed.

Bioactive Coatings and Biofunctionalization
Room: Sunset - Session TS3-1

Bioactive Coatings and Surface Biofunctionalization

Moderator: D.V. Shtansky, State Technological University "Moscow Institute of Steel and Alloys", S. Kumar, University of South Australia

1:30pm **TS3-1-1 Nitrogen-Rich Plasma Polymer Coatings ("PPE:N") for Bio-Medical Applications in Orthopaedic and Vascular Therapies**, ***M.R. Wertheimer*** *(michel.wertheimer@polymtl.ca), P.L. Girard-Lauriault, F. Truica-Marassescu, J.-C. Ruiz,* École Polytechnique, Canada, *H.T. Wang, J. Antoniou, F. Mwale,* McGill University, Canada, *S. Lerouge*, Notre-Dame Hospital, Canada **INVITED**

We have recently reported that nitrogen (N)-rich (amine-bearing) polymer surfaces can promote cell adhesion and influence processes such as differentiation of stem cells. A well-known route for obtain-ing high nitrogen concentrations, [N], is plasma-polymerisation (PP) of suitable N-containing "monomers" (e.g. allyl-amine). In this study, however, we used binary gas mixtures of varying ratios, X, comprising ethylene and nitrogen or ammonia, and we compared the physico-chemical properties of our "PPE:N" films, which were deposited either in conventional low-pressure ("L-PPE:N") r.f. glow discharges, or in a dielectric barrier discharge (DBD) reactor operating at atmospheric ("H-PPE:N") pressure. Both types of deposits were characterised by several analytical techniques, such as X-ray Photoelectron Spectroscopy (XPS), Infrared Spectroscopy (IR), and Near-edge X-ray Absorption Fine Structure (NEXAFS), to determine their surface-chemical compositions and molecular structure; and Contact Angle Goniometry (CAG) for evaluating their surface energies. Various N-containing species were identified by combining complementary analyses; for example, the concentration of primary amines was determined by the selective reaction of 4-(trifluoromethyl)benzaldehyde (TFBA) vapour with the surface-near (-NH2) groups, while XPS, IR and NEXAFS allowed semi-quantitative analyses of unsaturated species such as nitriles, iso-nitriles and imines to be carried out. XPS data revealed [N] values of up to 40 at.-%, primarily comprising nitrile and amine functionalities, where [N] could be controlled systematically and reproducibly by varying X. PPE:N-coated substrates have already proven their value in several bio-technological applications: for example, usually non-adherent human U-937 monocytes could readily adhere to them, as did chondrocytes, human mesenchymal stem cells (MSC), vascular smooth muscle cells (VMSC), and other cell lines of interest in orthopaedics and in vascular medicine. Furthermore, we demonstrated the existence of [N]crit values, below which different cell lines ceased to adhere, that MSC differentiation could be regulated, and that resistance to apoptosis of VMSC could be increased. In several of these situations, for example in studying U-937 and MSC, we have carried out detailed experiments using reverse transcriptase-polymerase chain reaction (RT-PCR) to investigate gene expression and possible mechanisms underlying observed behaviours of the adhering cells.

2:10pm **TS3-1-3 Surface Nano-Texturing to Combating In-Stent Restenosis and Late-Stent Thrombosis**, ***L. Clark***, Southern Illinois University Carbondale, *Y.T. Shiu,* University of Utah, *M. Duggins, P. Kohli, S. Aouadi (saouadi@physics.siu.edu)*, Southern Illinois University Carbondale

A completely new non-polymeric based prototype of Titanium alloy and Tantalum-based heart stents were developed. These new stents are created by nano-texturing or micro-texturing treatments of their surfaces. The surface treatments were also modified utilizing silane chemistry. The treated surfaces were subsequently investigated as a suitable carrier for selected anticoagulant drugs to combat neointima hyperplasia. More specifically, the drugs were immobilized, and both drug-saturation and drug-elution processes were investigated as a function of the surface architecture and biofunctionalization. The biocompatibility of these

materials were assessed by investigating: (i) their ability to undergo endothelialization and cell proliferation; (ii) the adsorption of plasma albumin and fibrinogen on their surfaces; and, (iii) platelet adhesion on their surfaces.

2:30pm **TS3-1-4 Characterisation of Ca- and P-Containing PEO Coatings on Ti Substrates**, ***A. Yerokhin*** *(A.Yerokhin@sheffield.ac.uk),* University of Sheffield, United Kingdom, *A. Pilkington,* University of Sheffeild, United Kingdom, *A. Matthews*, University of Sheffield, United Kingdom

Plasma electrolytic oxidation (PEO) of Ti is currently attracting major attention for surface engineering of intrabone implants. Enhancements in bioactive properties of PEO titania can be achieved by tailoring surface morphology, controlling anatase to rutile ratio and incorporating appropriate elements (e.g. P and Ca) into the coating material. The account for these routes was taken during development of an advanced PEO process based on application of pulsed reversed current modes. Produced coatings were characterised in terms of thickness, roughness, elemental and phase compositions. Nanoindentation measurements and scratch adhesion tests were performed to evaluate mechanical properties of the surface layers. A series of in vitro assays was carried out to evaluate cell viability, collagen activity and attachment to the substrate. It is shown that uniform and strongly adhered to Ti substrate oxide ceramic coatings with 5 to 30 micron thickness can be obtained using the PEO method developed. The coatings possess high ratio of Ca/P = (1.5..3)/1 which results in formation of bioactive components (e.g. hydroxyapatite and calcium phosphates), in both amorphous and crystalline from, within the titania matrix. The coating stiffness lies between that of Ti substrate and bone material, thus providing graded transition in mechanical properties across the bone implant interface. New coatings possess high biocompatibility and enhanced bioactive properties compared to both untreated Ti and conventional PEO coatings. The versatility of the method is demonstrated by examples of coating deposition on different Ti alloys and components of prosthetic devices of complex shape.

2:50pm **TS3-1-5 Effects of Sterilization and Heat Treatment on Alumina Coated Surgical Instruments**, ***A.C. Ruiz***, *M. Kavdia,* University of Arkansas, *S. Kumar,* University of South Australia, *M.H. Gordon (mhg@uark.edu),* University of Arkansas, *H.H. Abu-Safe,* Lebanese American University, Lebanon, *S. Davis*, Philander smith College

Alumina thin films were deposited on surgical stainless steel scalpel blades to test the sharpness of the coated blades, to investigate the effects of sterilization on both the phase of the film and the adsorption of protein, and to study the thermal stability of the film. The films were deposited using an inverted cylindrical dual magnetron sputtering system. Process parameters such as power, pressure, oxygen partial pressure, and substrate bias were varied to grow the films. Alumina coated samples were sterilized in a steam autoclave for 1-10 cycles, each of 40 minutes. The film phase before and after sterilization was determined by X-ray diffraction. The adhesion of BSA protein was quantified by Fourier transform infrared spectroscopy. Preliminary results indicate that the coated blades adsorbed 50% less protein when compared to the uncoated blades. Coated samples were annealed to temperatures up to 1200°C to study thermal and structural stability. Sharpness of the coated surgical scalpel blades was investigated by pushing the blade into an elastomeric material and measuring the cutting force. Preliminary results indicate that the blades coated with alumina remain sharper for a longer period of time.

3:10pm **TS3-1-6 H and N Low Energy Ion Implantations in Ti: Mechanical and Tribological Properties**, *G.B. de Souza,* ***C.E. Foerster*** *(carlosef@uepg.br),* Universidade Estadual de Maringá, Brazil, *C. Lepienski, N.K. Kuromoto,* Universidade Federal do Paraná, Brazil, *S.L.R. da Silva, A.L. Chinelatto*, Universidade Estadual de Ponta Grossa, Brazil

Titanium is usually employed as biomaterial but its biocompatibility must be improved for certain applications. Recent and promising researches reported the use of H ion implantation by PIII in order to enhance Ti osseointegration capability and blood compatibility. The aim of the present study is understand the influence of H at near surface region on the mechanical and tribological properties of Ti and Ti previously plasma nitrided. The N and H ion implantations were performed at low ion energy in glow discharge regime. The nitrogen and hydrogen content profiles were obtained by NRA and the structural changes by X-ray diffraction. Hydrogenation process increases the near surface hydrogen content from 11 at% up to 18 at%. The H presence is in solid solution and/or in δ-TiH precipitation. It was observed that the previous nitriding process make difficult the implanted hydrogen to form hydrides or even to be dissolved into Ti. Instrumented indentation at submicron scale was employed in order to study the near surface region mechanical properties. Similar elastic modulus and hardness profiles were obtained for hydrogenated Ti in comparing to untreated Ti. However, nanoscratch tests indicated an improvement in scratch resistance after the H implantation. As well known, Ti nitriding improves the mechanical and the tribological surface properties. The subsequent hydrogenation on nitrided Ti raises hardness and elastic modulus which can be attributed to the hydrogen intake and outgassing at the near surface region. Intensive surface embrittlement was also verified in the indentation imprints and in the scratch tracks.

3:30pm **TS3-1-7 Spray-Pyrolyzed Hydroxyapatite Thin-Film Coatings**, ***M. Aguilar-Frutis***, CICATA-IPN, Mexico, *S. Kumar (sunil.kumar@unisa.edu.au),* University of South Australia, *C. Falcony*, CINVESTAV-IPN, Mexico

Plasma-sprayed hydroxyapatite (HA) coatings have been used on metallic bone implants for about 20 years now, but there are still many issues that continue to be addressed for improving the use of HA coatings for improved implant fixation. One such issue is the deposition of HA thin-film coatings with high purity and crystallinity, primarily aimed at realising cost-effective coatings with improved adhesion and predictable properties. A range of deposition methods such as sputtering, electrodeposition and ion-beam deposition have been employed to achieve this goal, with some success. The work presented in this paper is motivated by the need to deposit cost-effective HA thin-film coatings with high purity and crystallinity. For this, we have employed the technique of spray pyrolysis, a simple thin-film deposition method with low setting-up costs and capable of batch processing at atmospheric pressure air ambient. The spray pyrolysis set-up we have used consists of an ultra sonic generator used for mist production from a spraying solution containing the precursor materials. The mist is transported through a glass tube to the substrate surface which is heated to achieve the pyrolytic chemical reaction. Some preliminary results obtained on the deposition and characterization of HA thin-film coatings deposited by this spray pyrolysis system on 2 mm thick Ti6Al4V sheet substrates will be presented and discussed. A 0.042 M calcium spraying solution was prepared by dissolving calcium acetylacetonate hydrate 99.95% in N,N-Dimethylformamide. Phosphoric acid (H3PO4) was used as source of phosphorous. A 0.27% vol. solution of H3PO4 in deionized water was prepared and supplied simultaneously and in parallel during the deposition. The coatings were deposited for 25 minutes at a substrate temperature of about 500ºC. The chemical composition, surface morphology and structure of the HA coatings were determined by scanning electron microscopy (SEM), X-ray phot oelectron spectroscopy (XPS) and X-ray diffraction (XRD). The high purity of the coatings was revealed by the presence of Ca, P and O as the main constituents, with a Ca/P ratio of about 1.3. Some carbon, inherent to the precursors used, was also detected. The XRD patterns obtained suggested preferred (113) and (321) crystal orientations in the coatings.

3:50pm **TS3-1-8 The Influence of Electrochemical Parameters on the Microstructures and Bactericidal Abilities of Anodized Titanium Dioxide Nanotube Arrays**, ***L.-S. Liu***, *Y.-J. Chang, J.-W. Lee (cwlee@mail.tnu.edu.tw), H.-P. Chen*, Tungnan University, Taiwan

The photocatalytic titanium dioxide has been widely applied in the pollution removal and environmental protection field. Recently, the role of titania is getting more and more important due to its water splitting ability to produce hydrogen. In this work, a pulsed DC power supply was adopted to anodize the pure titanium plate to produce TiO2 nanotubes. Amplitudes of the negative and positive voltages and time were adjusted to study their influences on the morphologies and dimensions of TiO2 nanotubes. The anodized titanium plates were further annealed at 450••C for 30 minutes in air. The antibacterial tests of TiO2 nanotubes with different dimensions were performed to evaluate the bactericidal ability against E. coli. It was found that the amphorous titanium dioxide nanotubes were produced under suitable experimental parameters on the surface of pure titanium plate. The crystalline anatise structure of TiO2 nanotubes were formed after annealing at 450••C for 30 minutes in air. The length of nanotubes increased with increasing amplitudes of positive voltage. The antibacterial abilities of the annealed TiO2 nanotubes upon low dose of UV irradiation were enhanced effectively. It is concluded that the anodized TiO2 nanotube array can be used for photo-induced bacteria killing.

4:10pm **TS3-1-9 Biocompatibility of Niobium-Based Coatings**, *G. Ramírez,* ***S.E. Rodil*** *(ser42@iim.unam.mx), H. Arzate,* Universidad Nacional Autónoma de México, *J.J. Olaya,* Universidad Nacional de Colombia, *E. Camps,* Instituto Nacional de Investigaciones Nucleares, México, *S. Muhl*, Universidad Nacional Autónoma de México

Niobium based thin films were deposited on stainless steel substrates to evaluate them as possible biocompatible surfaces that might improve the biocompatibility and extend the life time of stainless steel implants. Niobium nitride and niobium oxide thin films were deposited by reactive unbalanced magnetron sputtering on AISI 316L substrates under standard deposition conditions and without substrate bias or heating. The biocompatibility of the surfaces was evaluated by testing the cellular

adhesion, proliferation and the viability of human cementoblasts cells, during different periods of time up to 7 days. Moreover, preliminary studies of the mineralization process were done up to 14 days. The response of the films was compared to the bare substrate and Ti6Al4V pieces; the most commonly used implant material for orthopaedic and osteosynthesis applications. The physico-chemical properties of the films were evaluated by different means; X-ray diffraction (XRD), Rutherford Backscattering spectroscopy (RBS), energy dispersive X-ray spectroscopy (EDX) and contact angle measurements. The results of XRD and RBS suggested that the niobium oxide films were amorphous but having the Nb_2O_5 stoichiometry, while the niobium nitride films were crystalline showing a one to one composition and fcc phase. The biological evaluation showed that the biocompatibility of the stainless steel can be improved by any of the two films, but in any case it was better than the Ti6Al4V alloy. On the other hand, comparing the two films the NbN seems to be a better surface than the oxide in terms of the adhesion and proliferation of cemetoblasts human cells.

4:30pm **TS3-1-10 Low-temperature Deposition of Titanium Dioxide on Medical Grade Polyetheretherketone to Assist Osseous Integration**, ***H.-K. Tsou***, *P-Y. Hsieh,* Feng Chia University, Taiwan, *C.-J. Chung (cjchung@seed.net.tw),* Central Taiwan University of Science and Technology, Taiwan, *C.-H. Tang,* China Medical University, Taiwan, *T.-W. Shyr, J.-L. He*, Feng Chia University, Taiwan

Polymeric polyetheretherketone (PEEK) has been widely applied in spinal interbody fusion cages for its chemical inertness and bioinertness. PEEK itself is also free from osteo-induction because of its bioinertness and hydrophobicity. Titanium dioxide (TiO_2), known to exhibit good antimicrobial activity, hydrophilicity and bioactivity, is used in this study to apply TiO_2 coatings onto polymeric PEEK substrate at low deposition temperature by using an arc ion plating (AIP) technique. Microstructure, mechanical properties and osteoblast compatibility of the AIP-TiO_2 deposited PEEK are evaluated to investigate the feasibility of using this technique for faster osseous integration on the cages.

Experimental results showed that the deposited TiO_2 possessed a dense columnar structure with anatase TiO_2 as the main phase and rutile phase as the minor phase. Pencil hardness of the TiO_2 deposited specimen reached over 9H, and the high est rank (5B) can be achieved in an adhesion tape test. This firm and compact TiO_2 film corresponded to a critical load of 14.5 N as measured by the scratch test, with the coating experiencing a cohesive failure mode; an indication of strong film adhesion force. High corrosion potential with very low corrosion current was detected both in 3.5 wt.% NaCl electrolyte and simulated body fluid (SBF), indicating the electrochemical inertness of the TiO_2 coating prepared by using AIP technique. In addition, TiO_2 coating presented better osteoblast compatibility than the bare PEEK material in terms of cell adhesion, cell proliferation, and alkaline phosphatase (ALP) activity in an osteoblast culture. This brings an idea for the AIP-TiO_2 coating to use as a supplement for faster and better osseous integration on PEEK cages.

4:50pm **TS3-1-11 Investigation of Microstructure and Properties of Plasma-Sprayed HAp-TiO_2 graded Coatings**, ***A. Iwaniak*** *(aleksander.iwaniak@polsl.pl), M. Sozanska*, The Silesian University of Technology, Poland

The article presents results of microstructural investigation and properties characterization of HAp-TiO_2coatings on Ti6Al4V alloy. Hydroxyapatite and TiO_2coatings were deposited on titanium alloy substrates by atmospheric plasma spraying (APS) using of commercial powders. The layers hydroxyapatite/TiO_2bond coat consists different percent amount hydroxyapatite and TiO_2. The microstructural characterization of the HAp-TiO_2coatings before and after heat treatment was conducted by using scanning electron microscopy (SEM), electron probe microanalyser, X-ray diffractometer (XRD) and transmission electron microscopy (TEM). The as-sprayed coating consists mainly of crystalline HAp, rutile $TiO_2$and amorphous Ca-P phase. The results of the preliminary investigations were used to optimize the spraying conditions for the analyzed functionally graded hydroxyapatite depositions and, accordingly, the final graded coatings was obtained and cha racterized.

Coatings for Use at High Temperature
Room: Royal Palm 1-3 - Session A1-1

Coatings to Resist High Temperature Oxidation
Moderator: Y. Zhang, Tennessee Technological University, K. Stiller, Chalmers University of Technology

8:00am **A1-1-1 Platinum Nucleation Mechanisms and their Role in Platinum Aluminide Coatings**, ***M.A. Craig***, Cranfield University, United Kingdom, *L. Chirivi,* Cranfiedl University, United Kingdom, *K. Long, M. Robinson,* Cranfield University, United Kingdom, *D. Rickerby,* Rolls Royce, United Kingdom, *J.R. Nicholls (j.r.nicholls@cranfield.ac.uk)*, Cranfield University, United Kingdom

Platinum aluminide coatings are the bond-coat of choice for the aerospace industry in areas which are exposed to very high temperatures and corrosion products. Production methods for the deposition of the platinum vary; with industry favouring electroplating whereas academia favours sputtering by physical vapour deposition.

Platinum aluminide coatings have been shown in the past to show exceptional resilience to high temperature oxidation and corrosion. Various types of platinum aluminide coating are commercially available (RT22, SS82A, CN91 and MDC 150L type) and are all produced with an initial platinum layer deposited followed by aluminising (either in pack (RT22), out of pack (SS82A) or vapour deposition (CN91 and MDC150L).

This paper will show the importance of how the nucleation of the initial platinum layer affects the final structure of the platinum aluminide coating for both high and low temperature aluminide structures. Electrodeposition of platinum using the 5Q salt clearly shows that different nucleation mechanisms form different platinum aluminide coatings as the platinum, in some structures, acts as a diffusion barrier. The platinum deposited using PVD methods do not show as clearly that the microstructure has changed, however, cyclic oxidation tests are conclusive that nucleation affects the final quality of the coating.

8:20am **A1-1-2 Oxidation and Volatilization of Coatings for Power Generation: New Super Sultra Critical Steam Turbines**, ***F.J. Perez*** *(fjperez@quim.ucm.es), S.I. Castaneda, M.P. Hierro, F.J. Bolivar*, Universidad Complutense, Spain

The steels with chromium contents between 9 and 12% wt are used for power plants with advanced steam oxidation conditions. They present good creep properties as 9% Cr steels and good oxidation resistance, at the temperatures range between 500-600ºC. In the last years numerous investigation in development of coatings has been realized with the aim the protected them against the oxidation in order to allow operation of steam turbines at 650ºC. In this study, Al-Hf protective coatings were deposited by CVD-FBR on ferritic steel HCM-12A followed by a diffusion heat treatment, have shown to be protective at 650ºC under steam for at least 3000 hours of laboratory steam exposure under atmospheric pressure. Morphology and composition of coatings were characterized by different techniques, such as scanning electron microscopy (SEM), electron probe microanalysis, and X-ray diffraction (XRD). On the other hand, volatile species by mass spectrometry (MS) and thermogravimetric mea surements (TG) during the initial stages oxidation of the P92 ferritic steel without and of Al and Al/Si coating by chemical vapour deposition in fluidized bed reactors (CVD-FBR) at 650ºC in Ar+20%H2O for 150h were studied. In order to go forward to coatings design the Thermocalc code as used as base for the MS-data. TG-MS experiments were conducted in a closed steam loop in order to obtain information about the oxyhydroxides formation as reaction between coatings and steam. From those results the role of the different coating element could be established and optimized for the coating durability. An oxidation mechanism based on the TG-MS results is given. The morphology/composition and structure of the oxidized samples were also studied using SEM/EDS and XRD techniques.

8:40am **A1-1-3 High Temperature Coatings for Gas Turbine Engines**, ***B.T. Hazel***, GE Aircraft Engines **INVITED**

Waiting for clearance

9:20am **A1-1-5 High Temperature Oxidation Behavior of RF Magnetron Sputtered NiCrAl Coatings on Superalloys**, ***R.A. Mahesh*** *(ra.mahesh@gmail.com), R. Jayaganthan, S. Prakash*, Indian Institute of Technology, India

Magnetron sputter deposition is one of the most important techniques to produce thin films. Sputter deposition processes have been applied to produce overlay coatings for turbine components to improve oxidation resistance. Ni-Cr-Al ternary alloys are widely used for turbine blades, engine parts and many high temperature resistant components in energy production, aerospace and chemical industries. Since the 1970s, over-lay coatings of these alloys have been effectively used to provide oxidation and corrosion protection for the components exposed to hot gases. Microstructures have also been found to affect the high temperature corrosion resistance of many alloys. Researchers have showed that a small grain size promoted the formation of Cr2O3 protective scales on stainless steels. Ni-Cr-Al alloys are the main components of certain coatings used on Ni-base superalloys, therefore, the study of the oxidation of Ni-Cr-Al alloys is beneficial to the understanding of oxide forma tion of the complex coated superalloys. Recently, nanocrystalline coatings prepared by the magnetron sputtering technique have attracted much attention. In the present work, the oxidation behaviors of NiCrAl thin film deposited by RF magnetron sputtering was studied at 900ºC, which represents the average service temperature for turbine components. The oxide scale formed on the surface of the film was analysed by various techniques like XRD, FESEM/EDAX and X-ray mapping analysis. NiCrAl coated Superni 750 has provided better protection in the given environment.

9:40am **A1-1-6 Computational Investigation into the Phase Stability and Phase Distribution in the Ni-Cr-Al Ternary System**, ***K. Ma***, *J.M. Schoenung (jmschoenung@ucdavis.edu)*, UC Davis

The Ni-Cr-Al ternary system is of great significance in high temperature coatings. The stability and distribution of phases within the Ni-Cr-Al system are sensitive to the presence of a fourth element even if its content is not high. In this study, thermo-calc® software was applied to calculate the effects of C, N, O and Fe on the phase stability and phase distribution in the Ni-Cr-Al system, especially at elevated temperatures. The mass fraction of each phase, including Ni (Cr) γ phase, NiAl β phase, Ni_3Al γ' phase and so forth, was calculated for select temperatures in the range of 200°C up to 1600°C while allowing the concentration of the additional element to be variable. In addition, the diffusion path of the fourth element in the material system was illustrated by evaluating its distribution in the different phases as a function of temperature. It was observed that minor change in Fe or O content has a significant influence on the mass fraction of the primary phases in the Ni-Cr-Al system. The computational results were compared with our previous experimental study and they are consistent with each other.

10:00am **A1-1-7 New Generation HIPIMS Nanostructured Coatings to Perform in Severe High Temperature Environment**, ***P.Eh. Hovsepian*** *(p.hovsepian@shu.ac.uk), A.P. Ehiasarian,* Sheffield Hallam University, United Kingdom, *C. Leyens,* TU-Cottbus, Germany, *R. Braun,* German Aerospace Centre, Germany, *F.J. Trujillo*, Universidad Complutense de Madrid, Spain

CrAlYN/CrN were designed to meet the demands for highly oxidation resistant and wear resistant coatings for environmental protection of light weight materials such as @gama@-TiAl alloys for future applications in aero and automotive engines. CrAlYN/CrN coatings utilising nanscale multilayer structure with a typical bi-layer thickness of 4.2nm were successfully produced. The surface pre-treatment was carried out by bombardment with Cr^+ ions generated by High Power Impulse Magnetron Sputtering, (HIPIMS) discharge, whereas the nanoscale multilayer CrAlYN/CrN coating was deposited by Unbalanced Magnetron Sputtering (UBM) or by HIPIMS.

Scanning Transmission Electron Microscopy, (STEM) revealed that the coating/substrate interface was extremely clean and sharp. Large areas of coating grown epitaxially were observed. STEM-Energy Dispersive Spectroscopy (EDS) profile analysis further showed that during the HIPIMS ion bombardment Cr had been implanted into the substrate to a depth of 5 nm. In contrast to UBM coatings, HIPIMS deposited coatings showed extremely sharp interfaces between the individual layers in the nanolaminated material and almost no layer waviness. This improved structure resulted in further enhancement of the coatings barrier properties.

CrAlYN/CrN showed potential for reliable protection of @gama@-TiAl alloys against wear and aggressive environmental attack. For coated @gama@-TiAl alloys, thermo gravimetric quasi-isothermal oxidation tests in air at 750ºC after 1000 hours exposure showed four times smaller weight gain compared to the uncoated material. HIPIMS coatings were superior to UBM deposited coatings. Tested even at higher, 850ºC temperature the HIPIMS coatings showed by factor of 2 lower mass gain as compared to the UBM deposited coatings.

In sulphidation tests after 1000 hours exposure to aggressive $H_2/H_2S/H_2O$ atmosphere the CrAlYN/CrN protected @gama@-TiAl alloys showed

reduced weigh gain by factor of four as compared to the uncoated substrate. High temperature pin-on-disc tests revealed that CrAlYN/CrN reduces its friction coefficient from 0.56 at room temperature to 0.4 at 650°C, which demonstrates the excellent high temperature tribological behaviour of the coating. HIPIMS deposited coatings showed extremely low wear coefficient of KC= 1.83 E@-@17 $m^3 N^{-1}m^{-1}$ at this temperature.

10:20am **A1-1-8 Temperature Influence on Wear Characteristics of Functionally Graded Ceramic Coating by Detonation Gun**, ***S. Raja Kumar*** *(me06d007@smail.iitm.ac.in), L. Vijayaraghavan, M.M. Mayuram*, Indian Institute of Technology, India

Engineering design of tribo-contact pair is largely centered on design for minimal wear. Wear resistant ceramic/ceramic - composite coatings are widely employed to conserve material in numerous wear combating situations. Thermal sprayed ceramic coatings are widely used in a number of critical applications wherein the present scenario looks on multi-layer coating. Functionally graded coating (FGC)@super 1@ offers advantages in such situations. FGC's is developed by depositing layers of Alumina, Alumina-Titania (AT-13) and Alumina Zirconia (AZ-25) using detonation-gun (D-gun) process. The ceramic composite coatings are tested for their sliding wear behavior, at room temperature and elevated temperature@super 2@ by pairing with hardened surface of similar and dissimilar material. Monolayer deposit exhibits a higher wear rate at early stage of sliding at room and elevated temperature. The mono layer deposition has high wear rate due to localized spalling/pull out of material at roo m temperature on coated disk. With graded deposition, glazing with minimal localized pull out of material has occurred. The amount of wear largely depends on the layer proportions. The transfer of counter surface material (hardened steel) on to ceramic can contribute higher order sliding friction with FGC's at elevated temperature.

@super 1@ Xueliang Qiao, Yanhong Hou, Yiping Wu, Jianguo Chen, Study on functionally gradient coatings of Ti–Al–N, Surface and Coatings Technology, Volume 131, Issues 1-3, 1 September 2000, Pages 462-464. @paragraph2@super 2@B.S.Mann, Braham Prakash, High temperature friction and wear characteristics of various coating materials for steam valve spindle application, Wear 240 (2000) 223-230.

10:40am **A1-1-9 Investigation of an Oxidation Barrier for IN 617 at Low pO_2**, ***E.A. Clark*** *(eaclark@engineering.ucsb.edu), J.Y. Yang,* University of California, Santa Barbara*, A.G. Evans,* University of California Santa Barbara*, C.G. Levi*, University of California, Santa Barbara

Inconel 617 is a candidate material for He heat exchangers in the GenIV very high temperature nuclear reactor concept. However, impurities in the He may lead to degradation by oxidation, carburization or decarburization of the alloy. Alpha-alumina is attractive as a protective layer, but to assure that this phase forms (rather than transient aluminas) in the low pO_2 environment, at the use temperature (T≤1000°C), surface modification is required. This investigation focuses on the formation of alpha alumina at various temperatures and pO_2 environments using two surface modification approaches. (i) Diffusion aluminizing with subsequent heat treatment to the desired composition and phase constitution at the surface. (ii) Cladding (by diffusion bonding) with FeCrAl-Y/RE, an alloy, expected to readily form alpha alumina at low temperatures and low pO_2. The following concerns associated with these approaches are examined. The aluminized surfaces are prone to the formation of persistent transient aluminas. The clad materials exhibit rapid interdiffusion that may compromise the long-term durability of the barrier layer. Solution approaches are discussed in the context of recent research.

11:00am **A1-1-10 Stable Electrodes and Passivation Coatings for High Temperature Acoustic Wave Sensors**, ***B. Sturtevant***, *D.J. Frankel, G. Bernhardt, T. Moonlight, M. Pereira da Cunha, R.J. Lad (rjlad@maine.edu)*, University of Maine

A critical need exists for sensors that can monitor temperature, strain, and corrosion in strategic hot spots (up to 900°C) within a turbine engine. We have developed surface acoustic wave (SAW) sensors based on langasite ($La_3Ga_5SiO_{14}$) piezoelectric crystals that remain stable to at least 900°C. A major challenge for this technology is to develop oxidation resistant metallic electrodes and ultra-thin passivation coatings that can mechanically protect the active sensor surface. We have been able to produce electrodes that are stable up to 900°C by synthesizing nanostructured ultra-thin (< 100 nm) Pt-10%Rh / ZrO_2 electrode films grown by e-beam co-evaporation. X-ray diffraction (XRD), resistivity, and electron microscopy (EM) studies indicate that the incorporation of a dispersed ZrO_2 phase into a Pt-10%Rh electrode nanocomposite architecture prevents recrystallization and dewetting of the electrode film from the langasite substrate, which rapidly occurs for pure Pt electrodes. We have also developed ultra-thin (<50 nm) SiAlON passivation coatings that mechanically protect the sensor surfaces, yet allow interaction with the environment. Different SiAlON stoichiometeries were produced by rf magnetron sputtering of Al and Si targets in $O_2/N_2/Ar$ mixtures. The SiAlON films are amorphous and extremely smooth (< 1 nm rms) and remain so even after extended annealing at 900°C in air.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B5-1

Properties and Characterization of Hard Coatings and Surfaces

Moderator: Y.W. Chung, Northwestern University, G. Abadias, Universite de Poitiers-CNRS, M. Fenker, Forschungsinstitut Edelmetalle & Metallchemie

8:00am **B5-1-1 Contribution of an In Situ Microscopic Approach for a Better Understanding of the Oxidation Phenomena of TiSiN Nanocomposite Films Deposited on Steel**, ***P. Steyer*** *(philippe.steyer@insa-lyon.fr),* Laboratoire MATEIS, France*, A. Mège-Revil, G. Thollet,* INSA de Lyon, France*, R. Chiriac,* UCBL-LMI, France*, C. Sigala, C. Esnouf*, INSA de Lyon, France **INVITED**

Lifetime of TiN coatings is limited by its poor resistance at high temperatures. Often, an addition of silicon enhances both mechanical and physico-chemical properties, owing to the formation of a nanocomposite structure. In this study, pure Ti and TiSi (80/20) targets were arc-evaporated to produce hard, single-layered coatings. Magnetron sputtered SiNx films were also synthesized for a comparison purpose. The nanocomposite structure was validated by nanohardness measurements and confirmed by XRD and TEM. Through thermogravimetric experiments it is shown that in isothermal and dynamic conditions, the refractory character inherent to SiN_x governs the oxidation behaviour of TiSiN. However, in thermal cycling conditions TiSiN greatly resists while SiN_x does not withstand temperature variations. The aim of this study is to understand the role of SiN_x on the oxidation of the TiSiN nanocomposite film. For this purpose, an in situ approach of the oxidation phenomena is detail ed, based on experiments performed in a SEM operating in environmental conditions up to 1000°C. The "scenario" of attack is described: oxidation first initiates at coating defects (open pores and droplets), then extends throughout the surface. On the other hand, the poor thermal fatigue resistance of SiN_x is also evidenced at the microscopic scale by the whole flaking of the film during the cooling step. This strong propensity to cracking would be the consequence of the huge difference of thermo-mechanical behaviours between the film and the substrate. The nanocomposite structure thus allows to combine the refractory nature of SiN_x matrix with the beneficial thermo-mechanical effect due to TiN nanograins.

8:40am **B5-1-3 Synthesis and Oxidation Resistance of TiAlSiN and Multilayer TiAlSiN/CrAlN Coating**, ***N. Fukumoto*** *(naoya_bobob@livedoor.com), H. Ezura, T. Suzuki*, Keio University, Japan

Si incorporation to conventional nitride coatings have become a popular research in the area of hard coatings for cutting tools. The coatings with Si-incorporated show high hardness, high temperature oxidation resistance. This nanocomposite structure of crystal phase and tissue-like amorphous Si rich phase is the cause for the superior characteristics. Multilayer coatings are similarly widely studied and show unique characteristics compared to monolayer coatings. The choice of the two alternating layers and the multilayer period becomes an important factor for the coatings characteristics. For both types of coatings, the oxidation resistance is a demanding factor for usage in cutting tools and therefore the oxidation behavior needs to be clarified.

Here we report on the investigation into the oxidation resistant of Si-incorporated TiAlN monolayer and TiAlSiN/CrAlN multilayer coating. The coating was deposited by cathodic arc ion plating (AIP) with $Ti_{65}Al_{25}Si_{10}$ target at 300°C and bias voltage of 100 V. XRD and HR-TEM results showed that a cubic structured coating with a nanocomposite structure was formed for TiAlSiN coating. The existence of Si-rich amorphous phase at grain boundary was confirmed by EDX. The coating showed a high hardness of 38 GPa. The oxidation resistance was investigated by annealing the samples in air. Cross-sectional SEM observation and an elemental depth profile analysis were done by glow discharge optical emission spectroscopy (GD-OES). TiAlSiN coating showed good oxidation resistance with Ti,Si-rich oxide layer operating as diffusion barrier and nitride coatings still existed after 900°C. TiAlSiN multilayer coating with $Cr_{40}Al_{60}N$ was also deposited by AIP. The substrate was rotated at rotational speeds of 5.0 and 10.0 rpm to form two samples of different multilayer periods. Changes in characterization by forming a multilayer structure were observed by XRD, nanoindentation, and HR-TEM. XRD results showed that a single cubic

structured TiAlSiN/CrAlN multilayer coating was formed. From HR-TEM observation the multilayer periods were 4.3 and 9.8 nm. The multilayer coatings were also annealed in air to investigate its oxidation resistance by XRD, SEM, and GD-OES and compared with monolayer coatings.

9:00am **B5-1-4 Non-Linear Finite Element Modeling of Ultra-Hard Nanocomposites: The Ratio of Hardness to Yield Strength, Origin of the Ultra-Hardness and the Limits of Nanoindentation to its Measurement**, *M.G.J. Veprek-Heijman,* Technical University Munich, Germany, *R.G. Veprek,* Integrated Systems Laboratory, ETH, Switzerland, *A.S. Argon, D.M. Parks,* Massachusetts Institute of Technology, ***S. Veprek*** *(veprek@ch.tum.de)*, Technical University Munich, Germany

Using a non-linear constitutive material model for super- ($H \geq 40$ GPa) and ultra-hard ($H \geq 80$ GPa) materials that accounts for the pressure enhancement of elastic moduli and of flow strength[1], we show that the ratio of the hardness to yield strength (H/Y) amounts to about 2.3 to 2.9 for the super- and ultra-hard nanocomposites, even though the ratio of hardness to Young's modulus (H/E) is relatively high. These results are briefly discussed in terms of the expanding cavity model and the elastic-plastic transition. It is shown that a well defined elastic-plastic transition and the pressure enhancements are responsible for the different mechanical behavior of these materials, compared to that of softer ones having a similarly high H/E ratio but a lower H/Y, such as elastomers. These results lend strong support to our recent explanation of the origin of hardness enhancement in the ultra-hard nanocomposites[2]. We further present the effect of plastic deformation and resultant blunting of the diamond indenter, and discuss the limitations to the measurement on ultra-hard materials by means of nano-indentation.

[1]R. G. Veprek et al., Mater. Sci. Eng. A 448(2007)366.

[2] S. Veprek et al., Phil. Mag. Lett. 87(2007)955.

9:20am **B5-1-5 Secondary Ion Mass Spectrometry and Auger Electron Spectroscopy Used at nm Range Depth Resolutions and Tens of nm Range Lateral Resolution for Ultimate Characterization of Nanostructured Hard Coatings**, ***D. Duday*** *(duday@lippmann.lu), P. Choquet, V. Valle, J.N. Audinot, J. Guillot, V. Hody, H.N. Migeon*, CRP-Gabriel Lippmann, Luxembourg **INVITED**

The latest developments in the field of Secondary Ion Mass Spectrometry (SIMS) and Auger Electron Spectroscopy (AES) have provided powerful instruments for the characterisation of nanostructured hard coatings like nanocomposite or superlattice coatings. SIMS can now achieve a depth resolution in the nm range, which allows an excellent chemical characterisation of any chemical element including H even at trace concentrations in, for example, superlattice coatings. AES achieves a spatial resolution able to image small features of some 20nm like grains in hard coatings. The SIMS technique is also able to detect small quantities of contaminants (ppm or less) or isotopes with a spatial resolution of 50nm. Then, the influence of low concentrations of contaminants (<0.1at.%) on hard coating mechanical properties can be studied with SIMS. For a-C/M_xN nanocomposite film, SIMS also allows to compare the H content and repartition in different coatings of close composition and in some cases, using standard samples, it is possible to quantify the H content in coatings. The detection of isotopes by SIMS allows to determine the origin of elements in coatings e.g. O coming from coating or from oxidant atmosphere during oxidation resistance tests. The good spatial resolution of Auger and SIMS can also be used to study the chemistry inside the wear tracks. In this presentation, SIMS and Auger techniques, associated to TEM, will be used to analyse several nanostructured hard coatings before and after oxidation or wear tests with the objective to focus on new relationships between chemical composition, microstructure and wear or oxidation resistance.

10:00am **B5-1-7 Solid Solution Nanocomposite Cr-Si-N Thin Films**, ***L. Castaldi*** *(lorenzo.castaldi@empa.ch),* Empa, Switzerland, *V. Shklover,* ETH Zürich, Switzerland, *P. Schwaller,* Empa, Switzerland, *R. Sanjines,* EPFL, Switzerland, *J. Patscheider*, Empa, Switzerland

Cr-Si-N thin films were deposited by closed field reactive unbalanced magnetron sputtering onto cemented carbide and Si substrates in order to investigate the relationship between their nanostructure and mechanical properties. The systematic variation of the Si and Cr target power ratio allowed to adjust the Si atomic concentration Si/(Si+Cr) between 0 and 100 at. %. Structural analysis by X-ray powder diffraction (XRD) showed a shift of the cubic CrN diffraction peaks up to Si/(Si+Cr) ~ 7 % and a progressive decrease of the crystallite size with increasing Si/(Si+Cr). XPS analysis suggest the formation of solid solution Cr(Si)N up to Si/(Si+Cr) ~ 7 % and a segregation of X-ray amorphous SiN_x at higher Si/(Si+Cr) values, causing the formation of a nanocomposite consisting of a solid solution of Cr(Si)N and SiN_x. A maximum of hardness, strain to failure and resistance to plastic deformation was obtained in the region Si/(Si+Cr) ~ between 12 and 35 %, which is distinctively above the percolation threshold found for nanocomposites with (almost) zero solubility for Si. Optical and conductivity measurements were performed as well to investigate the relationship between Si/(Si+Cr) and the percolation behavior of the solid solution nanocomposite coatings.

10:20am **B5-1-8 Microstructure and Erosion Resistance of Nanocomposite Ti-Si-C-N Coatings Deposited using HMDSN Precursor**, ***R. Wei*** *(rwei@swri.org), C. Rincon, E. Langa*, Southwest Research Institute

Thick nanocomposite Ti-Si-C-N coatings (20-30µm) were deposited on Ti-6Al-4V substrates by magnetron sputtering of Ti in a gas mixture of Ar, N@2@ and HMDSN (hexamethyldisilazane) under various deposition conditions. Microstructure and composition of the coatings were studied using SEM, XRD and EDS. It has been identified that the Si concentration of these coating varies from 0% (TiN) to 16 at% (Ti-Si-C-N), while the structure of these coatings is similar to the nanocomposite Ti-Si-N coatings and consists of nanocrystalline TiC@0.3@N@0.7@ and/or TiN in an amorphous matrix of SiC@x@N@y@ with the grain size of 5 to100 nm, depending on the coating preparation process. These coatings exhibit excellent adhesion when subjected to Rc indentation tests at 150kG load. The mechanical properties of the coatings were studied using nano-indentation and micro-indentation techniques. The microhardness of these coatings varies from 1200 Hv (25g) to 3400, while the nano-hardness v aries from 10 to 26 GPa. Both the microhardness and nanohardness are lower than those of similar coatings prepared using TMS (trimethylsilane). However, the erosion test using a micro sand erosion tester at both 30@o@ and 90@o@ incident angles shows that these coating have very high erosion resistance up to a few hundred times improvement has been observed. There are a few advantage of using the HMDSN precursor to prepare the Ti-Si-C-N coatings over conventional magnetron sputtered deposition of Ti-Si-N coatings including composition uniformity, precursor handling safety and high deposition rate. The coating can be applied to protect gas turbine compressor blades from solid particle erosion and steam turbine blades from liquid droplet erosion, as well as other mechanical components that experience severe abrasion.

10:40am **B5-1-9 Microstructural and Mechanical Properties of Sputter Deposited TiN/SiN_x Multilayer Coatings on Si Substrate**, ***V. Chawla***, *R. Jayaganthan (rjayafmt@iitr.ernet.in), R. Chandra*, Indian Institute of Technology Roorkee, India

Titanium Nitride (TiN) has been widely used as protective coatings for bearings, gears and cutting tools due to its extreme hardness, high thermal and chemical stability. However, it oxidizes at 600°C which is of major concern for the aforementioned applications. To obviate these difficulties, TiN multilayer coatings, TiN/SiN_x, has been identified as potential material for realizing the improved oxidation resistance, ultrahigh hardness and strength properties. An interlayer, especially, ceramic layer, between TiN coatings, reduces the porosity, cutting off the paths for the corrosive solution to the substrate and thus increasing the protection against corrosion. Therefore, the development of multilayer coatings based on TiN/SiN_x with the desired microstructural morphology with good mechanical properties is gaining importance in recent times. The experimental studies pertaining to the influence of process parameters on microstructural and interfacial ch aracteristics of TiN/SiN_x coatings are limited in the literature. Therefore, the present work has been focused to investigate the microstructural and mechanical features of TiN/SiN_x multilayer coating deposited on Si substrate by DC/RF-Magnetron sputtering under different processing conditions. XRD was used to identify the formation of different phases and FE-SEM and AFM for microstructural characteristics of films formed under different process conditions. The formation of phases such as (111), (200) and (220) were observed. The equiaxed grain morphology in the TiN/SiN_x multilayer coating has been observed, which is due to activation of renucleation kinetics and periodic interruption of its growth due to the optimized sputtering conditions. HR-TEM has been used to characterize the crystal structure and orientation of the grains in the TiN/SiN_x coatings. The cross sectional TEM images of the coatings were analyzed to substantiate the morphology of TiN and SiN_x layers. The size of the TiN crystal was around 7.0 nm and SiN_x was found to be an amorphous matrix. The mechanical properties such as hardness and Young's modulus of the TiN/SiN_x coatings were measured by nanoindentation technique. The observed hardness of the coatings was around 30 Gpa. The deformation characteristics of the coatings were explained using its microstructural characteristics such as grain size and amorphous SiN_x.

11:00am **B5-1-10 Mechanical and Tribological Properties of Nano-Scale Multilayered TiN/CrN Coatings**, ***M. Konchady*** *(manohar82@gmail.com), S. Yarmolenko, D. Pai, J. Sankar*, North Carolina A&T State University

Multilayer TiN/CrN coatings with varying bilayer periods have been deposited on silicon (100) and 316 stainless steel substrates using reactive magnetron sputtering technique. Structural properties of the coatings were analyzed by X-ray diffraction (XRD) and cross-sectional transmission electron microscopy (XTEM). Multilayer coatings have shown predominant <100> orientation with superlattice structure formation below 20 nm bilayer period on silicon substrates. Nanohardness measurements, fracture indentation and scratch tests were performed to characterize the mechanical properties. Multilayered coatings, in general, have shown superior mechanical properties to both homogenous TiN and CrN coatings with nano-hardness values reaching 32 GPa in the nanometer range which is 40 % higher than the predicted value from rule of mixtures. Tribological behavior of the coatings was evaluated on micro-tribometer using reciprocating pin-on flat configuration with WC ball as counterface. Single-layer TiN coatings deposited using the same technique is used as a reference.

11:20am **B5-1-11 The Effects of Interlayer Grading and Pulsed Biasing on Chromium Nitride Films**, ***J.A. Freeman*** *(johnfreeman15@hotmail.com), P.J. Kelly, G. West,* Manchester Metropolitan University, United Kingdom*, N.M. Renevier,* University of Central Lancashire, United Kingdom*, J.W. Bradley*, University of Liverpool, Untied Kingdom

Although chromium nitride (CrN) coatings are an industry standard for hard wear-resistant thin films, methods of improving /modifying film properties are still being sought. Here we present the results of a study into the effects of interlayer grading, and the use of pulsed biasing (100-350 kHz) during both the sputter cleaning and deposition stages on the structural and tribological properties of CrN films. Pulsed biasing is known to significantly increase the ion current drawn at the substrate, compared to continuous DC biasing, which can modify film properties. And the use of a graded Cr-to-CrN interlayer can reduce interface stresses and, thereby, improve coating-to-substrate adhesion. Coatings were deposited reactively in a closed field unbalanced magnetron sputtering system. The substrates used were tool steel and aluminium coupons. The films were characterised by SEM, EDX and XRD and the tribological properties were measured by scratch testing, wear testing and n anoindentation. The preliminary results suggest both approaches can significantly improve the tribological properties of CrN films.

11:40am **B5-1-12 A New Approach to the Synthesis of Adherent Hard Coatings with High Toughness**, ***A.N. Ranade*** *(a-ranade@northwestern.edu),* Northwestern University*, L.R. Krishna,* International Advanced Research Centre (ARCI), India*, Y.W. Chung*, Northwestern University

Traditional ceramic coatings provide abrasive wear protection because of high hardness. However, these coatings have low fracture toughness, making them susceptible to surface or internal flaws and failure under high impact loads. In addition, when deposited onto metal substrates, lower thermal expansion coefficients of ceramic coatings compared to those of metals can cause thermal stress that may result in delamination. This paper explores a new approach to the synthesis of adherent hard coatings with high toughness. The approach begins with a metal matrix identical to that of the substrate, followed by the incorporation of nanoscale hard particles to increase hardness by Orowan strengthening. Theoretical estimates indicate that incorporation of 10 vol. % of such nanoscale particles can raise the hardness by as much as 20 GPa. Since the coating matrix is identical to that of the substrate, this should result in maximum adhesion and minimum thermal stress. Furthermore, by choosing nanoscale particles whose structure is semi-coherent with the metal matrix, local stress at the particle-matrix interface may activate the motion of screw dislocations, thus preserving the high fracture toughness of the matrix. This paper will present initial results of this exploration, using Ti as the matrix and semi-coherent TiB_2 nanoparticles as the strengthening agent. Characterization tools include x-ray diffraction (structure), AFM (surface roughness), SEM/TEM (size and distribution of nanoparticles), nanoindentation (elastic modulus, hardness, and fracture toughness), and scratch testing (adhesion), as a function of nanoparticle concentration. These studies should provide a general strategy for designing adherent hard coatings with high toughness.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D2-3

Diamond and Diamond-Like Carbon Materials
Moderator: O. Shenderova, International Technology Center, R. Hauert, Empa

8:00am **D2-3-1 Diamond Functionalizatin for Biosensing Applications and the Detection of NO_x**, ***J.T. Glass*** *(jeff.glass@duke.edu), J.B. Sund, C.P. Causey, S.D. Wolter, C.B. Parker,* Duke University*, B.R. Stoner,* RTI International*, E.J. Toone,* Duke University*, P. Natishan*, US Naval Research Laboratory **INVITED**

Due to its inert nature and carbon composition, diamond has long been presumed to be very biocompatible. Coupled with various exceptional properties such as strength, wear resistance, thermal conductivity and wide bandgap, it has long been considered to have high potential as a biomaterial. More recently, progress in surface functionalization and diamond electrochemistry have generated interest in diamond as a biosensor platform. The covalent bonding that is possible between carbon atoms at the diamond surface and carbon-based biochemicals implies robust and chemically specific surface modifications are possible. In particular, attaching specific surface molecular groups and utilizing electrochemical detection modes are expected to provide a wide array of versatile applications for diamond biosensors. These sensors have the potential to sense a variety of biochemicals from small molecules to proteins, thereby impacting metabolomics, genomics and proteomics. An increasing body of literature providing insight into the electrochemistry of diamond enables the design of sensing protocols, moving the sensors from laboratory curiosity to prototype.

This presentation will start with a general review of diamond biosensing followed by a discussion of more recent highlights in the literature. Specific results for the detection of a particularly important class of biomolecule, NO_x species, will then be discussed. In particular, it was found that NO (aqueous) oxidation at approximately 1V with hydrogen terminated boron-doped diamond in phosphate buffered solution was insufficiently distinct to electrochemically detect NO bubbled into the system. However, an amine-thiol functionalized Boron Doped Diamond surface provided a distinct oxidation peak from 25 nanomolar to 1 micromolar concentrations. Multiple cyclic voltammagrams with amine-thiol functionalized Boron Doped Diamond electrode indicated that the covalently attached surface molecules resisted decomposition during both anodic and cathodic potentials.

8:40am **D2-3-3 CVD-Diamond Coating on Steel Substrates: Basics and Applications**, ***J. Fandrey*** *(jan.fandrey@ww.uni-erlangen.de), K. Kellermann, S.M. Rosiwal, R.F. Singer*, University Erlangen-Nuremberg, Germany

CVD-diamond coating on steel promise an enormous potential for a multiplicity of technical applications. The combination of the unique properties of diamond (e.g. highest hardness, low coefficient of friction) and steel, the material with a countless number of applications, seems to be the material matching. However, the deposition of crystalline diamond coatings on steel comes along with several problems. Under the occurring coating conditions during diamond deposition iron acts as a catalyst for graphite formation on the steel surface. The metastable iron carbide decomposes into iron and graphite. And so a graphite layer grows, on which diamond deposition occurs without any adhesion to the steel substrate. To solve this problem, we use a diffusion chromium carbide interlayer which is deposited at 900 °C. The polycrystalline diamond layer shows excellent adhesion on this interlayer.A further problem is the great difference in the thermal expansion coefficients of diamond ($1.05 \cdot 10^{-6} K^{-1}$ at 20 °C) and steel ($\sim 10 \cdot 10^{-6} K^{-1}$ at 20 °C). This generates high compressive stresses in the diamond layer, already at low coating temperatures which directs to low diamond growth speed. By using the austenite-ferrite transformation, which leads to a volume expansion of the steel during cooling down, lower compressive stresses and therefore well adherent diamond layers can be achieved. The deposition processes (interlayer and diamond layer) act simultaneously as incorrect heat treatment processes for the steel substrate and affect consequently the mechanical properties.

In this presentation the basics for adherent diamond deposition on steel and first industrial applications are shown.

9:00am **D2-3-4 CVD-Diamond on Titanium Substrate as Electrode for Wastewater Treatment**, ***K. Bayerlein*** *(katharina.bayerlein@ww.uni-erlangen.de),* University Erlangen-Nuremberg, Germany, *M. Foreta,* Diaccon GmbH, Germany, *S.M. Rosiwal, R.F. Singer*, University Erlangen-Nuremberg, Germany

In recent years the investigation and application of boron doped diamond (BDD) electrodes for wastewater treatment and electrosynthesis are increased. Usually the diamond films are deposited on niobium or silicon as substrate material. To lower the substrate costs the substitution of niobium or silicon with titanium is aspired.

The in-situ formation of titanium carbide during the diamond coating process on titanium deteriorates the properties of BDD coated titanium electrodes for wastewater treatment due to the electrochemical instability of the carbide. The titanium carbide layer on the other side prevents the diamond coating from flaking of the substrate. The formation of the carbide is favoured at high substrate temperatures because of the higher diffusion of carbon in the β-microstructure. Pure Titanium transforms above 882°C from hexagonal α- to a cubic β-microstructure. The hydrogen rich atmosphere of the CVD-coating process adjusts this allotropic transformation temperature to lower values. Therefore substrate temperatures wide below the transformation temperature of 882°C have to be reached in the coating process to stabilize the α-microstructure and decrease the titanium carbide thickness. At this low substrate temperature also the diamond growth rate is very slow, which raise the production costs of stable electrodes.

An oxygen pretreatment can stabilizes the α-phase at the titanium surface. According to the time and temperature of this oxygen pretreatment and the diamond coating process parameters, it is possible to achieve α-phase, β-trans and mixtures of these in the microstructure. The pretreatment enables a CVD diamond coating process at substrate temperatures of 770°C with reduced titanium carbide thickness and still sufficient diamond growth rate.

9:20am **D2-3-5 Manufacturing and Properties of Self-Supporting Nano-Crystalline Diamond Foil**, ***M.A. Lodes*** *(matthias.lodes@ww.uni-erlangen.de), S.M. Rosiwal, R.F. Singer*, University Erlangen-Nuremberg, Germany

The hot filament chemical vapour deposition (HF-CVD) of crystalline diamond layers offers the possibility to bring highly wear-resistant coatings on heavily used technical surfaces. Besides the problem of adherence to the substrate the high process temperatures up to 900 °C limit the spectrum of materials to be coated. A two step process offers the possibility to circumvent these difficulties:

1. High temperature deposition of diamond on adequate templates which do not build any carbide interlayers.

2. Joining of grown self-supporting diamond foils with thicknesses of up to 80 µm to substrates at considerably lower temperatures afterwards, allowing the coating of otherwise uncoatable materials like polymers or light metals.

Process parameters like filament temperature, pressure and gas-flow of methane and hydrogen determine the microstructure of the diamond foils. Low pressure and high methane concentration lead to the growth of crystallites with a grain size in the range of nanometers. The surface quality of the template is mapped onto the diamond foil, for which reason the preparation of the template's surface plays an immensely important role. By altering the process parameters and the condition of the template's surface the foils' mechanical properties like Young's Modulus, hardness and fracture toughness can be tuned. The structure of the foils and the inherent residual stresses also strongly depend on the process parameters and are investigated.

Altogether this talk is to show how self-supporting diamond foils can be manufactured in a HF-CVD process and how the process parameters and the preparation of the template's surface influence their structure and mechanical properties.

9:40am **D2-3-6 Mechanical Properties of Ultrananocrystalline Diamond Films with Different Nucleation Densities**, ***G. Favaro*** *(gregory.favaro@csm-instruments.com),* CSM Instruments SA, Swizerland, *C. Popov,* University of Kassel, Germany, *W. Kulisch*, European Commission Joint Research Centre, Italy

The influence of the nucleation density on the development of the morphology of ultrananocrystalline diamond/amorphous carbon (UNCD/a-C) composite films and their mechanical properties has been investigated by variation of the substrate pre-treatment used to enhance the nucleation. The films have been prepared by microwave plasma Chemical Vapour Deposition (CVD) on silicon substrates. Their morphology and topography have been characterized by Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). It is shown that by successive addition of ultradispersive diamond powder (3-5 nm average grain size) to the suspension of nanocrystalline diamond powder (250 nm average grain size) in n-pentane used for the ultrasonic pre-treatment, the nucleation density can be enhanced by two orders of magnitude from 1 x 10^8 cm^{-2} to higher than 1 x 10^{10} cm^{-2} Advanced mechanical properties of these films have been investigated by Nanoindentation and Nano-Scratch testing. Nanoindentation measurements (NHT, CSM Instruments) were performed following the ISO 14577 standard with a diamond Berkovich indenter with a linear loading/unloading rate of 8 mN/min up to a maximum load of 4 mN. For the evaluation of the load/displacement curves, the Oliver and Pharr method was used, assuming a Poisson's ratio of 0.3. Five indentations per sample were performed at different positions. The determined hardness was about 25 – 28 GPa for all samples under investigation, the elastic modulus 262 - 271 Gpa, and the elastic recovery 62 - 65%. Some other measurements was done in order to better understand the behaviour of the thin film (stress-strain, working energy, fracture toughness) For the Nano-Scratch tests (NST, CSM Instruments), a diamond Rockwell C indenter was used. The experiments were performed with a progressive loading rate at a scanning speed of 6 mm/min. The critical loads for full delamination of the film and rupture of the substrate were determined from the images taken from the scratches. The scratch tests proved a strong adhesion of the UNCD/a-C coatings and their protective effect on silicon substrates. Finally, the correlation between the nucleation density, the macroscopic structure of the films and their mechanical properties is discussed.

10:00am **D2-3-7 Recent Developments in the Application of Single-Nano Diamond Particles**, ***E. Ōsawa*** *(OsawaEiji@aol.com)*, Shinshu University, Japan **INVITED**

Our understanding on the properties and behaviors of detonation nanodiamond advanced in recent years beyond comparison to the last 40 years. Following successful isolation of primary paricles with diameter 4.8±0.7 nm in 2003, a notable breakthrough was recently made by Barnard and Sternberg. They performed DFTB simulation of crystalline polyhedral models including truncated octahedra up to C1700, and found these nanocrystals have novel triple-decked structure consisting of sp^3 (diamond), sp^{2+x} (intermediate) and sp^2 (graphitic) carbons and their surface covered with high but localized electrostatic potential with signs and distributions of charges characteristic to facet types. Their proposal not only clarified the reason for agglutination among primary particles that occurred during detonation, but also solved a number of mysterious behaviors that have been observed in the past few years for the primary particles of detonation nanodiamond but remained unexplained. Sample distribution of primary particles began in 2006 and a handful of scientists who obtained the samples achieved considerable success. This lecture will concentrate on introducing Barnard's theory and the works of these pioneering scientists but with some new interpretations. The novel ultrananocrystalline diamond particles isolated in dispersed state are called here 'single-nano diamond (SND)' for simplicity. The following topics are mentioned: 1. Nanophase water on the particle surface of SND, 2. Drug carrier actions of SND hydrogel, 3. SND as seeding for homoepitaxial growth of CVD diamond film, 4.Lubricating capabilities of SND particles, 5. Production of SND on industrial scale, 6. Biocompatibility of SND, 7. Contribution to DNA sensor.

10:40am **D2-3-9 Comparative Study of Polishing Processes of Semi-Conductor Crystals and other Materials Surfaces by Detonation Nanodiamonds (DNA) and Colloidal Silica Compositions**, ***A.S. Artemov***, Russian Academy of Sciences, Russia, *V.M. Bogatyrev,* National Academy of Sciences of Ukraine, *S.B. Farafonov (artpol@land.ru),* State Technological University, Russia, *I.G. Ruzavin*, Lomonosov Moscow State Academy of Fine Chemical Technology, Russia

Expanding DNA application field in technologies of materials-processing demands a comparison with the modern leader of finishing - chemical-mechanical polishing technology (CMP) by colloidal silica.

The work purpose consisted in the comparative analysis and experimental study of basic polishing components (composition, pad, machine) work in technology of production of materials surfaces with ultimate geometrical and structural properties by using polycrystalline DNA particles and amorphous SiO_2 compositions.

Analyzing manufacturing technology of these particles, their structural and superficial properties (size, phase condition, modified chemical layers, etc.), colloidal-chemical properties of their water suspensions and the general requirements to optimum polishing compositions are formulated.

Experimental results of studying of colloidal-chemical properties of DNA and SiO_2 water suspensions and their mixes at different ratio w ith chemical agents allowed to define intervals of their sedimentation stability in a range of pH 1-14 and optimum viscosity values.

Polishing rate of Si, Ge, ZnO and other crystals by DNA compositions is several µm in hour while colloidal SiO_2 CMP depending on the equipment gives from 0,2 to 1,5 µm in minute. After polishing by DNA and its mixes with SiO_2 AFM methods revealed on a crystals surface nanoscratches by width of hundreds of nanometers. After SiO_2 CMP abrasion traces were not

found. Influence of modes of processing and pad type of two technologies on a relief roughness also determined.

TEM method indicated a processing dislocations absence in Si and Ge after CMP. RBS reveals the increasing of superficial areas structural perfection according to sequential reduction of size of diamond particles from 130 to 3-5 nanometers. The minimum amorphization depth (2-3 lattice parameter) corresponds to a surface after CMP.

Perspective areas of application of DNA compositions in comparison with CMP for achieve highly perfect surfaces of materials (semiconductors, dielectrics, conductors, composites) with high efficiency are defined.

11:00am **D2-3-10 Influence of the Processing Factors on Detonation Nanodiamond Surface Properties**, *Y.A. Fedutik, A.A. Antipov, A.A. Kalachev (Plasmachem@t-online.de),* PlasmaChem GmbH, Germany, *E.A. Maltseva,* Federal Institute for Materials Research and Testing, Germany, *A.N. Gorbacheva,* ***T.M. Gubarevich***, Institute of Physics of National Academy of Science of Belarus

In present work the influence of detonation synthesis and chemical purification conditions on the set of surface properties of DND was studied. Different DND powders (soots) were manufactured by "dry" and "wet" explosive techniques from identical charge composition mixtures "TNT - RDX" at different conditions of detonation products quenching.

Diamond–containing soot of each type was treated in similar conditions by common oxidizing mixtures on basis of mineral acids: $H_2SO_4 + HNO_3$; $H_2SO_4 + HNO_3 + SO_3$; $H_2SO_4 + NaNO_3$; $HClO_4 + HNO_3$; $H_2SO_4 + KMnO_4$ as well as by air oxygen over an inhibitor B_2O_3[1]. Nanodiamonds purified by each mentioned technique were subjected to comparative investigations by the following parameters: specific area and absorption – structural powder behavior (BET method with nitrogen, noble; polar and non-polar gases), surface chemical functional groups composition (FTIR - spectroscopy), protonogenic center density on the DND surface (potentiometric titration), particle size distribution (dynamic light scattering) and sedimentation stability in aqueous media (nano-sedimentometry).

On the basis of the results obtained, as stable as labile nanodiamond surface parameters were revealed, their range of changing depending on DND production and chemical purification conditions was presented. The possibilities and restrictions of approved technologies for the new low – aggregative DND particles[2] manufacturing were shown, as well as for some industrial application (composite plating, nano-abrasives and nano-lubricants).

super 1@Purification of detonation diamond. Chiganov A.S., Chiganova G.A., Tushko Yu.M., Staver A.M. Russian Patent n2004491 from 15.12.93.

super 2@ Yu. A. Fedutik, A.A. Antipov, E.A. Maltseva, A.A. Kalachev, T.M. Gubarevich. Non-aggregated nanoparticles of detonation diamonds. – in book: proceeding of the 3rd International Symposium Detonatio Nanodiamonds: technology, properties and applications. July 1-4, 2008, St-Petersburg, Russia, p.65-72.

11:20am **D2-3-11 The Thermostability of Detonation Nanodiamond**, ***A.N. Panova***, *G.P. Bogatyreva (bogatyreva@ism.kiev.ua),* National Academy of Sciences of Ukraine, *V.J. Zabuga, G.G Tsapyuk,* National Taras Shevchenko University of Kyiv, Ukraine, *S.A. Lisovenko*, National Academy of Sciences of Ukraine

Detonation nanodiamond is presently used with high efficiency for processes of polishing, superfinishing and besides as adsorbents and catalysts of different processes.

Due to unique specific properties such as overdeveloped surface in combination with the original structure characteristics and nanodimensional effects, the nanodiamond is a perspective material for medicine to make a range of various new treatments.

The new use of nanodiamond is the instruments with the nanodiamond coating for effective cutting. Nanodiamond particles are also doped as carbon binding into the hard metal compositions in order to increase their strength, hardness and productivity. At that, the nanodiamond capacity to maintain the constant chemical structure and physical properties at high temperature conditions i.e. its thermostability is one of the questions of high importance. On that reason the investigation of nanodiamond powders thermostability is very important and perspective.

The aim of our work was to study the effects of various factors on nanodiamond powders thermostability and to establish the main regularities to their oxidizing process.

The experiments were conducted on samples of nanodiamond grades ASUD-75 and ASUD-99. The grades were prepared from a mixture synthesized by the Ukrainian company ALIT with detonation of oxygen-deficient explosives.

Thermooxidation of nanodiamond powders was analysed at different thermal conditions. At nonisothermal conditions thermooxidation of samples was assessed by derivatography at the temperature range from 273 to 1273K at a heating rate of 5 deg/min. The kinetic of thermooxidation of nanodiamond by atmospheric oxygen was studied by gravimetric control of the sample mass variation at the constant temperature for an hour and by the kinetic method using the "coal in cylindrical beaker" model as well as by methods with chromatography control for given reaction product.

The predominated influence of chemical composition and energetic state of nanodiamond surface on thermostability of nanodiamond powders at the temperature range from 273 to 1273K has been shown. The kinetic parameters of chemical reaction of diamond with atmospheric oxygen were determinate.@It was stated that the objective characteristic for thermostability of diamonds may be the rate constant of chemical reaction of diamond with components of aggressive environment.

11:40am **D2-3-12 Influence of Surface Roughness on the Tribological Properties of HF-CVD Diamond Coated Heat-Treatable Steel**, ***K. Kellermann*** *(karsten.kellermann@ww.uni-erlangen.de), S. Ehrhardt, J. Fandrey, S.M. Rosiwal, R.F. Singer*, University Erlangen-Nuremberg, Germany

Coating of steel surfaces is a standard technique to adjust the tribological properties for the requirements of technical applications. Crystalline CVD diamond, due to its unique properties like the highest wear resistance and a marginal friction is an ideal coating material for severe applications. Especially in mechanical face seals CVD diamond is increasingly popular as engineered surface coating. Steel as substrate material with CVD diamond seems to be an extraordinary combination.

In the last years the process route for diamond coating of steel was developed. To prevent graphite formation during diamond deposition on the iron surface because of the decomposition of the metastable iron carbide we use a diffusion chromium carbide interlayer. The temporary volume expansion caused by phase transformation of the steel substrate during cooling down from coating temperature (~ 800 °C) reduces the compressive stresses in the diamond layer and enables a homogeneous and well adherent deposition.

In this study we have investigated the tribological properties of diamond coated C35 (1.0501) specimens, that were differently mechanically treated before HF-CVD-coating and chromium-carbide-coating respectively to achieve different surface roughnesses. The tests were performed in a water lubricated ring-on-disc configuration to simulate conditions of mechanical face seals. The pre-treatment of the steel surface has high influence on the friction and wear of the CVD-diamond-coating, which is explained in a model for tribological behavior of diamond coated steels.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E2-3

Mechanical Properties and Adhesion
Moderator: R. Chromik, McGill University, L. Davies, Caterpillar, J. Michler, Empa

8:00am **E2-3-1 Mechanical Properties of Metals Below 100 nm - Effects of Size, Temperature and Geometry**, ***R. Spolenak*** *(ralph.spolenak@mat.ethz.ch)*, ETH Zurich, Switzerland **INVITED**

Metals become stronger and stronger, when their external dimensions approach the micrometer scale. When their dimension, however, is reduced by another two orders of magnitude, novel effects appear: the scaling law for yield strength changes, strength depends on the loading mode, metals become more brittle and some metals start to exhibit a strong temperature dependence in their mechanical properties. These observations will be illustrated by studies on nonporous gold in a combinatorial approach, thin single crystal gold films by nanoindentation and in-situ X-ray studies on arrays of gold nanointerconnects. The lecture is rounded off by theoretical considerations of scaling limits and how they can be used to design optimal materials microstructures.

8:40am **E2-3-3 Hardness Mapping and Nanoindentation of Cold-Spray Coatings**, ***D. Goldbaum*** *(dina.goldbaum@mail.mcgill.ca), T. Shariff, A. Rezaeian,* McGill University, Canada, *E. Irissou, J.-G. Legoux,* National Research Council Canada (NRC), Canada, *R. Chromik, S. Yue*, McGill University, Canada

Cold-spray is a relatively new method of coating deposition that is achieved at low temperatures by accelerating powders to supersonic speeds through a converging-diverging nozzle. Upon impact, the particles undergo high shearing stresses with localized adiabatic shear instability regions. The

material deformation aids in creating a metallurgical bond to the counterpart surfaces, but also results in inhomogeneous strain within particles and perhaps throughout the coating. In this work, hardness profiles and hardness mapping were used to investigate the effect of the deposition conditions and material deformation mechanisms on the nanomechanical properties of cold-sprayed coatings. Pure Cu, Ni, Ti and Ti alloys powders were deposited on a mild steel substrate with particle velocities measured in situ and ranging from 400-800m/s. Nanoindentation testing was conducted on polished cross-sections of coating/substrate specimens. When averaging all measurements for a given coa ting, a trend for hardness with increasing deposition velocity was found, with hardness increasing for Ti coatings, decreasing for Cu coatings, and remaining constant for Ni coatings. A closer inspection of data showed no consistent trends with indent position with respect to the substrate. Instead, variations in hardness were observed between particles and within them as well. Hardness mapping is currently being compared to determinations of strain from electron backscatter diffraction (EBSD). Differences in coating mechanical properties will be discussed in terms of the particle deformation, microstructure and morphology.

9:00am **E2-3-6 Ultra Low Indentation Approach to Stress-Strain Behavior of Thin Films**, *J. Nohava (jiri.nohava@csm-instruments.com),* CSM Instruments SA, Switzerland, *G. Favaro,* CSM Instruments SA, Swizerland, ***N. Randall***, CSM Instruments Inc.

The elastic-plastic behavior of thin films has always been of a great interest. However, obtaining of a plot similar to uniaxial stress-strain curve has been facing serious methodological and technical obstacles. Recently, nanoindentation methods have been employed for determination of such curves and theories relating indentation stress and strain to uniaxial stress and strain were developed. The new CSM Instruments Ultra Nanoindentation Tester has been used for measuring of elastic-plastic response of several thin films. Bulk materials were also used for comparison. The main advantage of the UNHT instrument is its extremely low thermal drift and frame compliance that are indispensable for long term and cyclic measurements. The measurement method consisted of performing repeated indentation with increasing force on one spot of the tested sample. The indentations were carried out using linear and quadratic load increase. Spherical indenters of 10 µm and 20 µm radii were used for indentation. The load applied on the indenter varied from loads of 0.05 mN to stay in the elastic regime up to high loads of 50 mN to enter well into the plastic regime. The indentation stress as a function of indentation strain was plotted and the curve similar to uniaxial stress-strain curve was obtained. The so-obtained curves were compared with plots of elastic to plastic indentation energy ratio as a function of depth. The onset of plastic deformation was observed and it corresponded to change in the elastic to plastic indentation energy ratio. The results show that though such measurements do not have the same physical meaning as the well known stress-strain curve, they can be used as an efficient and fast method to determine the elastic-plastic response of thin films to mechanical deformation in compression.

9:20am **E2-3-5 Mechanical Properties of TiN/Ti Multilayer Coating Deposited by LAFAD Technology**, ***Y. Cheng*** *(yh_cheng@yahoo.com), T. Browne, B. Heckerman,* American Eagle Instruments, Inc., *C. Bowman, V.I. Gorokhovsky*, Arcomac Surface Engineering, LLC.

Multilayer coatings consisting alternating ceramic and metallic layers have attracted great attentions due to the combination of the high hardness of the ceramic layer and the high toughness of the metallic layer. Our previous results showed that the addition of Ti interlayer into TiN ceramic layer significantly reduce the internal stress in the coatings. It is important to characterize the mechanical properties, i.e. hardness, Young's modulus, and plasticity, of the TiN/Ti multilayer coatings. A large area filtered arc deposition (LAFAD) technique was used to deposit TiN/Ti multilayer coatings with fixed TiN layer thickness and different Ti layer thickness. Scanning electronic microscopy and nanoindenter were used to observe the surface morphology, and characterize the hardness, Young's modulus and plasticity of the multilayer coatings, respectively. The dependence of the surface morphology and mechanical properties of the coatings on the Ti interlayer thickness was systematically studied. It was found that all TiN/Ti multilayer coatings deposited by our LAFAD technique are dense and smooth. The increase in the Ti layer thickness results in a increase in the grain size and surface roughness, a decrease in the effective hardness and Young's modulus, and an increase in the plasticity and toughness. The coatings with Ti layer thickness of below 50 nm possess excellent combination of high effective hardness (>20GPa), high plasticity (>69%), and excellent toughness.

9:40am **E2-3-4 Small-Scale Tests for Investigating Plastic Flow in Brittle Crystals**, ***S. Korte*** *(sk511@cam.ac.uk), W.J. Clegg*, University of Cambridge, United Kingdom

Plastic flow in brittle materials is conventionally studied by testing material that is sufficiently constrained to prevent cracking, using, for instance, a Griggs cells or, much more easily, by nanoindentation. Much useful information has been obtained in these ways, however, many brittle crystals have complex structures where multiple slip systems operate. In indentation, accommodating the indenter tip in the sample requires that sufficient slip systems operate, so that the measured mechanical behaviour is dominated by the properties of the hardest slip system operating. An alternative approach is to make the sample sufficiently small that insufficient elastic strain energy is available to drive cracking. This can be done using micropillar compression and has been used extensively in metals to study volume effects on flow. These effects are much less marked in hard materials, so that this approach can be used to study flow. Furthermore, it allows deformation based on a single slip system. Here, microcompression of MgO pillars with subsequent TEM analysis was used to demonstrate this. MgO single crystals possess a hard and a soft slip system which can be targeted individually orienting the crystal such that the Schmid factor is close to 0.5 for one and zero for the other. Micropillars of different crystal orientations were subjected to uniaxial compression and analyzed by transmission electron microscopy to identify the Burgers vectors and slip planes involved in order to correlate these results with the respective mechanical properties in each direction.

10:00am **E2-3-7 Mechanical and Electrical Behavior of Oxide-Dispersion Strengthened Au Microcontacts**, ***R. Vinci*** *(rpv2@lehigh.edu), T. Bannuru, W.L. Brown, T. Humplik, K. Mongkolsuttirat*, Lehigh University **INVITED**

Incorporation of hard ceramic particles in the surface layer of a metal component can be an effective method for reducing sliding wear. We have extended this approach to address adhesion and degradation of micrometer-scale thin gold film contacts in MEMS devices. Vanadium pentoxide nanoparticles are formed in a gold matrix during reactive sputter deposition. Control of the deposition parameters determines the extent of vanadium oxidation and the volume fraction of the oxide phase. Nanoindentation has been used to characterize the hardness of the films as a function of composition and oxidation extent. Together with characterization of electrical resistivity via four-point probe measurements, the ideal contact resistance under normal force loading can be predicted. We measure the real contact resistance using a custom ball-on-flat test configuration. This test is also used to evaluate adhesion during separation of the contacts. Film strength, contact resistance, and degree of adhesion can be modeled using approaches borrowed from prior studies of either oxide-dispersion strengthened alloys or metal matrix composites. General conclusions are drawn about the applicability of these measurement techniques and models to nanoparticle-reinforced gold films, and about the potential of this reinforcement approach for enhancing wear resistance in normal-force contacts without unacceptable increases in contact resistance.

10:40am **E2-3-9 Factors Affecting the Critical Indenter Penetration for the Measurement of the Hardness and Young's Modulus of Coatings**, ***S.J. Bull*** *(s.j.bull@ncl.ac.uk), J. Chen*, Newcastle University, United Kingdom

In many coating applications, it is very important to obtain the mechanical properties (such as hardness, elastic modulus, etc.) of the coating independent of the substrate for instance for use in design calculations. The nanoindentation test is only viable approach to assess the properties of very thin coatings (<1µm) since it can operate at the required scale and provides a fingerprint of the indentation response of the coating/substrate system. If coating properties are to be assessed, the key point is to ensure any measured value is free from the influence of the deformation of substrate (or at least the substrate effect is minimized). To measure the hardness of the coating only it is traditionally assumed that, as a rule-of-thumb, when the relative indentation depth (RID, i.e the penetration divided by the coating thickness) is less than 0.1, the substrate will not affect the measured hardness of the coating. However, it is found that this rule is too strict for some systems and too loose other coated systems. A more rigorous assessment is therefore necessary. For the Young's Modulus a much smaller critical penetration is expected and there is doubt about whether it is possible to measure the elastic properties of the coating independent of the substrate at all. In this paper we present a comprehensive investigation on the critical relative indentation depth (CRID) using finite element simulation. The elastic-plastic properties mismatch between coating and substrate has been highlighted and other factors affecting the CRID which have previously been ignored will be discussed. The condition that indenter penetration must be less than a fraction of the coating thickness has been explored and it has been shown that in a particular type of coated system no rule-of-thumb can apply. Furthermore, it is shown that elastic property

mismatch has an important effect on the measured hardness and this means that the Oliver and Pharr method generally used to extract hardness from nanoindentation data may give inaccurate results in coating/substrate systems with significant elastic mismatch.

11:00am **E2-3-10 Influence of Intrinsic Stress on the Measurement of the Onset of Yielding for Thin Films Using Depth-Sensing Spherical Indentation**, ***M. Herrmann*** *(matthias.herrmann@physik.tu-chemnitz.de), A. Clausner, F. Richter*, Chemnitz University of Technology, Germany

It is often observed that hardness is influenced by intrinsic stress in the sample. With increasing uni- or biaxial tensile stress hardness is reduced while with growing compressive stress it increases. This observation complies with basic considerations about the influence of those stresses on shear stresses and is thus frequently used. However, there were also reports which could not prove this finding. For instance in[1] a variation of hardness with changing stress was shown to be due to varying pile-up. When this was properly considered, a stress-independent hardness was obtained.

In contrast to hardness, the determination of yield strength refers to the initial stage of yielding and should therefore be better suited to investigate the influence of intrinsic stress. Such measurements are done by loading-partial unloading experiments with spherical indenters and analysing the stress field for the onset of plastic deformation. Here, the intrinsic stress field is considered in addition to the field due to the loaded indenter.

We have investigated amorphous hydrogenated carbon (a-C:H) films deposited on silicon having varying compressive stress. The yield strength of the films was determined using the von Mises yield criterion and their hardness was measured applying the Oliver-Pharr method. The intrinsic film stresses do influence the onset of plastic deformation and, hence, have been considered in the analysis of yield strength. To interpret the obtained hardness data, one should keep in mind that a proper hardness testing is connected to a fully-developed plastic zone, thus including an intensive amount of plastic deformation. This can lead to a dramatic change of the stress fields within the volume of the sample and therewith to the above-mentioned effects. However, for the investigated a-C:H films it was found that the ratio of hardness to yield strength was constant within the accuracy of measurement.

[1]T.Y.Tsui et al, J.Mater.Res. 11(1996)752-759.

11:20am **E2-3-11 Influence of the Nitriding and TiAlN Coating Thickness on the Mechanical and Tribological Properties of a Duplex Coating System on H13 Die Steel**, ***R. Torres*** *(ricardo.torres@pucpr.br),* PUCPR, Brazil, *L. Suzuki,* Neodent Co, Brazil, *P. Soares,* Pontificia Universidade Católica do Paraná, Brazil, *C. Lepienski,* Universidade Federal do Paraná, Brazil, *J.J. Moore*, Colorado School of Mines

H13 die steel substrates were low pressure gas nitrided with three different nitriding cases, A, B, and C. The hardness profile revealed that two surface hardness levels and two thickness depths of the nitrided layer were obtained. In the nitriding case A the surface hardness was around 12 GPa and the nitriding thickness around 40 µm. In the nitriding case B, the hardness was the same as in case A, but the nitriding thickness was around 70 µm. The nitriding thickness in case C was the same as in case B, but the surface hardness was around 14.5 GPa. The XRD results showed that the nitriding case microstructure is composed mainly of a diffusion layer with a small amount of CrN precipitates. These nitrided samples were subsequently coated with TiAlN using cathodic arc evaporation in two thicknesses of 3 and 7 µm. These duplex coating samples were characterized with respect to phase chemistry, adhesion, hardness and scratch tests. The phase chemistry determined through X RD and EDS revealed that the TiAlN coating was mostly $Ti_{0.7}Al_{0.3}N$ with some peaks of TiN. The instrumented hardness performed on the coated samples showed that the coating hardness changes with the nitriding case depth for a TiAlN coating thickness of 3 µm. On the other hand, the nitriding characteristics do not influence the coating hardness when the TiAlN coating is 7 µm thick. In addition the 7 µm thick coating is harder than the 3 µm thick coating. In the last part of this work, TiAlN was deposited on the H13 substrate without nitriding; it was found that the hardness in this condition is higher than the duplex nitrided/TiAlN coated samples. The worn area probed by the scratch test was smaller for the samples coated with TiAlN of 7 µm thickness.

11:40am **E2-3-12 Deformation Mechanisms in TiN/NbN Multilayer Thin Films**, ***K.A. Rzepiejewska-Malyska*** *(karolina.rzepiejewska@empa.ch),* Empa, Switzerland, *A. Baranska, A. Szerling,* Institute of Electron Technology, Poland, *S. Korte, W.J. Clegg,* University of Cambridge, United Kingdom, *J. Michler*, Empa, Switzerland

The aim of the study was to compare TiN/NbN multilayered ultrathin film systems regarding deformation mechanisms during indentation. TiN/NbN films with different combinations of single layer thicknesses were deposited in the sputtering process with two different temperatures, ~70 and ~1000°C respectively. Single layer reference coatings of TiN and NbN were synthesized for comparison. Mechanical properties were determined by in situ nanoindentation inside a high resolution Scanning Electron Microscope using a cube corner indenter tip. This allowed for observation of pile-up, sinking-in and crack propagation of the coating during the indentation loading cycle which were taken into account for hardness and reduced modulus calculation. In situ SEM indentation at the microscale allowed for observation of the toughness and crack propagation in this scale. In order to get a complete understanding of the fundamental deformation phenomena occurring within the layers and to anal yze the microstructure, transmission electron microscopy (TEM) observations on the cross-sections of the indented areas were prepared using a focus ion beam (FIB) technique. TEM observations revealed that films deposited in lower temperature were nanocystalline, while those obtained with higher temperature were coherent nanocrystalline coatings. We have observed that the mechanical behavior strongly depends on the microstructure of the film. For polycrystalline multilayer films grain boundary sliding was identified as one of the acting mechanisms, while for monocrystalline samples shear band formation and cracks perpendicular to the substrate dominate. The difference in deformation mechanisms observed will be discussed and linked to the features occurred on the load – displacement curves.

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G6

Coatings, Pre-Treatment, Post-Treatment and Duplex Technology

Moderator: K. Bobzin, RWTH Aachen University, E. Kusano, Kanazawa Institute of Technology

8:00am **G6-1 Exploring the Potential of Triode Plasma Diffusion Treatments to Improve the Wear Behaviour of Titanium Alloys**, ***G. Cassar*** *(mtp06gc@sheffield.ac.uk), A. Leyland, A. Matthews*, The University of Sheffield, United Kingdom

Owing to their many attractive properties, nowadays titanium alloys have a wide range of static load-bearing applications. However, there is still concern on their adaptability to use on tribological components. Continued research and development in surface engineering has to some extent improved the wear-resistant capabilities of these alloys. Diffusion-based surface treatments of titanium alloys have received increased attention, but many of these are performed at high temperatures exceeding the β-transformation temperature of the material and/or for excessively long periods of time. This work is focused on developing a cost effective process which will allow the use of titanium alloys for tribological applications even under relatively high loading. The use of low pressure intensified plasmas was shown to allow the diffusion of nitrogen (for example) to generate deeper hardened layers than conventional thermal or diode plasma processes. The process temperature w as limited to 800°C in order to minimize degradation in mechanical properties of the bulk material. The treated specimens were tested and characterized using a variety of techniques using X-ray diffraction, optical microscopy, scanning electron microscopy, microhardness, nanoidentation, reciprocating wear tests and micro-abrasion wear tests. The results obtained indicate that the proposed technique is indeed capable of significantly improving the surface characteristics of Ti alloys.

8:20am **G6-2 RF-Plasma Technique for Duplex Coatings on Aluminum**, ***S. Meier*** *(Sven.Meier@iwm.fraunhofer.de), M. König, C. Hormann*, Fraunhofer Institute for Mechanics of Materials IWM, Germany

An innovative approach to improving behavior of aluminum surfaces and meeting long-term durability requirements of aluminum devices is to design and develop novel systems incorporating duplex diffusion/plasma coating treatments. The composite layers consists of a N-diffusion zone obtained by rf-plasma nitriding, followed by a rf-plasma deposited DLC film. A RF-PACVD processing technology is described that helps to overcome some of the most important limitations of existing technology to generate duplex coatings on technical aluminum substrates. Application of this technique to plasma assisted nitriding of pure aluminum and different aluminum alloys is demonstrated. The influence of the process parameters on the discharge characteristics was examined. Material properties and tribological testing results are shown for both materials.

8:40am **G6-3 Plasma Nitrocarburizing and Laser Hardening Duplex Treatment of AISI 4340 Steel**, ***V.H. Baggio-Scheid*** *(scheid@ieav.cta.br), G.de Vasconcedos, A.J. Abdalla*, General-Command of Aerospace Technology, Brazil

The 4XXX steel series are structural steels widely used in automobile and aerospace components. However, when the applications involve wear, a protective coating or surface treatment might be required. In this work the duplex process of plasma nitrocarburizing and laser hardening of AISI 4340 steel was investigated. The plasma nitrocarburizing process was conducted at 773 K during 3 h. Hard cases with 790 HV and 160 μm thickness have been measured. A CO_2 laser with a power density of 70 kW cm^{-2} was used for the surface heat treatments. In order to overcome the high reflectivity of the metal, their surface was covered with a thin absorber layer of graphite powder. After the laser irradiation a top graphite layer of about 10 μm was incorporated to the steel surface. Below this graphite coating a diffusion layer with 20 μm thickness and a maximal hardness of 1200 HV has been formed. The heat affected zone extends up to 100 μm towards the matrix. Duplex processes involving combinations of these two treatments were carried out. At all, five different processes have been done: Nitrocaruburizing, Laser hardening with graphite layer, nitrocarburizing plus laser hardening, nitrocarburinzing plus laser hardening with graphite layer, Laser hardening with graphite layer plus nitrocarburizing. The properties of the treated surfaces were investigated by optical microscopy, scanning electron microscopy, X-ray diffraction and by microhardness and wear testing. Influences of the process parameters on the coating properties are analyzed.

9:00am **G6-4 Dedicated Coatings for High Precision Cutting Tools – Coating Design and Architecture Adjusted with Most Modern Process Technology**, ***T. Leyendecker***, *R. Cremer (Rainer.Cremer@CemeCon.de)*, CemeCon AG, Germany **INVITED**

Metal-Ion-Sputtering as well as Diamond coating technology are essentially strong tools for creating designed coatings. In some segments "coatings off-the-shelf" may be still appropriate, but steadily increasing demands in performance and specialized operations and workpiece-materials require more and more adapted tools and tailored coatings. Moreover each machining operation, whether drilling, turning, milling, reaming, threading, gear cutting or grooving has different needs. The dedicated coatings are developed in a defined and reproducible process consisting of: - Definition of the objective - Determining of coating specifications - Production of test tools, evaluation and, if necessary, optimization - Finalization of the specifications Tool micro- and macro-geometry as well as tool material are closely related to the corresponding coating. Nowadays coating is a complex but controlled process merging different elements und steps, like pre-treatment, coating material, coating thickness, tolerance, color, post treatment and others. Only for sputtered coatings all compounds and combinations of metals and non-metals in different structures are possible. New technologies like HPPMS (High Power Pulsed Magnetron Sputtering) can produce unsurpassed high coating-adhesion and -smoothness.

9:40am **G6-6 Edge Honing for High Performance Drilling Operations**, ***M. Maes*** *(Michel.Maes@kometgroup.com)*, KOMET GROUP GmbH, Germany

Modern machining processes are exposed to an enormous pressure regarding their cost efficiency. Only highly efficient processes like High Speed Cutting (HSC), High Performance Cutting (HPC), High Hard Cutting (HHC) are able to survive within these countries. Also new high-tensile constructive materials like Sibodur 700-1, Austempered Ductile Iron (ADI), Compact Graphitic Iron (CGI) and glass fibre reinforced plastics that are used in the automotive and aerospace industry require new demands for tooling concepts. High performance cutting tools are the key to enable this demand for cost effective machining processes and machinibility of these high-tensile materials. Therefore tool suppliers have to generate durable tool concepts that also allow high material removal rates to sustain cost effective machining. Within this presentation, tool concepts are presented that focus on high performance drilling. The carbide inserts used are optimised for this machining purpose with respect to substrate and coating. It is well known that the cutting edge radius has a tremendous effect on both edge toughness and cutting forces. Therefore the cutting edge was prepared with different radii to evaluate suitability for several drilling operations. The effect of the cutting radius on machining performance and cutting forces is evaluated within this presentation.

10:00am **G6-7 Process Temperature and Post Annealing Effects on Microstructure and Mechanical Behavior of the Sputtering Ni-P-Al Coatings**, *Y.M. Su, J.C. Wu,* ***F.B. Wu*** *(fbwu@nuu.edu.tw)*, National United University, Taiwan **INVITED**

The Ni-P-Al ternary coatings were fabricated by magnetron sputtering technique with composite target design. The effects of thermal treatments, including post annealing and deposition temperature, on phase transformation phenomenon and related mechanical properties were investigated. The as-deposited coatings produced under process temperature below 475°C showed an amorphous/nanocrystalline microstructure. Significant recrystallization of Ni matrix and precipitation of Ni-P and Ni-Al compounds were observed for the coatings manufactured under high sputtering temperature of 500 to 600°C. In comparison, the amorphous Ni-P-Al coatings were post-annealed at 500 to 600°C in vacuum environment. The amorphous feature of the Ni-P-Al coating remained under a high annealing temperature of 550°C, showing a better thermal stability as compared to those fabricated under high process temperatures. Furthermore, the hardness and indentation behavior of the coating under various thermal histories were evaluated. Superior hardness and mechanical behavior were found for the post-annealed Ni-P-Al coatings due to volumetric constrain of recrystallization and precipitation. On the other hand, the overaged phenomenon and subsequent degradation in hardness were found for the Ni-P-Al coatings fabricated under high temperature deposition processes. The phase transformation mechanisms of the coatings through different thermal treatments were intensively discussed.

10:40am **G6-9 Influence of Different Post-Treatment Techniques on Arc Coated Carbide Drills**, ***S. Sattel*** *(stefan.sattel@guehring.de), I. Garrn*, Guehring oHG, Germany

Using arc PVD coatings on cutting tools for improved function is well established since the last two decades. The usage of pre- and post-treatment of coated tools expands more and more to a key knowledge of optimizing the performance of coated tools. Post-treatment is mostly important for the smoothness of the flute, the decreasing of friction and the reduction of residual stress in the coating. Different mechanical post treatment techniques such as magnet finishing and brushing will be compared with automatized dry- and wet-blasting, as well as granulate grinding. All these methods have been used on similar pre-treated and coated carbide drills in order to examine the influence of the post-treatment on the tool performance. All carbide drills have been coated with a standard TiAlN based film using an arc-PVD-technique for deposition. Depending on the different post-treatment techniques the carbide drills have been examined concerning to their surface roughness, possible changes of the cutting edge and finally to their durability.

New Horizons in Coatings and Thin Films
Room: Tiki Pavilion - Session H1-1

Nanotube, Nanowire and Nanoparticle Thin-Films and Coatings
Moderator: R. Martel, Université de Montréal

8:00am **H1-1-1 Chiral Architectures in Thin Films: Fabrication and Applications**, ***M.J. Brett*** *(brett@ece.ualberta.ca)*, University of Alberta, Canada **INVITED**

The fabrication of thin films with a chiral or helical microstructure has enabled development of a variety of devices that utilize the ability of these coatings to interact with circular polarized light, mimic the periodicity of the diamond lattice, and behave as microscopic springs. Techniques used to make thin films with a helical architecture include serial bideposition[1] and direct laser writing using 2-photon absorption[2]. We use Glancing Angle Deposition (GLAD)[3], a physical vapour deposition process that combines the self shadowing effects of oblique incidence vapour flux with precise substrate motion. Recent developments in optimizing architectural control of chiral films and in organic materials by GLAD will be highlighted, in particular for periodic or self-organized arrays. A variety of devices based on chiral thin film structures will be described. Photonic crystal structures based on the square spiral geometry will be presented, including our initial efforts to fabricate inverse square spirals. Other devices such as chiral optical filters, electrically variable nanospring interferometers and thin film chromatograph layers will be described as time permits. [1] [1] I. Hodgkinson and Q.H. Wu, Adv. Mater. 13, 889 (2001).[2] K.K. Seet, V. Mizeikis, S. Matsuo, S. Juodkazis, and H. Misawa, Adv. Mater. 17 (2005).[3] M.M. Hawkeye and M.J. Brett, J. Vac. Sci. Technol. A25, 1317 (2007).

[28:40am] **H1-1-3 Shape Modification of Silica Submicroparticles Assembled in Compact Monolayer by Dry Etching**, ***S. Portal*** *(sabine.portal@ub.edu), M.A. Vallvé, M. Rubio-Roy, C. Corbella, J. Ignes-Mullol, B. Bertran*, University of Barcelona, Spain

Monolayers of silica submicroparticles were produced by self-assembly methods and were subsequently nanostructured by dry etching. The first step of the process concerned the synthesis of the particles from an alkoxide precursor by sol-gel process. The particles obtained were spherical with a size ranging from 300 to 500 nm. In a second step they were assembled in

closed-packed 2D crystal monolayers by means of the Langmuir-Blodgett (LB) technique. The structure was transferred on silica and silicon wafers. The LB technique allowed to control surface compactness of the colloidal crystals as well as the number of the particle layers deposited on the substrate. Further nanostructuring of monolayer films was made by etching with Ar ion beam at 550 eV, 7.10-2Pa and room temperature over areas of about 3 cm^2 of the particle film. The incident beam angle was varied from 0º to 60º with respect to the normal of the substrate in order to change the morphology of the etched 2D crystal. The shape of the submicroparticles was altered and the particle size decreased but the original close-packed arrangement was conserved. The etching process both affected the particles and the interstitial uncovered substrate. The substrate etching was more pronounced for long time and high etching angle: this resulted from a higher physical sputtering yield at higher angle; the particle shadowing effect accentuated the nanostructuring of the substrate. Surface nanopatterning using silica particles as templates is one possible application of this technique. In turn, large nanostructured wafers could be used as template itself for nanoimprinting. The main advantages of this technique consist in nanostructuring over large areas compared to nanolithography and also the possibility of controlling the final features of the pattern by selecting the adequate particle size, etching time and beam angle. Applications in photonics, sensors,catalysis and tribology are envisaged.

9:00am **H1-1-4 Self-Organized Functional Metallic Nano-Arrays by Combined Ion Beam Sputtering and Plasma Deposition**, ***T.W.H. Oates*** *(oates@ifm.liu.se), J. Rosen,* Linköping University, Sweden*, A. Keller, S. Facsko,* Forschungszentrum Dresden-Rossendorf, Germany*, S. Noda*, University of Tokyo, Japan

We demonstrate the formation of self-organized aligned arrays of silver and cobalt nanoparticles; important materials in nanophotonics and nano-scale magnetism. The arrays are produced using a combination of ion beam sputtering and physical vapor deposition. Self-organized periodic ripple patterns with 3 nm amplitude and 35 nm period are formed on silicon and silicon oxide by low-energy ion beam sputtering. These are subsequently used as template substrates for the deposition of metal island films, promoting aligned nucleation and growth of metallic nanostructures. A combinatorial mask deposition technique is utilized to investigate optimal deposition conditions using magnetron sputtering, including deposition rate, film thickness and post-deposition annealing. Investigations into the use of directed velocity plasmas from a pulsed cathodic vacuum arc are also presented. Silver nanoparticle arrays display biaxial localized surface plasmon resonance absorption due to anisotropic interparticle coupling[1]. Under appropriate deposition conditions arrays of cobalt nanowires, many microns in length, are formed[2]. A large range of self-organized substrate materials and patterns are available to this simple, scalable technique.

[1]T.W.H. Oates, A. Keller, S. Facsko, A. Muecklich, Aligned silver nanoparticles on rippled silicon templates exhibiting anisotropic plasmon absorption, Plasmonics, 2(2) 47-50 (2007).

[2]T.W.H. Oates, A. Keller, S. Noda, S Facsko, Self-organized metallic nanoparticle and nanowire arrays from ion-sputtered silicon templates, Applied Physics Letters 93 063106 (2008).

9:20am **H1-1-5 Nanostructures and Mechanical/Optical Properties: a New Approach for Transparent Functional PECVD Coatings**, ***S. Costacurta*** *(costacurta@civen.org), P. Falcaro, S. Vezzù, F. Marinello,* Associazione CIVEN, Italy*, E. Zanchetta, M. Guglielmi,* University of Padova, Italy*, A. Patelli*, Associazione CIVEN, Italy

Nanoparticles are increasingly used in coating technology in order to impart specific functional properties to surfaces. Nanoparticles are typically embedded in matrices such as sol-gel or polymeric films. However, these coatings often require post-treatments which may be inadequate either for the embedded nanoparticles or for the substrate on which they are deposited. For example, a thermal treatment after deposition may alter the functional properties of the nanoparticles, or the substrate can be thermally degraded. Furthermore, these coatings often possess insufficient mechanical properties and so they are often unusable from an industrial viewpoint. Coatings obtained by plasma-enhanced chemical vapour deposition (PECVD) and atmospheric pressure plasma jet (APPJ) are good candidates as matrices for nanostructures with improved functional or mechanical properties and are expected to widen the industrial application fields of such nanostructured coatings. In this work we present a preliminary study of an approach to anchor nanoparticles to a surface in order to impart specific properties, based on the deposition of nanoparticles via dip-coating and the fixing of such nanoparticles by depositing a ceramic film via PECVD and APPJ. This method was applied to the preparation of nanostructured coatings based on silica nanoparticles synthesised by the sol-gel technique and fixed on different substrates for antiscratch applications. The fundamental aspects of this process such as surface density and size of the nanoparticles and evaluation of their functional efficiency were considered. The diameter of the silica nanoparticles was varied between 60 and 200 nm and different densities of nanoparticles (number of particles per coating volume) were tested. These coatings were studied by AFM, SEM, FTIR, the mechanical tests were performed by microscratch. The coating showed an increase in antiscratch performances, avoiding crack propagati on and decreasing penetration depth. Optimised conditions were found in order to avoid the interference fringes, maintaining transparency. The present method provides a means to overcome the difficulty in anchoring the nanoparticles to different types of substrates. It can be generalised to fabricate multilayered films and can be extended to the use of nanoparticles encompassing many sizes and chemical compositions, exhibiting different functional properties (e.g. optical properties and antibacterial activity).

9:40am **H1-1-6 Nano Multilayer Transparent SiO_x-Based PECVD Coatings for Barrier and Protective Applications**, ***A. Patelli*** *(patelli@civen.org), S. Vezzu', S. Costacurta, L. Zottarel, M. Colasuonno*, Associazione CIVEN, Italy

Transparent corrosion protection or gas barrier and diffusion blocking coatings are increasingly requested in various application fields, and above all, on flexible substrates. The silicone or epoxy-based paints, already in use for protection of metal sheets, show problems of ageing, highlighted by a progressive yellowing. They are characterised by low mechanical properties such as anti-scratch or adhesion. Paints are also used as barrier for nickel and chromium diffusion for costume jewellery or glasses frame or for heavy elements in perfume and pharmaceutical packaging and also for gases in food packaging. In order to face these application fields at CIVEN it has been developed a ceramic/organic multilayer barrier coating deposited in a simple single-step RF-PECVD process. HMDSO has been used as precursor in an oxygen atmosphere and the ceramic or organic layer behaviour is obtained just pulsing the precursor flow in order to change the precursor/oxygen ratio and keeping all the other parameters constants. The multilayer system allows flexibility by means of the organic layers and barrier effect and corrosion protection by means of the ceramic layer. The different deposition phases are monitored during the process by a mass spectrometer, in order to control different layers composition and interfaces thickness and modulation. The multilayer interface thickness has been investigated by SIMS as a function of the deposition parameters and coating features have been characterised by ellipsometry and FTIR. The ceramic layer thickness has been varied from 5 to 50nm, and different series as a function of number of layers and interlayer thickness have been deposited in order to study the multilayer design effect on mechanical properties. Moreover the gas barrier properties will be investigated showing the effect of the multilayer design on permeability. Finally, in order to show the possibility of such kind of coating to accommodate surface defects preserving the corrosion protection properties, particles of about 400 and 900nm of diameter will be deposited on the substrate before coating growth. The evolution of the morphology and topography of the surface will be followed by SEM and AFM, and defects effect on corrosion protection and permeability will be highlighted.

10:00am **H1-1-7 Patterning of Semiconductor Surfaces with Ordered Metallic Nanowires and Nanoparticles via Self-Assembly**, ***J.M. Buriak*** *(jburiak@ualberta.ca), J. Chai*, University of Alberta, Canada

Metal nanostructures continue to be the focus of intense research because of their fascinating properties that can be distinctly different form their bulk counterparts, and thus show great promise for a range of applications. One of the key ingredients required for these future applications is the ability to integrate numerous 1D addressable nanostructures through assembly, patterning, and alignment on technologically relevant solid supports, such as semiconductor surfaces. The challenge lies in fabricating large areas of high density metallic nanostructures, with feature sizes below 100 nm, in an economically feasible manner. While photolithography will justifiably remain a core technology with respect to upcoming, sub-65 nm nodes on the semiconductor industry association roadmap, cost considerations for mass manufacturing will be one potential constraint. As a result, there is interest in the development of complementary patterning strategies that involve large scale self-assembly, for use as a soft organic template for metal nanostructure development.[1] Here we describe our work towards the use of self-assembled block copolymer nanostructures on technologically relevant semiconductor materials, to produce metallic features, using approaches compatible with existing silicon-based fabrication. [1] Buriak et al, Nature Nano, 2007, 2, 500-506. Buriak et al, ACS Nano, 2, 489-501.

10:20am **H1-1-9 Carbon Nanotube Films as Transparent, Conductive and Flexible Electrodes**, ***C. Zhou*** *(chongwuz@usc.edu)*, University of Southern California **INVITED**

I will present our progress on the preparation and application of carbon nanotube films as transparent, conductive, and flexible electrodes. These films have been further exploited as hole-injection electrodes for organic light emitting diodes (OLEDs) on both rigid glass and flexible substrates. Our experiments reveal that films based on arc discharge nanotubes are

overwhelmingly better than HiPCO-nanotube-based films in all the critical aspects, including the surface roughness, sheet resistance, and transparency. Further improvement in arc-discharge nanotube films has been achieved by using PEDOT passivation for better surface smoothness and using SOCl2 doping for lower sheet resistance. The optimized films show a typical sheet resistance of ~160 Ω/□ at 87% transparency and have been successfully used to make OLEDs with high stability and long lifetime.

11:00am **H1-1-12 Structural Characteristics and Photocatalytic Capabilities of Visible Light Responsible Nanoporous TiO_2/ITO Composite Thin Films**, ***C.H. Hung***, National Kaohsiung First University of Science and Technology, Taiwan, *K.R. Wu (krwu@nkmu.edu.tw),* National Kaohsiung Marine University, Taiwan, *W.C. Lo,* National Kaohsiung First University of Science and Technology, Taiwan, *T.P. Cho*, Metal Industries Research and Development Centre, Taiwan

This study investigates photocatalytic properties of nanoporous titanium oxide (TiO_2)/indium tin oxide (ITO) composite thin films and shows their capabilities to both pollution control and hydrogen production. There are two types of ITO substrates, i.e. commercial ITO (c-ITO) and in situ sputtered ITO (s-ITO) glass, used in the study. Both of the TiO_2 thin films contain some levels of nitrogen in the lattices. The experimental results indicate that both films exhibit primarily a crystallized anatase TiO_2 phase by being characterized with X-ray diffraction (XRD) and Raman spectra. The XRD spectra of the TiO_2 thin films also show a distinct preferential (211) plane along with extremely weak (101) and (004) planes. The shift of preferential plane is mainly due to the diffusion of tin ions from the ITO substrate. Very low doping concentration of N (~0.2 %) estimated by X-ray photoelectron spectroscopy (XPS) at the N1s peak of ~396.8 eV is responsible for visible-light photocatalytic activity for both of the catalysts. However, the secondary ion mass spectrometry (SIMS) elemental depth profiles and the cross-sectional transmission electron microscopy (TEM) images reveal different degree of tin diffusion between the catalysts. For the TiO_2/s-ITO catalyst, tin ions can significantly permeate into the overlaid TiO_2 thin film and promote the formation of crystalline $Ti_{1-x}Sn_xO_2$ layer, resulting in a better photocatalytic oxidation capability than the TiO_2/s-ITO over methylene blue. Conversely, the photoelectrochemical activity of the TiO_2/c-ITO catalyst is enhanced due to a higher Schottky barrier formed at the interface between TiO_2 and c-ITO layers. Thus, the TiO_2/c-ITO catalyst shows a remarkable photocurrent density of 180 @micro@A/cm^2, as compared with that of 100 @micro@A/cm^2 of the TiO_2/s-ITO thin film under UV illumination.

Bioactive Coatings and Biofunctionalization
Room: Sunset - Session TS3-2

Bioactive Coatings and Surface Biofunctionalization

Moderator: D.V. Shtansky, State Technological University "Moscow Institute of Steel and Alloys", S. Kumar, University of South Australia

8:00am **TS3-2-1 Surface Functionalization of High Strength Biomedical Ceramics**, ***J.R. Piascik*** *(jpiascik@rti.org),* RTI International, *J.Y. Thompson,* NOVA Southeastern University, *E.J. Swift,* University of North Carolina at Chapel Hill, *S. Grego, B.R. Stoner*, RTI International

There has been much advancement in the development of ceramics for biomedical use, expanding the use of high strength materials (e.g. alumina and zirconia) in varying prosthetic and reconstructive applications. In dentistry, high strength ceramics have become a popular alternative to traditional porcelains, due to their superior fracture resistance and long-term viability. A major clinical problem with the use of indirectly placed alumina and zirconia restorations is the difficulty in achieving adequate bonding with the underlying substrate (tooth structure, implant abutment). Traditional adhesive bonding techniques used with silica-based ceramics like porcelain do not work effectively with these higher strength materials. Initial investigations focused on functionalizing alumina and zirconia surfaces with an ultra-thin Si-based layer (1-25 nm thick) allowing for silane treatments to tether hydro-carbons. These hydro-carbon groups are a common reactant with acrylic poly mers, allowing for strong covalent bonding between prosthesis and the underlying structure. As a proof of concept, several planar test structures, consisting of dental alumina and zirconia, were prepared, treated, and analyzed, before and after Si-O modification, using x-ray photoelectron spectroscopy (XPS). XPS studies indicated that the pretreatment process deposited less than five monolayers of Si_xO_y on the surfaces. A commercially available silane was then applied to the treated surfaces. High resolution core scans of the C(1s) and Si(2p) supports successful chemical bonding of the organo-silane to the Si-O treated surfaces. Mechanical property analysis, consisting of micro-tensile testing of zirconia bonded to a dental composite, revealed that pretreatment of bonding surfaces increased the failure strength over traditional bonding techniques.

Research was supported by NIH/NIDCR DE013511-09 and an internal research grant provided by RTI International.

8:20am **TS3-2-2 Osteoblast Adhesion to Metal-Oxide Incorporated Diamond-Like Carbon Films**, ***L.K. Randeniya*** *(lakshman.randeniya@csiro.au), A. Bendavid, S. Amin, P.J. Martin,* CSIRO Materials Science and Engineering, Australia, *R. Rohanizadeh*, University of Sydney, Australia

The interaction of osteoblast cells with diamond-like carbon (DLC) films modified by the oxides of Si, Ti, Zr and Hf is investigated. The films were prepared using a pulsed direct-current plasma-activated chemical vapour deposition method. The correlations between cell-attachment properties and the surface energy/chemical composition of the films are discussed. The variations in the mechanical properties (hardness and residual stress) as a function of the concentrations of oxides in the films are presneted. Using these results, the feasibility of these films as coatings for prosthesis in orthopaedic and blood contact applications is discussed.

8:40am **TS3-2-3 Tribology and Corrosion Behavior of Alpha Alumina-coated Ti-6Al-4V for Surgical Implantation**, ***M.T. Montgomery***, University of Arkansas, *P. Mohanty,* University of Michigan, *H.H. Abu-Safe,* Lebanese American University, Lebanon, *M.H. Gordon (mhg@uark.edu)*, University of Arkansas

Alpha alumina thin films are excellent candidates for implantation due to the material's resistance to corrosion, wear, and protein adsorption. Applying such films to a suitable bulk material can be problematic however, as current methods reach high temperatures unsuitable for commonly used alloys or involve undesirable template materials such as chrome.

Thin films of reactively sputtered aluminum oxide were deposited at 480C on Ti-6Al-4V ELI substrates with no template layer. Process parameters (including magnetron power, substrate biasing, process pressure, oxygen partial pressure, film thickness, and substrate orientation) were varied to investigate the influence of deposition conditions on film quality, crystallography, and mechanical and corrosion behaviors. Film phase has been determined by XRD. Corrosion behavior was assessed using electrochemical impedance experiments. Tribological testing of film hardness and wear resistance supports the XRD resul ts. A detailed discussion of the effect of varying deposition parameters on the properties and performance of the coatings will be presented as well as a comparison to previous literature results.

9:00am **TS3-2-4 Fabrication of Hierarchical Wrinkled Micro-Pillars using Diamond-Like Carbon Coating**, ***Y. Rahmawan***, Seoul National University, Korea, *Sk.F. Ahmed,* Korea Institute of Science and Technology, Korea, *K.J. Jang,* Seoul National University, Korea, *K.S. Kim,* Brown University, *M.W. Moon (mwmoon@kist.re.kr),* Korea Institute of Science and Technology, Republic of Korea, *K.R. Lee,* Korea Institute of Science and Technology, Korea, *K.Y. Suh*, Seoul National University, Korea

Superhydrophobic surfaces can be utilized to prevent cells proliferation that causes most of failures in biomedical devices. We have shown novel method to fabricate superhydrophobic surfaces based on 3-D hierarchical wrinkle structures combined with the replica molding to make the micro-scale pillar array on PDMS as the soft base material, covered by nanoscale wrinkle patterns formed with hydrophobic diamond-like carbon (DLC) using RF-CVD to make nanometer scale roughness. Nanoscale wrinkle patterns were controlled by varying the thickness of film, causing different pattern width.

Superhydrophobicity of the surfaces can be achieved when we have 3-D hierarchical structures on pillars with spacing ratio of 1 to 4. It was found that theoretical wetting analysis was well matched the experimental results that the wetting states are Cassie and Cassie for microscale roughness and nanoscale roughness, respectively (C^m-C^n) at spacing ratio of 1 to 4, while transformed into Wenzel and Wenzel (W^m-W^n) at spacing ratio higher than 5.

The superhydrophobic surface with hierarchical wrinkled patterns was applied for the cell template with the Calf Pulmonary Artery Endothelial (CPAE) cells. It was observed that the CPAE cells were very difficult to adhere to the 3-D wrinkled surface and the rejection of filopodia extension was observed on the protrusion of nanoscale wrinkles. Further more, as the spacing between the pillars close to 1, the filopodia spreading was highly hindered by the small focal adhesion point of the cells on hierarchical structures since the filopodia spread from top to top of the pillars. This

limited focal point adhesion of the cells to superhydrophobic surfaces would prevent the growth and proliferation.

9:20am **TS3-2-5 Anti-Microbial Properties of Silver Modified Amorphous Carbon Films**, *A Almaguer-Flores (argelia.almaguer@mac.com),* Instituto de Investigaciones en Materiales, Universidad Nacional Autónoma de México, Mexico, *R Olivares-Navarrete,* Department of Biomedical Engineering, Georgia Institute of Technology, *L.A. Ximénez-Fyvie, O. Garcia, S.E. Rodil*, Universidad Nacional Autónoma de México

Silver has been used as an anti-microbial material since historic times. It is well known that microbial infection on implant surfaces has a strong influence on healing and long-term outcome of dental implants. During the last year we have been investigating amorphous carbon (a-C) films as a surface modification for metallic dental implants, but the antimicrobial properties of the a-C films evaluated using oral bacteria and compared to medical grade stainless steel (SS) were similar. Therefore, in this work, we tested if by addition of silver we could improved the anti-microbial properties of amorphous carbon films. The a-C:Ag films were deposited by co-sputtering using a graphite target (10 cm diameter) with an small piece (1 cm^2) of pure silver and under an Argon plasma. Bacterial adhesion on the a-C, a-C:Ag, the SS substrates and samples of Ti6Al4V (which is the standard implant material) was evaluated during 24 hrs, 3 and 7 days of incubation. We used nine oral bacteria strains: Actinobacillus actinomycetemcomitans serotype b, Actinomyces israelii, Campylobacter rectus, Eikenella corrodens, Fusobacterium nucleatum ss nucleatum, Peptostreptococcus micros, Porphyromonas gingivalis, Prevotella intermedia and Streptococcus sanguinis. After anaerobic incubation, the total number of colony forming units (CFUs) was counted and the surfaces were observed under the scanning electron microscope (SEM). The effect of including silver on the a-C films was characterized by Raman, X-ray Diffraction, Energy Dispersive spectroscopy, Atomic Force Microscopy and SEM. The results showed that under certain deposition conditions the films have silver nanoparticles (40-60 nm) uniformly distributed in the carbon matrix. The silver was crystalline with a maximum composition around 10 at%. Concerning the antimicrobial properties we did not observe a great effect of the silver particles on the CFU's after 24 hrs and 3 days of incubation, but small reduction was observed after 6 days. Nevertheless, in this study we showed that the a-C surface has better anti-microbial properties than the Ti6Al4V.

9:40am **TS3-2-6 Rapid Antimicrobial Susceptibility Testing of Escherichia Coli and Staphylococcus Aureus by Using the Cr-Cu-N Nanocomposite Thin films with Gradient Copper Content**, ***Y.-J. Chang***, *K.-J. Weng, J.-S. Lu, J.-T. Chen, J.-L. Li, J.-W. Lee (cwlee@mail.tnu.edu.tw)*, Tungnan University, Taiwan

The E-test is an antimicrobial agent gradient-coated plastic test strip which allows for quantitative antimicrobial susceptibility testing, or Minimum Inhibitory Concentration (MIC), on agar media. In this study, The Cr-Cu-N nanocomposite thin films were deposited on SUS304 substrates with different Cu contents using bipolar asymmetry pulsed DC reactive magnetron sputtering system. The thin film with gradient copper content ranging from 3.5 to 20.0 at.% was achieved by adjusting the distance between the Cu/Cr targets and substrates. The antibacterial tests of thin films containing different copper content were performed to evaluate the bactericidal ability against both E. coli and S. aureus. A film attachment method (modified JIS Z 2801:2000) was adopted for evaluating the antibacterial activity of the specimens with uniform Cu contents. The MICs were estimated after 24 hr incubation on the measurement of the elliptical zone of growth inhibition due to the copper ion wh ich diffused from the coupons with gradient copper content. It was observed that the interspaces of the antibacterial zones are almost proportional to the antibacterial rates under different copper content of thin films. The nanocomposite thin films with gradient copper content produced in this work are useful for rapid screening antimicrobial susceptibility testing.

10:00am **TS3-2-7 Electrochemical Characterization of Biocompatible TiO_x and NbO_x Films**, ***P.N. Rojas*** *(nayuritkd@yahoo.com.mx), S.E. Rodil, H. Arzate, S. Muhl*, Universidad Nacional Autónoma de México

The corrosion resistance of materials in the body fluids is one essential factor to determine the lifetime of medical implants. Therefore, it is of great relevance to understand the interface process occurring when a surface is exposed to a body fluid, which can be studied by impedance spectroscopy. Titanium and Niobium oxide films were deposited on medical grade stainless steel using a magnetron sputtering system working. These films were evaluated using Potentiodynamic polarization (PP) in 0.89% NaCl (7.4 pH) in order to determine the conditions that lead to the best corrosion resistance. The biocompatibility of best films was evaluated by adhesion and viability/proliferation assays using human cells. Moreover, the long-time stability of the films was evaluated by electrochemical impedance spectroscopy (EIS) as a function of time up to 500 hrs using three different simulated body fluids; the NaCl solution and Hartman (Ringer's + Lactate) and Gey's (Ringer's + Phosphates + Glucose) solution, in order to correlate their protective properties against different composition of electrolytes. The results indicated that there are strong differences according to the chemical composition of the solution and different electrochemical response can be obtained for the same coating, just by changing the solution. For example, NbO_x showed a better resistance than the TiO_x films in the Hartam's solution but it failed when Gey's solution was used. Meanwhile TiO_x showed a well passivated response for both NaCl and Gey's solution. The EIS spectra were analyzed using circuit models in order to understand the different kinetic and interfacial processes occurring for each film and solution.

10:20am **TS3-2-8 Effect of Modes of Corrosion on Cellular Viability of Ag-TiCN Coatings**, *S. Ribeiro, N. Jordão,* Universidade do Minho, Portugal, *A.P. Piedade,* Grupo de Materials, Portugal, *M. Henriques, R. Oliveira,* Universidade do Minho, Portugal, *R. Escobar-Galindo,* Instituto de Ciencia de Materiales de Madrid (ICMM -CSIC), Spain, ***S. Carvalho*** *(sandra.carvalho@fisica.uminho.pt)*, Universidade do Minho, Portugal

Application of coatings in the biomedical engineering field represents an attractive challenge. Implant failure is a huge problem for both the patient and the government, once it involves repeated surgeries and high costs. This failure can be attributed to excessive wear and wear debris and also to microbial infection. Toxic effects possibly caused by excess of metal ion release during corrosion process in the physiological environment, it is still not well known. For this purpose Ag-TiCN films were deposited by DC unbalanced reactive magnetron sputtering by changing the synthesis conditions. Through variation of composition different coatings have been attained. Rutherford backscattering (RBS) and Glow Discharge Optical Emission Spectroscopy (GDOES) were used in order to obtain the chemical composition of as-deposited coatings. The microstructure and phase composition of coatings were studied by means of X-ray diffraction (XRD). The aim of this work is to compare surfa ce chemical and morphological changes introduced by commonly used simulated physiological conditions during corrosion process. Surface analytical techniques such as Auger electron spectroscopy (AES) combined with Scanning electron microscopy (SEM) examinations will provide details about the chemical composition as well as morphology of the surface samples. Observations of metal ion release from different biomaterials will be achieved by atomic absorption. Particular attention is paid to possible surface chemical and morphological changes which may be decisive for their interactions with animal cells.

10:40am **TS3-2-9 Preparation of Plasma-Polymerized Para-xylene as an Alternative to Parylene Coating for Biomedical Surface Modification**, ***C.-M. Chou***, Feng Chia University and Taichung Veterans General Hospital, Taiwan, *K.-C. Hsieh,* Feng Chia University, Taiwan, R.O.C., *C.-J. Chung (cjchung@seed.net.tw),* Central Taiwan University of Science and Technology, Taiwan, *J.-L. He*, Feng Chia University, Taiwan

Parylene (or poly-para-xylene) coating known to be an effective barrier with good biocompatibility is however with some drawbacks, including expensive starting material, high thermal energy consumption for monomer generation, high vacuum requirement and low growth rate. In this study, low-cost para-xylene was used as the starting material to form plasma-polymerized films. A pulsed-dc plasma generator was used and the deposits were examined their microstructure, mechanical properties and biocompatibility.

Experimental results reveal that the coatings present an amorphous structure with their deposition rate ranging from 0.05 to 0.48 μm per hour, which depends on the pulse frequency (ω_p) of the input power and para-xylene flow rate ($f_{p\text{-}x}$). It tends to produce short-chain alkenes and alkanes in the deposited film obtained at low ω_p of the input power and high $f_{p\text{-}x}$, in contrast to the film presenting inorganic feature ob tained at high ω_p of the input power and low $f_{p\text{-}x}$. As for its mechanical properties, a pencil hardness of 7H-8H of the coated specimen is higher than that of parylene thin films, and a film adhesion graded as 4B determined by cross-cut test is similar to that of parylene thin films. Water contact angle is measured ranging from 60° to 85° and is associated with chemical nature of the film deposited at different ω_p of the input power and high $f_{p\text{-}x}$. Cell culture on plasma-polymerized para-xylene deposited specimens performs a higher cell count than the parylene deposited one. The plasma-polymerized para-xylene deposited at high ω_p of the input power and low $f_{p\text{-}x}$ exhibit a higher cell count than those on the bare glass. These quantitative indications as revealed above imply that the plasma-polymerized para-xylene can possibly be an alternative to parylene coating for surface modification of biomedical devices.

11:00am **TS3-2-10 Micro-arc Oxidation of β Titanium Alloy: Structural Characterization and Osteoblast Compatibility**, ***H.-T. Chen***, Feng Chia University; China Medical University Hospital, Taiwan, *C.-H. Hsiao, H.-Y. Lung,* Feng Chia University, Taiwan, *C.-J. Chung (cjchung@seed.net.tw),* Central Taiwan University of Science and Technology, R.O.C., *C.-H. Tang,* China Medical University, Taiwan, *K.-C. Chen, J.-L. He*, Feng Chia University, Taiwan

β titanium alloys have been aware of their importance for applications on bone implants recently. In this study, micro-arc oxidation (MAO) was used to modify the surface of β titanium alloy (Ti-13Cr-3Al-1Fe) using NaH_2PO_4 solution as electrolyte. The relationship of applied voltage and oxidizing time with microstructure of the oxidized layer were investigated. The cell adhesion, proliferation and alkaline phosphatase (ALP) activity of osteoblast were measured to reveal cell compatibility of the treated specimens.

Experimental results show that under an applied voltage of 420 V, the oxidized layer can be found to gradually increase layer thickness but decrease in number and diameter of the pores that exist in the oxidized layer as a function of oxidizing time. The anatase TiO_2 presents in the oxidized layer but eventually replaced by the amorphous phase by increasing oxidizing time. Under an oxidizing time for 60 min, layer thickness of the ox idized layer increases and the anatase TiO_2 exists but again amorphous phase appears with increasing applied voltage. Osteoblast compatibility test shows that bare β titanium alloy is superior to bare α titanium metal (CP-Ti, Grade II). The MAO treated β titanium alloy presents cell compatibility significantly better than the bare β titanium alloy. Ultimate cell compatibility can be achieved by an MAO treatment using an applied voltage 350 V for 60 min. This study may provide an alternative process of osteoblast cell compatible surface modification for β titanium alloy bone implants.

11:20am **TS3-2-11 Nanoindentation and Corrosion Studies of TiN/NiTi Thin Films for Biomedical Applications**, ***D. Kaur*** *(dkaurfph@iitr.ernet.in), A. Kumar*, Indian Institute of Technology Roorkee, India

Proper passivation to prevent surface layer degradation and nickel releasing into the environment has been considered crucial for the medical applications of NiTi based shape memory alloys (SMA). To apply NiTi surgical devices in the human body, the surface properties and corrosion resistance are important material characteristics. Therefore, a stable, biocompatible and corrosion resistant passive layer is required.The present study explored the deposition and effect of nanocrystalline TiN protective layer on NiTi thin films prepared by dc magnetron sputtering to improve the surface and mechanical properties of SMA thin films.The structural, electrical, and mechanical studies were performed on both uncoated and TiN coated NiTi films and the results were compared. The size and preferred orientation of grains in the TiN passivation layer were observed to have a significant influence on the properties of TiN/NiTi heterostructure. Nanoindentation studies were performed at t emperatures of 297K, 323K, and 380K to determine the hardness and reduced modulus. Topographical in-situ images taken on the surface of pure NiTi and TiN coated NiTi showed an improvement in surface roughness after passivation coating. The investigations showed that TiN (200)/NiTi films exhibit high hardness, high elastic modulus, and better corrosion resistance as compared to pure NiTi and TiN (111)/NiTi films. In addition the presence of TiN (200) improves the top surface quality of NiTi films while retaining the phase transformation effect.

Coatings for Use at High Temperature
Room: Royal Palm 1-3 - Session A1-2

Coatings to Resist High Temperature Oxidation
Moderator: Y. Zhang, Tennessee Technological University, K. Stiller, Chalmers University of Technology

1:30pm **A1-2-1 Compositional, Geometrical and Manufacturing Related Parameters Affecting the Oxidation Resistance of MCrAlY-Coatings**, ***D. Naumenko*** *(d.naumenko@fz-juelich.de), J. Toscano, M. Subanovic, L. Singheiser, W.J. Quadakkers*, Forschungszentrum Jülich GmbH, Germany **INVITED**

MCrAlY (M = Ni,Co) overlay coatings and bondcoats (BC) in combination with thermal barrier coatings (TBC) are commonly used to protect gas-turbine components from high-temperature oxidation and corrosion. The oxidation behavior of MCrAlY coatings is, therefore, a crucial factor for the component lifetime. In the present paper a number of factors affecting the oxidation resistance of state of the art MCrAlY-coatings are reviewed.

It is shown that the scale formation on the MCrAlY-coatings and TBC-lifetime are affected by the coating major chemical composition, i.e. the Co/Cr/Al-contents. Furthermore, the composition of the formed oxide scale is influenced by the coating geometrical parameters, i.e. surface roughness, profile and thickness. These parameters affect the depletion of elements, such as Y and Al in the coating sub-surface regions resulting in formation of inhomogeneous oxide scales underneath rough, as-sprayed MCrAlY-surfaces. Moreover, the coating thickness not only determines the reservoir of the scale forming element Al, but also that of Y. It is shown that not only the Y-content but also the Y-reservoir has a significant effect on the growth rate and adherence of the alumina scale.

Finally, the effects of various processing parameters on the oxidation resistance of MCrAlY overlay coatings and bondcoats are considered. These parameters include vacuum quality during plasma spraying, temperature regime of the vacuum heat-treatment as well as smoothening treatment prior to electron-beam physical vapor deposition (EB-PVD) of the TBC. High temperature and high vacuum quality during heat-treatment promote selective oxidation of reactive elements (RE) such as Y at the coating surface. This results in RE-depletion from the coating and favors accelerated scale growth and high growth stresses during subsequent oxidation exposure. It is shown that minor variations in processing parameters can result in significant variations in the oxidation behavior and lifetime of nominally the same MCrAlY-coatings and bondcoats.

2:10pm **A1-2-3 Microstructure and Oxidation Resistance of Nanocrystalline 304SS-Al Coatings**, ***N.S. Cheruvu*** *(sastry.cheruvu@swri.org), R. Wei,* Southwest Research Institute*, R. Govindaraju,* Karta Technologies*, D.W. Gandy*, Electric Power Research Institute

The long-term oxidation behavior of nanocrystalline Fe18Cr8NiAl@x@ (where x = 0 to 10) coatings has been investigated. The coatings were deposited on 304SS samples by sputtering a 304 stainless steel target and an Al target using two magnetrons. Cyclic oxidation tests were conducted on the coated and un-coated samples at a peak temperature of 750@o@C for up to 1000 one-hour thermal cycles between peak and room temperatures. Optical, transmission and scanning electron microscopy and x-ray diffraction have been used to assess the microstructure, chemical composition of the coating and the oxide scale on the exposed samples. The crystal structure of the coatings, irrespective of Al content, in the as-deposited condition was found to be nanocrystalline BCC. The microstructure of the as-deposited Fe18Cr8Ni coating showed the presence of sigma phase. The addition of Al to the Fe18Cr8Ni coating stabilized the BCC structure and prevented the formation of the sigma phase. The re sults showed that the external scale (Cr@2@O@3@ on Fe18Cr8Ni and A@2@O@3@ aluminum containing coatings) formed during cyclic oxidation testing on the nano crystalline coated samples exhibited good spallation resistance compared to the scale on the un-coated samples. The addition of Al to the coating further increased the oxide spallation resistance. After exposure to approximately 1000 thermal cycles, the coating containing 10% Al was in good condition showed no evidence of internal oxidation. Inward diffusion of Al into the substrate during thermal cycling resulted in precipitation of iron-aluminde particles in the interdiffusion zone. The interdiffusion zone width increased with increasing Al content in the coating. Due to inward and outward diffusion of Al during thermal cycling, the Al content in the coating dropped from 10.5 to 3.7 wt.%. The improvement in oxide scale spallation resistance and accelerated depletion of aluminum are believed to be related to the fine grai n structure of the coating.

2:30pm **A1-2-4 High Temperature Resistant Magnetron Sputtered CrAlN and CrAlN:Y Coatings**, ***S. Vezzu*** *(vezzu@civen.org), A. Patelli, L. Zottarel, M. Colasuonno, S. Costacurta*, Associazione CIVEN, Italy

Coating of tools and machine parts is becoming a routine process due to the increasing performances required in terms of surface hardness, wear resistance, chemical and thermal stability. Furthermore, the high temperature reached in several industrial processes, such as aluminium and glass die casting, involves preservation of high hardness and chemical inertness at temperatures higher than 900°C. On this field, CrAlN and CrAlYN are promising candidates in high temperature applications owing to their properties in terms of oxidation resistance at elevated temperatures and high hardness. In fact, the formation of a thin aluminium oxide surface layer allows the chemical and mechanical stability of those coatings. In this study, CrAlN and CrAlYN coatings have been deposited via closed field unbalanced reactive magnetron sputtering on HSS substrates starting from Cr, Al and Cr:Y targets in argon-nitrogen atmosphere. The influence of different Al/Cr ratio on microstr ucture, mechanical properties and structure has been investigated. Thermal annealing at 900°C and 1000°C in air furnace has been performed; the oxidation processes have been studied by GDOES depth profiling while the thermal evolution of coating structure has been determined by XRD analysis. The hardness and Young's modulus of coatings have been determined by nanoindentation from room temperature up to 500°C using a special hot-stage nanoindentation module. Standard ball on disc test with AISI440C 10mm diameter sphere has been used to evaluate the friction coefficient and the specific wear rate in the same temperature range to verify the thermal stability of the mechanical properties. Finally, adhesion has been evaluated by microscratch test. The effects of addition of a small amount of Y in coating composition and the behaviour of mechanical properties versus Al/Cr ratio have been discussed in order to individuate a temperature range of applicabilit y for those coatings.

2:50pm **A1-2-5 Oxidation Resistance of a Zr-Doped NiAl Bondcoat Thermochemically Deposited on a Nickel-Based Superalloy**, ***S. Hamadi,*** *M.P. Bacos, M. Poulain (martine.poulain@onera.fr),* ONERA, France*, V. Maurice, P. Marcus*, ENSCP/CNRS, France

Turbine blades are exposed to oxidation at high temperatures (>1100°C) and are thus protected by coatings such as nickel aluminide. In industry, Pt-modified or unmodified NiAl are used, but investigations are carried out to replace them. The beneficial effects of adding reactive elements to NiAl in order to improve thermally grown alumina adhesion on top of NiAl are highlighted in the literature. Our study is based on a coating developed by Onera and Snecma that includes Zr in NiAl by a vapor phase thermochemical co-deposition of Al and Zr on a nickel-based superalloy. In previous experiments (SEM, spectral maps, GDMS), we pointed out that our aluminizing process led to a β-NiAl coating with 300 at. ppm of Zr located at the interface between the superalloy substrate and the coating layer. However, as soon as it was annealed or short-time oxidized, Zr migrated through the coating to the interface oxide/NiAl mostly as a metal (ToF-SIMS, XPS). In this study, we focus our effort on the oxidation resistance of this system. It appeared that doping NiAl with hundreds at. ppm of Zr highly increased the lifetime of the system under cyclic oxidation (1h 1100°C cycles under ambient air). Promising results were obtained when compared to AM1/(Ni,Pt)Al system. We investigated the influence of Zr on the morphology of the alumina developed and on the oxide/NiAl interface. Furthermore, the coating evolution during these oxidation tests was periodically characterized. Isothermal oxidation tests in a thermobalance were performed and revealed that Zr, initially present deep within the coating, played a role during the transient oxidation stages and lowered the parabolic constant of α-alumina in the steady-state stage.

3:10pm **A1-2-6 Reactive Element-Modified Aluminide and Platinum Aluminide Coatings on Ni-Base Superalloys**, ***M.S. Priest*** *(mspriest21@gmail.com), Y. Zhang,* Tennessee Technological University*, B.A. Pint, J.A. Haynes,* Oak Ridge National Laboratory*, B.T. Hazel, B.A. Nagaraj*, GE Aircraft Engines

A pack cementation process was developed for synthesizing reactive element (RE)-doped aluminide and platinum aluminide coatings on Ni-base superalloys. Three RE dopants, including Hf, Zr and Y, were investigated in this study by incorporating various RE-containing sources in the pack mixtures. All coatings were deposited by utilizing a non-contact arrangement where the substrates were physically separated from the powder mixture, which produced clean coating surfaces comparable to that made by chemical vapor deposition (CVD) or above-the-pack. The effect of

aluminizing parameters, particularly the amount of RE-containing powder in the pack, was studied. Different responses of the three dopants in the aluminizing process and the resultant coating microstructures were discussed. The cyclic oxidation behavior of RE-doped aluminide coatings was investigated at 1150°C in air, and compared with RE-free aluminide coatings.

3:30pm **A1-2-7 NiW Diffusion Barrier Influence on the Oxidation Behaviour of β-(Ni,Pt)Al Coated Fourth Generation Ni-Base Superalloy**, ***E. Cavaletti*** *(eric.cavaletti@onera.fr), S. Naveos, S. Mercier, P. Josso, M.P. Bacos,* ONERA, France*, D. Monceau*, ENSIACET-INPT, France

For long-term life at high temperature of gas turbine blades, interdiffusion between the Ni-base superalloy and its protective coating degrades the complete system. Firstly, diffusion of some substrate alloying elements into the coating damages the protective oxide scale adhesion. Secondly, oxidation and diffusion cause aluminium depletion in the coating which leads to phase transformations and affects the lifetime of the system. Finally, in Re-rich Ni-base superalloys coated with β-(Ni,Pt)Al, discontinuous precipitation leads to the formation of detrimental Secondary Reaction Zones for which interdiffusion is supposed to be a driving force. To limit interdiffusion phenomenon, diffusion barriers (DB) have been developed. Fabrication of DB layers based only on refractory metals is complex and often requires a Ni deposition before aluminisation. To simplify the process, a DB layer based on a Ni-W electrolytic coating was developed to limit interdiffusion between a Ni -base fourth generation superalloy (MCNG) and a β-NiAl coating. The DB layer is created thanks to the precipitation of α-W phase that forms definite compounds with Re from the alloy during high temperature exposure. In this paper, the Ni-W DB was applied on a (Ni,Pt)Al coated MCNG alloy. The cyclic and isotherm kinetics were determined at 1100°C in air. The structures and composition changes of both coating and superalloy were measured with image analysis, X-ray diffraction, SEM observation and EDS analysis. It was found that the DB acts at the onset of oxidation on the oxide formation and on the β to γ' transformation kinetics, e.g. after 300 h oxidation at 1100°C the γ' fraction is reduced from 70 to 40%.

3:50pm **A1-2-8 Formation and Oxidation Performance of Low-Temperature Pack Aluminide Coatings on Ferritic-Martensitic Steels**, ***Y. Zhang*** *(yzhang@tntech.edu), B. Bates,* Tennessee Technological University*, B.A. Pint*, Oak Ridge National Laboratory

A pack cementation process was developed to coat commercial 9% Cr ferritic-martensitic steel P91 at temperatures below its tempering temperature to avoid any potential detrimental effect on the mechanical properties of the coated alloy. To prevent the formation of Al-rich intermetallic phases such as Fe_2Al_5 in the coating, the Al activity in the pack cementation process was reduced by substituting the pure Al masteralloy with Cr-Al binary masteralloys containing 25 and 15 wt.% Al. When the Cr-25Al masteralloy was used, a duplex coating was formed at 700ºC, consisting of a thin Fe_2Al_5 outer layer and an inner layer of FeAl. With the Cr-15Al masteralloy, the Fe_2Al_5 phase was eliminated and an FeAl coating of ~12 µm thick was achieved. In addition, an effort was made to combine the coating process with the standard heat treatment for ferritic-martensitic alloys, e.g., 2h austenization at 1050°C and 2h tempering at 750°C. The coatings fabricated at 700°C are being tested in air + 10% H_2O at 650°C to evaluate their long-term oxidation performance. Aluminide coatings synthesized at 1050°C via pack cementation or chemical vapor deposition (CVD) are included in the test for comparison.

4:10pm **A1-2-9 Pt Modified Nickel Aluminides, MCrAlY-Base Multilayer Coatings and TBC Systems Fabricated by Sparks Plasma Sintering (SPS) for the Protection of Nickel Base Superalloys**, ***D. Monceau*** *(daniel.monceau@ensiacet.fr),* ENSIACET-INPT, France*, D. Oquab,* CIRIMAT Laboratory - Toulouse, France*, C. Estournes,* CIRIMAT and PNF2 CNRS - Toulouse, France*, M. Boidot,* CIRIMAT Laboratory - Toulouse, France*, Y. Cadoret*, SNECMA, France **INVITED**

Pt-modified Ni aluminides and MCrAlY coatings (where M = Co, Ni or Co/Ni) are widely used on turbine blades and vanes for protection against oxidation and corrosion and as bondcoatings in thermal barrier coatings systems. The present work shows the ability of a new fabrication technique, the Spark Plasma Sintering, to develop rapidly new coating compositions and microstructures. This technique allows combining powders and metallic foils on a superalloy substrate in order to obtain multilayered coatings in a single short experiment. Fabrication of MCrAlY overlays with local Pt and/or Al enrichment will be shown, as well as fabrication of coatings made of PtAl@sub 2@, PtAl, @alpha@beta@-AlNiPt@sub 2@, martensitic @beta@-NiPtAl or Pt-rich γ/γ' phases. The realization of an entire TBC system with a porous adherent YSZ layer on a γ/γ' low mass bond-coating will be demonstrated. Difficulties of this technique will be reviewed and discussed, such as unexpect ed segregations, risks of carburization, of local over-heating, or difficulty to coat complex shape parts. Finally, some first results of cyclic oxidation will be given including a comparison with industry made coatings and systems.

4:50pm **A1-2-11 Role of Al Oxide PVD Coatings in the Protection Against Metal Dusting**, ***J. Alvarez,*** ITESM-CCV*, B. Bautista, D. Melo,* IPN, Mexico*, O. Salas,* ITESM-CEM, Mexico*, R. Reichelt,* Wilhelms-Universitaet, Germany*, J. Oseguera (joseguer@itesm.mx),* ITESM-CEM, Mexico*, . López*, IPN, Mexico

The presence of some surface oxides in materials exposed to metal dusting have been proven to be an effective method to prevent this type of corrosion due to the very low diffusivity of carbon in oxides. However, the surface oxide films have to be dense and adhere well to the component to be protected. Reactive magnetron sputtering (RMS) is a promising method to produce these type of oxide layers due to its flexibility in terms of process parameters and resulting structures. In the present work, Al oxide/Al layers have been deposited by RMS on HK40 substrates under various conditions in order to develop the most adequate structure for protection against metal dusting. Some coated substrates were subjected to metal dusting conditions in a thermobalance. The microstructure of the coatings before and after metal dusting was characterized by scanning electron microscopy, atomic force microscopy and x-ray diffraction.

5:10pm **A1-2-12 Observation of High-Temperature Phase Transformation in the Si-Modified Aluminide Coating on Mild Steel Using EBSD**, ***W.-J. Cheng*** *(d9603505@mail.ntust.edu.tw), C.-J. Wang*, National Taiwan University of Science and Technology, Taiwan

Mild steel was coated by hot-dipping into a molten bath containing Al-10 wt.% Si. The phase transformation in the aluminide layer during diffusion at 750°C in static air was analyzed by Electron Backscatter Diffraction (EBSD). The results showed that the aluminide layer of the as-coated specimen consisted of an outer Al-Si eutectic topcoat and the inner Fe-Al-Si and Fe-Al intermetallic layers. The formation of τ_5-Al_7Fe_2Si and τ_6-Al_4FeSi can be observed with increasing exposure time at 750 °C, while the τ_1-$(Al,Si)_5Fe_3$ phase precipitated in Fe_2Al_5. After 60 min of exposure, the τ_5-Al_7Fe_2Si and τ_6-Al_4FeSi phases disappeared. The FeAl phase not only formed at the interface between Fe_2Al_5 and the steel substrate, but also transformed from τ_1-$(Al,Si)_5Fe_3$ after diffusion for 10 h. With prolonged exposure, the growing FeAl phase decreased the thickness of Fe_2Al_5 and forced the formation of $FeAl_2$ phase. Finally, the aluminide layer composed of $FeAl_2$ and FeAl.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B5-2

Properties and Characterization of Hard Coatings and Surfaces

Moderator: Y.W. Chung, Northwestern University, G. Abadias, Universite de Poitiers-CNRS, M. Fenker, Forschungsinstitut Edelmetalle & Metallchemie

1:30pm **B5-2-1 Structure, Stability and Bonding of Ternary Transition Metal Nitrides**, ***P. Patsalas*** *(ppats@cc.uoi.gr), G.M. Matenoglou, L.E. Koutsokeras, Ch.E. Lekka,* University of Ioannina, Greece*, G. Abadias,* Universite de Poitiers-CNRS, France*, C. Kosmidis, G.A. Evangelakis*, University of Ioannina, Greece

Nitrides of the group IVb-VIb transition metals have been well known for their unique combination of exceptional mechanical and electrical properties sharing high hardness values and electrical conductivity. Ternary transition metal nitrides have lately gained special attention (with TixZr1-xN being the most widely studied) in an effort to improve further these properties. In this work we present a comparative study of a very wide range of ternary transition metal nitrides of the form: TixMe1-xN (Me=Zr,Hf,Nb,Ta,Mo,W) and TaxMe1-xN (Me=Zr,Hf,Nb,Mo,W) over the whole x range (0<x<1) grown by Pulsed Laser Deposition (PLD) and by Dual Ion Beam Sputtering (in the case of TixTa1-xN only). We study the stability of the rocksalt structure in all these films using X-Ray Diffraction and Reflectivity (XRD/XRR), in-situ Auger Electron Spectroscopy (AES) and ab-initio calculations and we map the lattice constants. We consider the electron hybridizations and the bond nature of the ter nary nitrides based on the ab-initio calculation results. We investigate the validity of Vegard's rule and the effect of growth-dependent stresses to the lattice constant. We also perform a critical comparison of these ternary nitrides with the corresponding binary nitrides MeN (Me=Ti,Zr,Hf,Nb,Ta,Mo,W) grown by similar conditions.

1:50pm **B5-2-2 Ab-Initio Calculations of the Effect of Si and C on Elastic Properties of Metal Nitrides**, ***J. Houska*** *(jiri.houska@polymtl.ca), J.E. Klemberg-Sapieha, L. Martinu*, Ecole Polytechnique de Montreal, Canada

In this contribution, we report extensive ab-initio calculations of elastic properties of Ti(Si)N(C) and Cr(Si)N(C) materials. Atom cores and inner electron shells are described by Vanderbilt-type pseudopotentials, and Kohn-Sham equations for valence electrons are expanded using a wave-basis set. In fcc metal nitride networks, up to 1/8 of metal atoms are replaced by Si, and up to 1/4 of N atoms by C.

For various material compositions, bulk modulus (B) and lattice constant (a_0) is calculated by fitting the Birch equation on total energies calculated at different lattice parameters (94-106 % of the lowest energy value), and elastic tensor (C_{ij}) is derived using energy changes resulting from small volume-preserving strains and shears (up to 6% and 6 deg., respectively). Young's modulus (E), Poisson's ratio (v) and shear modulus (G) are calculated from C_{ij}. We examine dependence of density of unpaired spins on material composition and on the strains applied. We find that small Si or C contents in materials lead to decreasing G, increasing E_{100}, decreasing v and slightly increasing a_0, and calculate slopes of the changes. Furthermore, B decreases after addition of Si while there are no changes in B after addition of C. Dependence of electrical conductivity on material composition is examined by calculating electronic density of states.

The results, compared with experimental data and explained using the changes in valence electrons wavefunction, can be used to estimate properties of the previously deposited and simulated nanocomposite structures. The calculated lattice constants can be used to estimate compositions of crystals examined by x-ray diffraction.

2:10pm **B5-2-3 Growth Behavior and Microstructure of Arc Ion Plated Titanium Dioxide**, *C.-J. Chung (cjchung@seed.net.tw),* Central Taiwan University of Science and Technology, Taiwan, *H.-I. Lin, P-Y. Hsieh, K.-C. Chen,* ***J.-L. He***, Feng Chia University, Taiwan **INVITED**

Titanium dioxide (TiO_2) film has been prepared by physical vapor deposition (PVD) for a decade. It is considered for applications such as photocatalysis, self-cleaning and antimicrobial purposes. However, numerous questions regarding their growth behavior and microstructure remain unanswered. Arc ion plating (AIP), a popular PVD technique with a high ionization rate, was adopted in this work to reveal growth behavior by microstructural characterization and plasma diagnostics.

Experimental results show that TiO_2 film deposition can be strongly affected by oxygen partial pressure during deposition and deposition time. A film that is produced under oxygen-deficient conditions feature an amorphous matrix that is mixed with rutile crystallites. However, under oxygen-rich conditions, the initially grown TiO_2 film is found to be composed of rutile crystallites. Thermodynamic calculations indicate that the growth of the rutile phase is favored in al l PVD processes; however, the anatase phase appears is the major phase in the later stage of growth, and may be governed by a kinetic factor. The optical emission spectrum of plasma within the deposition chamber is therefore examined by optical emission spectrometry (OES). The results demonstrate that increasing the oxygen partial pressure or the deposition time tends to promote oxidation over the surface of the titanium cathode such that neutral titanium species that are emitted from the titanium cathode prevail over the ionized titanium species. The neutral titanium species, with less kinetic energy, are consequently unable to reach the activation energy of rutile formation and, instead, a metastable anatase phase is formed. Based on this finding, a model of AIP-TiO_2 growth that is based on an energy viewpoint is proposed, to help control the microstructure of PVD TiO_2 films.

2:50pm **B5-2-5 Structure-Property Relations of arc-Evaporated $Al_xNb_{1-x}N$ Hard Coatings**, ***R. Franz*** *(robert.franz@unileoben.ac.at),* University of Leoben, Austria, *M. Lechthaler,* OC oerlikon Balzers AG, Liechtenstein, *C. Polzer,* PLANSEE Composite Materials GmbH, Austria, *C. Mitterer*, University of Leoben, Austria

Ternary MeAlN hard coatings, especially TiAlN and AlCrN, have been intensively studied during the past 20 years. Due to the incorporation of Al into the crystal lattice, superior mechanical properties and improved oxidation resistance were obtained. Similar effects can be expected by alloying NbN with Al. However, the Nb-N system is of higher complexity as compared to Ti-N and Cr-N since several different phases can be formed. The aim of the present work was to synthesise $Al_xNb_{1-x}N$ coatings with the face-centered cubic structure of δ-NbN and to investigate the influence of the Al content on the crystal structure evolution as well as the mechanical properties and the oxidation resistance. All coatings were deposited in an Oerlikon Balzers arc-evaporation system using powder-metallurgically prepared AlNb targets with different Al to Nb ratios. The target composition is roughly mirrored in the metal ratio of the coatings as it was obtained by energy-dispe rsive X-ray spectroscopy. With the aid of X-ray diffraction analyses, the formation of the face-centered cubic structure could be evidenced. Only at very high Al contents hexagonal phases are formed as well. The mechanical properties were assessed by nanoindentation and biaxial stress measurements. Annealing experiments up to 1000°C in ambient air revealed an onset temperature for oxidation in the range of 700-800°C for the coatings with a low Al content. The increase of the Al content is beneficial in terms of oxidation resistance as it leads to higher onset temperatures. In summary, incorporating Al into NbN improves the coating properties and the presented $Al_xNb_{1-x}N$ hard coating family seems to be promising for industrial applications.

3:10pm **B5-2-6 Why a TiN Coating is not Suitable for Corrosion Protection of Steel**, *J.P.M. Groenewegen, R. Machunze,* ***G.C.A.M. Janssen*** *(g.c.a.m.janssen@tudelft.nl)*, TU Delft, Netherlands

Titanium nitride (TiN) is a hard coating well known for its excellent hardness and wear protective properties. Many studies have found that TiN is hardly useful for corrosion protection of steel. In order to find the cause for this absence of corrosion protection we did measurements on TiN coatings on steel and on glass.

We prepared two series of samples, TiN on glass, and TiN on steel. Thin TiN films (18 - 2000 nm) are deposited by reactive unbalanced magnetron sputtering on glass slides and hot working tool steel substrates, using an industrial setup. During deposition (dep. rate approx. 0.1nm/s) at 5kW power, 125V bias voltage, 450°C dep. temperature and $4x10^{-3}$mbar pressure, the samples performed a planetary motion in front of the Ti target (target size 600x120mm^2).

We first measured the open potential of TiN on glass to compare the corrosion properties of TiN with those of tool steel. From this measurement we conclude that TiN provides anodic protection, i.e. through coverage only. The corrosion current of TiN on glass was found to be orders of magnitude lower than that of steel. Therefore it is likely hat corrosion of the steel starts at defects in the coating.

The coatings showed defects, some related to the deposition process, some related to the substrate. Corrosion experiments, performed while observing the coating through an optical microscope, indeed showed that corrosion of the steel starts at defects in the coating.

Microscopy on the substrates before deposition showed some porosity in the surface of the polished steel samples. It was found that these defects in the steel substrate are not covered by a continuous TiN film during deposition. Therefore, even if the deposition related defects are eliminated, TiN is not a suitable coating for corrosion protection due to the porosity of the steel.

3:30pm **B5-2-7 Carbon Occupancy of Interstitial Sites in Vanadium Carbide Films Deposited by Reactive Magnetron Sputtering**, *E. Portolan, C.L.G. Amorim, G.V. Soares, C. Aguzzoli, C.A. Perottoni, I.J.R. Baumvol,* ***C.A. Figueroa*** *(cafiguer@ucs.br)*, Universidade de Caxias do Sul, Brazil

Hard coatings are widely used in surface engineering for wear protection and friction reduction of mechanical components. Numerous different coatings are currently available for specific applications. Among these coatings, vanadium carbide (VC) thin films show high hardness, high melting point, and good chemical stability. However, the crystalline structure of VC thin films is still open to investigation. On one hand, stoichiometric VC is an interstitial compound, where vanadium forms the principal lattice and carbon occupies the octahedral interstitial sites[1]. On the other hand, the equilibrium phase diagram of the V-C system is complex and many phases with different stoichiometries coexist[2].Although order-disorder phase transformations were already observed, the migration of carbon atoms associated with these transformations is still unclear[3]. In this work, vanadium carbide thin films were deposited on Si substrates by reactive magnetron sputtering f rom a V target in a Ar/CH_4 plasma, varying the Ar/CH_4 partial pressure ratio and substrate temperature. The films were characterized by glancing angle X-ray diffraction and Rutherford backscattering spectrometry. Better defined crystalline structures were obtained at higher CH_4 content than 13%. The increase of substrate temperature diminishes slightly the film thickness and substantially the C/V atomic ratio. The intensity ratio of the Bragg peaks (111)/(200) decreases as a function of substrate temperature. The results are discussed in terms of a proposed mechanism for interstitial diffusion of carbon atoms in vanadium carbide thin films with fcc-like crystalline structure and the carbon occupancy of tetrahedral or octahedral interstitial sites as a function of substrate temperature.

[1]V. N. Lipatnikov, A. I. Gusev, P. Ettmayer, and W. Lengauer, J. Phys.: Condens. Matter 11, (1999) 163. @paragraph2@[2]J. Hu, C. Li, F. Wang, and W. Zhang, J. Alloys Compounds 421, 120 (2006).

[3]V.N. Lipatnikov, W. Lengauer, P. Ettmayer, E. Keil, G. Groboth, and E. Kny, J. Alloy and Comp. 261, 192 (1997).

3:50pm **B5-2-8 Development of B4C Sputter-Deposited Coatings for Mechanical Applications**, *C. Patacas,* ***C. Louro*** *(cristina.louro@dem.uc.pt), J.C. Oliveira, A. Cavaleiro*, University of Coimbra, Portugal

Boron carbide (B4C) is one of the hardest materials and has the highest known dynamic elasticity. These properties, along with its high melting point, low mass density, high wear resistance and high thermal stability, explain the great interest in depositing this material as thin protective coatings. Boron carbide has a complex structure consisting of icosahedral arrangements of twelve atoms at the vertices of a rhombohedral unit cell connected with three-atom chains along the main diagonal. This structure is stable within a large carbon content range, extending from 8.8 to 22 at.% C, mainly due to the atomic similarity of boron and carbon atoms. Within the phase homogeneity range, B4C is believed to form several polytypes at room temperature such as B12(CCC), which consists of 12 boron atom icosaedras and 3 carbon atom chains, or B11C(CBC). Several studies have shown that different polytypes most likely coexist at the same chemical composition. Very recently, it was a lso shown that the mechanical properties of boron carbide can be improved by doping with silicon, mainly by the suppression of the B12(CCC) polytype. In this study, B-C and B-C:Si thin films were deposited by r.f. magnetron sputtering from a hot-pressed B4C polycrystalline target. In the first step of the work, simple B-C coatings were optimized by varying the bias, target power and coil current. After, the coatings were incorporated with Si contents up to 7 at.%. The EPMA analysis revel that with the exception of the unbiased film (high C/B ratio), all the other coatings were overstoichiometric in relation to carbon (~22 at.% C). The films exhibited poor crystallinity with only a broad XRD peak close to the main reflection of the boron carbide. Thus, in order to achieve long-range order, the coatings were thermally annealed in a protective atmosphere (Ar+5% H2) up to 1200ºC. The results concerning structure, chemical composition and hardness will be presented as a function of the annealing temperature as well as the Si content. Further results obtained by Raman, FT-IR spectroscopy and TEM analysis will be also presented for understanding both compositional and thermal effects on boron carbide-based coatings.

4:10pm **B5-2-9 Influence of the Chemical Composition, Electronic Structure and Phase Evolution on the Electric and Optical Behaviors of Decorative Zirconium Oxynitride Thin Films**, ***F. Vaz*** *(fvaz@fisica.uminho.pt), P. Carvalho, L. Cunha,* Minho University, Portugal*, N. Martin,* Institut FEMTO-ST, France*, J.P. Espinós, A.R. González-Elipe*, Instituto de Ciencia de Materiales de Sevilla (CSIC-Univ. Sevilla), Spain **INVITED**

This work is devoted to the investigation of decorative ZrO_xN_y films. Film's color changed from metallic-like to very bright yellow-pale and golden yellow, for low oxygen contents to red-brownish for intermediate oxygen amounts. Associated to this color change, there is a significant decrease of brightness. With further increase of the oxygen content, the color of the films changed from red-brownish to dark blue. The films deposited with high oxygen amounts showed only apparent colorations due to interference effects. This change in optical behavior from opaque to transparent (transition from metallic to insulating materials), revealed that significant changes were occurring in the films structure and electronic properties. In fact, the variations in composition disclosed the existence of four different types of films, which were also correlated to the structural features. For the so-called zone I, XRD revealed the development of films with a B1 NaCl face-ce ntered cubic zirconium nitride-type phase. Increasing the reactive gas flow, the structure of the films changed to an over-stoichiometric nitride phase, similar to that of Zr_3N_4, but with some probable oxygen inclusions within nitrogen positions. This region was characterized as zone II. Zone III was indexed as an oxynitride-type phase, similar to that of γ-Zr_2ON_2 with some oxygen atoms occupying some of the nitrogen positions. Finally, occurring at the highest oxygen amounts, zone IV was assigned to a ZrO_2 monoclinic type structure. The composition/structure variations were consistent with the chemical bonding analysis carried out by XPS, which showed a continuous decrease of density of states near the Fermi level associated to an increase of transference of d electrons to the p band. The electronic properties of the films exhibited significant changes from zone to zone. Resistivity measurements revealed a very wide range of values, varyin g from relatively high conductive materials (for zone I) with resistivity values around few hundreds of $\mu\Omega$cm, to highly insulating films within zones III and IV, which presented resistivity values in the order of 10^{15} $\mu\Omega$cm. Regarding zone II, corresponding to oxygen doped Zr_3N_4-type compounds, the observed behavior revealed resistivity values increasing steeply from about 10^3 up to 10^{15} $\mu\Omega$cm, indicating a systematic transition from metallic to insulating.

4:50pm **B5-2-11 Sub-Microstructure and Grain Texture of Reactive Unbalanced Magnetron Sputtered Titanium and Titanium Compound Thin Films**, ***S.D. Carpenter*** *(s.carpenter@mmu.ac.uk),* Manchester Matropolitan University, United Kingdom*, P. Kelly*, Manchester Metropolitan University, United Kingdom

Physical vapour deposition techniques (PVD) are non equilibrium processes that often produce non equilibrium microstructures. Nucleation occurs at many sites in close proximity over the substrate surface and is similar to electro deposition within an electrolyte in this respect. Thin film growth advances from the substrate to form a dense columnar structure with a growth direction parallel to the flux of incident energetic particles. Growth is usually along one of the preferred close packed crystal directions and results in a high degree of crystallographic texture within the deposit. Titanium was sputtered with varying nitrogen flow rates to vary the film composition from Ti to stoichiometric TiN. The thin films were grown at different DC pulse frequencies ranging between 100 kHz to 350 KHz using an Advanced Energy Pinnacle Plus power supply. For comparison purposes, films were also grown in DC mode. The substrates were RF biased using an Advanced Energy RFX-600 supply. Electron microscopy revealed that TiN and titanium metal grow as individual crystallites within sub stoichiometric TiN. Furthermore the growth direction of these crystallites was found to be influenced by deposition conditions. It was also found that the growth direction alters radically for cylindrical substrates rotated in front of a titanium cathode target. The results indicate that growth occurs by a ledge mechanism for rotated substrate and by a spiral growth mechanism for fixed substrates.

5:10pm **B5-2-12 Effect of Substrate Positioning for TiAlN Films Deposited by an Inverted Cylindrical Magnetron Sputtering System**, ***H.H. Abu-Safe*** *(habusaf@uark.edu),* Lebanese American University, Lebanon*, M.H. Gordon*, University of Arkansas

TiAlN films up to 300 nm thick were deposited on glass substrates at three different positions within an inverted cylindrical magnetron sputtering system. Titanium and aluminum targets were used and one substrate was located near the titanium target, another near the aluminum target, and third centered between the targets. In addition at each location, substrates were positioned at three different angles (0, 45, and 90). The sputtering power during deposition was maintained at 2 kW with constant Argon and nitrogen gas flow rates. The Films chemical and elemental composition at different elevations and tilts with respect to the targets was analyzed using Energy dispersive X-Ray diffraction. A preferred 200 crystal orientation was noticed in all samples. However, the peak intensity at this orientation varied with substrate tilt. This indicates the effect of surface exposure to the confining magnetic field on the arrangements of atoms during deposition. The scanning electr on microscope images showed variation surface textures with position and tilt for all samples. A model of the deposition mechanism at different position inside the sputtering chamber is presented.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D1

Boron Nitride and Carbon Nitride and Group-III (Al, Ga, In) Nitride Materials

Moderator: A.B. Djurišić, The University of Hong Kong, M. Keunecke, Fraunhofer Institute for Surface Engineering and Thin Films

1:30pm **D1-1 Cubic Boron Nitride Based Metastable Coatings and Nanocomposites**, ***J. Ye*** *(jian.ye@imf.fzk.de), S. Ulrich, M. Stüber, C. Ziebert*, Forschungszentrum Karlsruhe, Germany **INVITED**

Various PVD and plasma-assisted CVD methods presently used for the deposition of cubic boron nitride (c-BN) thin films demand adequate conditions relating to ion bombardment of growing films, growth temperature, film stoichiometry etc. The ion-bombardment conditions, often appearing rather apparatus-specific, can be however well categorized according to the fundamental parameters of bombarding ions as well as condensing neutral particles, namely their energy and flux density. In terms of these fundamental parameters, the mechanisms for the c-BN formation are briefly discussed, along with an evaluation of the available growth models. Due to intensive ion bombardment during deposition, c-BN films are known for their extremely high compressive stress and poor adhesion. The present study focuses then on the magnetron-sputtered, c-BN-based metastable films and nanocomposite films. Under suitable and well-controlled growth parameters, the deposited films exhibit microstructur es dominated by c-BN phase, while on the other side show considerably

reduced internal stresses in comparison to conventional c-BN films. Some examples will be shown, particularly c-BN/a-C nanocomposite and c-BN:O metastable films, including their deposition details, structure and composition characterization (HRTEM, SEM, XRD, XPS, AES, FTIR, etc.), and corresponding mechanical properties (hardness, E-modulus, stress). A film design concept will also be demonstrated as well as its successful realization for well-adherent films consisting of an adhesion-promoting base layer, a graded cubic-phase nucleation, and a low-stress, superhard, above 2 μm thick, c-BN-dominated top-layer.

2:10pm **D1-3 Machining of High Performance Workpiece Materials with cBN Coated Cutting Tools**, *E. Uhlmann,* ***J.A. Oyanedel Fuentes*** *(fuentes@iwf.tu-berlin.de),* Institute for Machine Tools and Factory Management, Germany, *M. Keunecke*, Fraunhofer Institute for Surface Engineering and Thin Films, Germany

The machining of high performance workpiece materials requires significantly harder cutting materials. In hard machining, the early tool wear occurs due to high process forces and temperatures. The hardest known material is the diamond, but steel materials cannot be machined with diamond tools because of the reactivity of iron with carbon. Cubic boron nitride (cBN) is the second hardest of all known materials. The supply of such PcBN indexable inserts, which are only geometrically simple available, requires several work procedures and is cost-intensive. The development of a cBN coating for cutting tools, combine the advantages of a thin film system and of cBN. Flexible cemented carbide tools, in respect to the geometry can be coated. The cBN films with a thickness of up to 2 μm on cemented carbide substrates show excellent mechanical and physical properties. This paper describes the results of the machining of various workpiece materials in turning and milling operatio ns regarding the tool life, resultant cutting force components and workpiece surface roughness. In turning tests of Inconel 718 and milling tests of chrome steel the high potential of cBN coatings for dry machining was proven. The results of the experiments were compared with common used tool coatings for the hard machining. Additionally, the wear mechanisms adhesion, abrasion, surface fatigue and tribooxidation were researched in model wear experiments.

2:30pm **D1-4 Magnetron Sputter Deposition of Low-Stress Cubic Boron Nitride Films using Ar-N_2-CH_4 Gas Mixtures**, ***S. Ulrich*** *(sven.ulrich@imf.fzk.de), J. Ye, M. Stüber, C. Ziebert*, Forschungszentrum Karlsruhe, Germany

Cubic boron nitride (c-BN) films produced by PVD and plasma-assisted CVD techniques typically exhibit undesired high compressive stresses. One of the effective and feasible methods to reduce stress and hence improve film adhesion has been a controlled addition of a third element into the film during deposition. In the present study, BN films were grown on to silicon substrates using reactive magnetron sputtering with a hexagonal BN target. An auxiliary flow of methane was mixed into argon and nitrogen as the working gas. The deposition was conducted at various methane flow rates at 400°C substrate temperature, 0.3 Pa total working pressure, and -250 V substrate bias. The structure and mechanical characteristics of the deposited films were then examined in dependence of the methane flow rate. With increasing methane flow rate from 0 to approx. 1.0 sccm, the fraction of the cubic BN phase in the deposited films decreased gradually down to approx. 70%, whereas the film stress was reduced much rapidly and almost linearly in relation to the methane flow rate. At 1.0 sccm methane, the stress became approx. 3 times reduced. Owing to the significantly decreased film stress, adherent, micrometer thick, c-BN dominant films can be allowed to form on silicon substrate. The microstructure and mechanical properties of the films will be illustrated through FTIR, SEM, AFM, nanoindentaion and so on.

2:50pm **D1-5 Texture Development and Microstructure Characterization of AlN Thin Films Fabricated by Pulsed Closed Field Unbalanced Magnetron Sputtering**, ***F. Wang*** *(fwang@mines.edu), A. Feldman, J. Lin, J.J. Moore, B. Mishra*, Colorado School of Mines

Aluminum nitride (AlN) thin films were synthesized by reactively sputtering Al metal target in pure nitrogen atmosphere using a pulsed closed field unbalanced magnetron sputtering system on different substrates with a thin Cr interlayer. The texture orientation and the microstructure of AlN films were characterized by means of X-ray diffraction and scanning electron microscopy. The mechanical properties of the coatings were studied using the nanoindentation. It was found that both the pulsing frequency and the working pressure significantly affected the (002) orientation in AlN films. Strong (002) orientation has been achieved in the AlN thin films under various pulsing conditions, which can be correlated to the high ion energies associated with the pulsed magnetron sputtering. In addition, the effects of the pulsing parameters and working pressures on the microstructure, crystalline size, and residual stress of the AlN films were investigated in detail.

3:10pm **D1-6 Theoretical and Spectroscopic Investigations on the Structure and Bonding in B-C-N Thin Films**, ***E. Bengu*** *(bengu@fen.bilkent.edu.tr), M.F. Genisel, O. Gulseren, R. Ovali*, Bilkent University, Turkey

In this study, we have synthesized boron, carbon, and nitrogen containing films through rf magnetron sputtering. We investigated the effects of deposition parameters on the chemical environment of B, C, and N atoms inside the films deposited. Techniques used for this purpose were GIR-FTIR, XPS, TEM and EELS. A hydrogen terminated surface was generated on the substrates before deposition. A 20nm Ti buffer-layer was deposited on the hydrogen terminated Si substrate just before the B-C-N films were deposited on the substrate. As for the boron and carbon source a 2 inch diameter B_4C target was used. During the deposition the pressure of the chamber was kept at 2.5 x 10^{-3} torrs during the deposition while the magnetron power on the B_4C target was adjusted to be 200 watts during period of deposition (~ 90 mins). A separate r.f. power supply attached to the substrate holder was used to apply and independently adjust the d.c. bias on the substrate between gr ound and -500 V. Meanwhile, gas flow into the reaction chamber was controlled by two mass flow controllers which helped manage the Ar to N_2 gas ratio for the deposition.

GIR-FTIR experiments on the B-C-N films deposited indicated presence of multiple features between 600 cm^{-1} to 1700 cm^{-1} range in the infrared spectra. Analysis of the IR spectra and the corresponding XPS data from the films has been done in a collective manner. The results from this study suggested under nitrogen rich synthesis conditions (higher N_2 ratio in Ar/N_2 mixture) carbon in the B-C-N films prefers to be surrounded by other carbon atoms rather than boron and/or nitrogen. Furthermore, we have observed a similar behavior in the chemistry of B-C-N films deposited with increasing substrate bias using again IR and XPS data together. In order to better understand these results, we have compared and evaluated the energetic for various nearest-neighbor and structur al configurations of carbon atoms on a single BN sheet environment using DFT calculations. These theoretical calculations also indicated that structures and configurations that increase the relative amount of C-C bonding with respect to C-B and/or C-N were energetically favorable than otherwise. As a conclusion, carbon tends to phase-segregate in to carbon clusters rather than displaying a homogeneous distribution in the films deposited in this study.

3:30pm **D1-7 Atomic and Electronic Structure of GaN Surfaces**, ***J.E. Northrup*** *(northrup@parc.com)*, Palo Alto Research Center **INVITED**

The InAlGaN materials system is employed in several important technologies including light emitting diodes, blue/green lasers, and electronic devices. Realization of the technological promise of the nitride materials system has required growth of high quality epitaxial material. This requirement has motivated many experimental and theoretical studies of GaN surfaces, with attempts to understand epitaxial growth in terms of the surface structure. Nitride surfaces exhibit considerable complexity in their atomic structure, the nature of which is dependent on the relative abundance of the group-III and group-V species and the crystallographic orientation of the surface. In addition surface states are the source of electrons in the two-dimensional electron gas at the AlGaN/GaN interface in high-electron mobility transistors, and this has motivated studies of surface electronic structure.

In this talk I will present the results of first-principles calculations employing pseudopotential density functional theory. The calculations provide insight into the atomic and electronic structure of nitride surfaces. Some emphasis will be placed on the group-III metallic adlayers which are stabilized under group-III rich conditions, and how these structures impact surface diffusion and impurity incorporation.

This work was supported in part by the US-DARPA-VIGIL program (Dr. H. Temkin).

4:10pm **D1-9 Highly Reflective and Thermally Stable Ni/Ag Multi-Layer Ohmic Contact to p-Type GaN**, ***J.H. Son***, *Y.H. Song, J.-L. Lee (jllee@postech.ac.kr)*, Pohang University of Science and Technology (POSTECH), Korea

Vertical-structure have been exploited in GaN-based light emitting diodes to improve light extraction efficiency and thermal stability. In VLEDs, emitted light from active regions is reflected-up from reflective ohmic contacts on p-type GaN. Ag is very suitable for reflective ohmic contacts due to its high reflectance (>95%)In addition, low contact resistivity have been obtained from Ag-based ohmic contacts annealed in oxygen ambient. However, annealing in oxygen ambient causes Ag to be oxidized and/or agglomerated, leading to degradation in both reflectance and adhesion to GaN and overlaid metals. Therefore, preventing Ag from oxidation and/or agglomeration is a key aspect in obtaining high quality Ag-based ohmic contacts suitable for application to high-power LEDs of solid-state lighting. Mg-doped p-type GaN films used in this work were grown on (0001)

sapphire substrate using MOCVD. For measurements of contact resistivity using the TLM, active regions were defined by ICP. Ni/Ag single-layer and multi-layer contacts were deposited by electron beam evaporation. Both samples were annealed at temperatures ranging from 300 to 500°C in air ambient. Current-voltage characteristics of the contacts were examined using a semiconductor parameter analyzer. Light reflectance of the contacts was measured using a monochromator and tungsten-halogen light source. In this paper, new metallization scheme with high reflectance and excellent thermal stabiltiy has been developed for obtaining low resistance ohmic contacts on p-type GaN. Ni/Ag multi-layer contact showed lower contact resistivity as low as 8.2×10^{-6} ωcm^2, higher reflectance of 84.3% at 460 nm than Ni/Ag single-layer contact after annealing at 450°C in air ambient. To evaluate the thermal stability of the contacts, the changes of contact resistivity and light reflectance as a function of annealing time at 500°C were measured. The contact resistivity increased from 4.6×10-5 to 6.1×10^{-3} ωcm^2 for the Ni/Ag single-layer contact after annealing for 24 hrs, but it increased from 2.1×10-5 to 8.5×10^{-4} Ωcm2 for the Ni/Ag multi-layer contact. The light reflectance of the Ni/Ag single-layer contact decreased to 72% after annealing for 24 hrs, but the Ni/Ag multi-layer contact showed higher reflectance of 81%. High resolution x-ray diffraction scans were to evaluate the microstructural changes of the contacts. We propose the origin on the enhanced thermal stability of Ni/Ag multi-layer ohmic contact to p-type GaN.

4:30pm **D1-10 Influence of rf Power on the Electrical and Mechanical Properties of CN Thin Films Deposited by Reactive RF Magnetron Sputtering**, ***I. Banerjee*** *(indranibanerjee@bitmesra.ac.in), N. Kumari, M. Kumar, S.K. Mahapatra,* Birla Institute of Technology, India*, S.K. Pabi,* Indian Institute of Technology Kharagpur, India*, P.K. Barhai*, Birla Institute of Technology, India

Nano structured Carbon nitride thin films were deposited at different rf power in the range of 150 to 225 W at a constant pressure and Ar/N2 ratio of 1:1 by reactive rf sputtering. The films were characterized by SEM, AFM for surface morphology and roughness respectively. Atomic weight percentage ratio of C/N in the films was determined using EDX and Raman Spectroscopy. The N/C ratio decreased from 0.75 to 0.59 with increase in rf power from 150 W to 225 W. The electrical properties were measured by LCR meter. The surface resistivity decreased with increase in rf power and C/N ratio in the films. The hardness of the films were measured using nano indenter. The hardness of the film increased with increase in rf power due to increase in sp3 hybridized C-N sites in the deposited films. The chemical bonding between Carbon and Nitrogen was characterized by FTIR spectroscopy and Raman spectroscopy. FTIR studies showed absorption band positioned at 1000-1700 cm-1 due to C–N, C=N, C=C, and at 2200 cm-1due to C≡N in each film.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E5

Nano- and Microtribology Instrumentation and Characterization
Moderator: N. Randall, CSM Instruments Inc., J. Hahn, Korea Research Institute of Standards and Science

1:30pm **E5-1 Nanotribology, Nanomechanics and Materials Characterization Studies and Applications to Bio/Mamotechnology and Biomimetics**, ***B. Bhushan*** *(bhushan.2@osu.edu)*, The Ohio State University **INVITED**

At most solid-solid interfaces of technological relevance, contact occurs at numerous asperities. A sharp atomic/friction force microscope (AFM/FFM) tip sliding on a surface simulates just one such contact. However, asperities come in all shapes and sizes which can be simulated using tips of different shapes and sizes. AFM/FFM techniques are commonly used for tribological studies of engineering surfaces at scales ranging from atomic- to microscales. Studies include surface characterization, adhesion, friction, scratching/wear, boundary lubrication, and surface potential and capacitance mapping[1-5]. AFMs and their modifications are also used for nanomechanical characterization, which includes measurement and analysis of hardness, elastic modulus and viscoelastic properties, and in-situ localized deformation studies. State-of-the-art contact mechanics models have been developed and are used to analyze dry and wet contacting interfaces. Experimental data exhibit scale effects in adhesion, friction, wear, and mechanical properties, and a comprehensive model for scale effects due to adhesion/deformation and meniscus effects has been developed. Generally, coefficients of friction and wear rates on micro- and nanoscales are smaller, whereas hardness is greater. Therefore, micro/nanotribological studies may help define the regimes for ultra-low friction and near-zero wear. New lubrication strategies such as the use of self-assembled monolayers promise to be very versatile and effective at these scales. To improve adhesion between biomolecules and silicon based surfaces, chemical conjugation as well as surface patterning have been used[6]. Friction and wear studies of biomolecules show that these act as a lubricant but wear away in AFM experiments, even when attached to the underlying substrate[7]. In the area of biomimetics, surface roughness present on lotus and other leaves has been measured and the surface films are characterized to understand the mechanisms responsible for superhydrophobicity (high contact angle). A model for surface-roughness-dependent contact angle has been developed and optimum distributions have been developed for superhydrophobic surfaces[8-10]. These fundamental nanotribological studies provide insight to molecular origins of interfacial phenomena including adhesion, friction, wear and lubrication. Friction and wear of lightly loaded micro/nano components are highly dependent on the surface interactions (few atomic layers). Nanotribological and nanomechanics studies are also valuable in the fundamental understanding of interfacial phenomena in macrostructures to provide a bridge between science and engineering. This talk will present an overview of nanotribological and nanomechanics studies and their applications.

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2:10pm **E5-3 Nanotribology of WSC and MoSeC Self-Lubricant Coatings**, ***T. Polcar*** *(tomas.polcar@dem.uc.pt),* SEG-CEMUC University of Coimbra & Czech Technical University - Prague, Czech Republic*, M. Evaristo,* University of Coimbra, Portugal*, R. Colaço,* Instituto Superior Tecnico, Portugal*, A. Cavaleiro*, University of Coimbra, Portugal

Transition metal dichalcogenides alloyed with carbon (TMD-C), namely WSC and MoSeC, deposited by magnetron sputtering were thoroughly studied with respect to their structure, mechanical and tribological properties. It was shown that TMD-C represented a new class of self-lubricating coatings maintaining low friction (below 0.05) in different environments, such as dry and humid air, elevated temperature or contact pressure. The coatings exhibited extremely high load-bearing capacity connected with remarkable low friction coefficient and wear rate. The unique tribological properties were attributed to two predominant features: (1) the formation of a thin tribolayer consisting of pure transition metal dichalcogenide (TMD) with basal planes optimally oriented for the low friction, i.e. parallel to the coating surface, and (2) the reorientation of the disordered TMD platelets under sliding. In this study, a nanotribometer (pin-on-plate) and atomic force microscope (AFM) with lateral force measurement (LFM) have been used to evaluate the nanotribology of selected TMD-C coatings. The friction under different contact pressure has been evaluated and compared with the macroscopic friction obtained on a standard pin-on-disc tribometer. The wear tracks have been characterized either by depth sensing methods or Raman spectroscopy, in order to determine the possible structural transformations in the contact. Moreover, the same techniques have been applied to the macroscopic wear tracks, where nanotribology helped to characterize the frictional properties of tribolayer.

2:30pm **E5-4 Micro-Tribological Performance of Metal/ MoS_2 Lubricants**, ***P. Stoyanov*** *(pantcho.stoyanov@mail.mcgill.ca), R. Chromik,* McGill University, Canada*, J.R. Lince*, The Aerospace Corporation

Solid lubricant coatings with co-sputtered metal and MoS_2 have shown favorable macrotribological properties at a wide range of contact stresses and humidity levels. These materials are also candidates for microcontacts and micro-electromechanical systems (MEMS), but their performance at this scale is poorly understood. For this study, microtribological properties of Au/ MoS_2 and Ti/ MoS_2 coatings, with varying metal additives of 0-80 at.% and 0-10 at.% respectively, were examined using a nanoindentation instrument. Titanium and gold were chosen for this study as metal additives due to their different influence on the mechanical properties of the coating. The hardness, H, and reduced modulus, E, of the coatings varied with the different additives and increased with increasing the metal composition. Au/ MoS_2 coatings had H = 1-2 GPa and E = 40-90 GPa, whereas the hardness and the reduced modulus for Ti/ MoS_2 was 5-6 GPa and 100-120 GPa respec tively. Reciprocating microscratch tests were performed with spherical diamond tips with radii between 10 μm and 100 μm. A range of normal loads were used between 0.2 mN and 5.0 mN. Characterization of the wear track and transfer films was performed using a micro-Raman spectrometer and an atomic force microscope. Friction measurements were analyzed with a contact model that contains the Hertzian elastic term and a plowing component. Non-linear fitting with the model revealed a variation in the two friction components at higher contact stresses with respect to the different metal concentration (i.e. the plowing component decreases with increasing the metal additive). These results were correlated to the mechanical properties, difference in the transfer film formation, and the surface of the wear track.

2:50pm **E5-5 Application of Diamond-Like Nanocomposite Coatings for Microsystems Tribology**[1], ***S. Prasad*** *(svprasa@sandia.gov),* Sandia National Laboratories*, T. Scharf*, The University of North Texas **INVITED**

A variety of fabrication methods are available to build Microsystems: bulk micromachining, surface micromachining (SMM), LIGA (German acronym for Lithography, Galvanoformung und Abformung), and meso-scale machining. Microsystems fabricated by LIGA and meso-scale machining are more amenable to applying thin films and coatings to mitigate friction and wear. The major focus of this presentation will be the on tribological issues associated with structures created in the LIGA process, specifically electroformed metal structures with dimensions of 0.01 to several mm, and thickness up to 500 μm. Diamond-like nanocomposite (DLN) coatings processed from siloxane precursors by plasma enhanced chemical vapor deposition are well known for their low friction and wear behavior.

In this study, we have examined the feasibility of applying these coatings on the sidewalls of LIGA-fabricated microsystems parts. Novel tribological tests were designed to evaluate the frictio n behavior of coated parts in planar-sidewall and sidewall-sidewall contacts, in regimes relevant to MEMS. Friction coefficients of DLN coated LIGA parts ranged from an extremely low value of 0.02 in dry nitrogen to 0.2 in air with 50% RH, with minimal amounts of wear in both environments.

Like many solid lubricating materials, DLN coatings exhibited non-Amontonian friction behavior; with coefficient of friction (COF) decreasing with an increase in Hertzian contact stress. The main mechanism responsible for low friction and wear is governed by the interfacial sliding between the DLN coating and the friction-induced transfer film on the counterface. This interfacial shear strength, computed from COF-inverse Hertzian contact stress plots, was found to be 9 Mpa in dry nitrogen and 78 Mpa in humid air. Time-of-Flight Secondary Ion Mass Spectroscopy (ToF-SIMS) analysis of the interfaces (wear tracks and transfer films) was used to gain a fundamental understanding of tr ibochemical phenomena. The role of finite element analysis and novel characterization techniques (e.g., FIB, EBSD, Micro-impact testing) to evaluate the surface coatings for microsystems technology will be discussed.

[1] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

3:30pm **E5-7 Nanotribological Properties of Topographically-Chemically Modified Silicon Surfaces**, *D.C. Pham, R.A. Singh, K. Na, S. Yang,* ***E.-S. Yoon*** *(esyoon@kist.re.kr)*, Korea Institure of Science and Technology, Korea

This work reports on the effect of combination of topographical and chemical modifications on the nano-scale adhesion and friction properties of the silicon surface. Silicon wafers (100) were patterned into nanopillars by MEMS fabrication process. The patterned surfaces were then chemically treated by coating thin diamond-like carbon (DLC) film (thickness 20 nm) using a plasma-assisted chemical vapor deposition method. The morphology and hydrophobicity of modified surfaces were investigated by scanning electron microscopy (SEM) and a water contact angle analyzer. The surfaces were tested for their adhesion and friction properties at nanoscale using an atomic force microscope (AFM). Borosilicate balls with two different sizes mounted on silicon nitride triangle cantilevers were used as tips. Results showed that the combined surface modification of patterning and DLC coating significantly increased hydrophobicity of silicon surface. Both the topographical and chemical mod ifications reduced the adhesion and friction forces; however, it was observed that a combination of these two kinds of modifications was more effective in reducing these forces. The physically reduced area of contact through patterning and the lower interfacial energy of DLC coating together contributed to the reduction in the adhesion and friction of the silicon surfaces with the combined modifications. It was also observed that these adhesion and friction forces increased with the tip size.

3:50pm **E5-8 Nano-Tribological Properties of Undulated Ultra Thin Diamond Patterns**, *D.-S. Lim (dslim@korea.ac.kr),* ***S.K. Lee***, *J.H. Kim*, Korea University, Korea

Surface roughness controlled ultra thin nano crystalline diamond film was fabricated on Si(100) surface [1]. To simulate MEMS and NEMS patterned surface, 800 @nanometer@ and 1 @micrometer@ wide lines with 200 @nanometer@ wide space pattern were prepared on Si(100) substrate by E-Beam lithography and RIE process. ESAND (Electrostatic Self-assembly of Nano Diamond) seeding process was employed to initiate nano crystalline diamond growth. For seeding procedure, negatively charged substrate surface was covered with cationic polymer (PMMA) monolayer, and anionic polymer (PSS) coated cationic nano diamond particles were spontaneously attached on the substrate. Through this process, undulated nanocrystalline diamond pattern was successfully formed by conventional hot filament CVD system. The roughness of deposited surface was controlled by regulating the size of seeding nano diamond particles. Crushing nanodiamond aggregates and dispersion of nano diamond solution was performed in attrition milling system. AFM probe was used for the wear test and surface profiling of nanocrystalline diamond coatings. 2D frictional coefficient mapping by LFM (Lateral Force Microscopy) scanning showed low friction coefficient (< 0.1) on line patterned diamond surface, and higher friction coefficient (> 0.3) on narrow area adjacent to undulated pattern edges. With prolonged LFM scanning, the high coefficient of friction was decreased down to below 0.1. The morphology of nanocrystalline diamond was analyzed with Raman spectroscopy.

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4:10pm **E5-9 Nanotribology of Multilayered Nano Thin Films by Molecular Dynamics Simulations**, ***J.-C. Huang*** *(jc-huang@mail.tnu.edu.tw)*, Tungnan University, Taiwan

The molecular dynamics simulation method was used to investigate the tribological characteristics of multilayered nano thin films. The NVT ensemble principle and COMPASS potential function were employed in the simulation. The multilayered nano thin film contained the Cu and Ni thin films in sequence. The nano tribological phenomena of the Cu/Ni multilayered nano thin film was studied under diamond tool in vacuum and water environments. It was concluded that the nanotribology behavior of Cu/Ni multilayered nano thin film differed under different environments.

Characterization: Linking Synthesis Properties and Microstructure
Room: Sunrise - Session F1-1

Advances in Characterization of Coatings and Thin Films

Moderator: P. Schaaf, TU Ilmenau, M. Baker, University of Surrey

1:30pm **F1-1-1 Characterization of Cu-Sn Intermetallic Layers by AFM Force Curves Analysis and Nanoindentation**, ***M.A. Monclus*** *(miguel.monclus@npl.co.uk), T.J. Young, D. Di Maio, N.M. Jennett*, National Physical Laboratory, United Kingdom

Atomic Force Microscopy (AFM) is increasingly being used as a nanoindentation tool to measure local elastic properties of surfaces. In this paper, a high-resolution AFM method was applied to characterise local mechanical properties of the intermetallic layer between a Sn alloy and a Cu interconnect. Force-distance curve maps were obtained on Cu-Sn interconnect regions using diamond AFM tips. The processing of AFM

indentation data was automated by customised software that can be applied to up to 4096 force-distance curves to obtain stiffness and elastic modulus maps. The program corrects the obtained force-distance curves by choosing the most suitable contact point and also takes into account the cantilever deflection to obtain true indentation curves. The most suitable model is then applied to the AFM indentation data. Depending on the AFM tip geometry and the type of material tested, a suitable reference material is used to estimate the tip radius and the area function needed to extract the elastic modulus values. The accuracy of the technique was discussed by comparison with values obtained by conventional nanoindentation on the Sn and Cu surface regions. Elastic modulus variations in the region of a Cu-Sn interface revealed the presence of the intermetallic layer, which could be distinguished by its different elastic properties. The proposed high-resolution AFM measurement method allows for the identification of materials at the nanoscale.

1:50pm **F1-1-2 Effect of Stoichiometry on Structure and Texture of $Zr_{1-x}Si_xN$ Thin Films**, *X. Zhang, M.S. Byrne,* ***R.J. Lad*** *(rjlad@maine.edu)*, University of Maine

A series of zirconium silicon nitride ($Zr_{1-x}Si_xN$) thin films were grown on r-plane sapphire substrates using rf magnetron co-sputtering of Zr and Si targets in a N_2/Ar plasma. X-ray diffraction pole figure analysis, x-ray reflectivity, x-ray photoelectron spectroscopy, atomic force microscopy, and optical absorption spectroscopy were used to characterize the film stoichiometries and structures after growth at 200°C and following annealing up to 1000°C in vacuum. ZrN films grow with high quality (100) heteroepitaxy on r-sapphire, but the addition of Si up to x=15% induces film strain and causes the films to adopt a polycrystalline structure with (100) fiber texture. For x>15%, the films are amorphous even after vacuum annealing at 1000°C. XPS spectra and x-ray reflectivity data indicate that the Zr-Si-N films at all stoichiometries are very homogeneous. AFM revealed that 150 nm thick films have a roughness on the order of 1 nm, except for Si stoichiometries corresponding to where the film transforms from polycrystalline to amorphous structure. At this transition, evidence was found for regions of film delamination and hillock formation, which is presumably driven by strain at the film-substrate interface. UV-visible absorption spectra also were found to depend on the Si content. For Si-rich films (x>15%), the absorption edge and band gap are directly proportional to the Si content, whereas for Zr-rich films (x<15%), there is no band gap and the films are highly conductive.

2:10pm **F1-1-4 Atom Probe Tomography of Thin Films**, ***D. Larson*** *(dlarson@imago.com)*, Imago Scientific Instruments **INVITED**

Atom probe tomography can provide 3-D atomic-scale structural and compositional analysis of materials difficult to duplicate using other high-performance microscopy techniques. Recent specimen fabrication techniques using focused-ion beam instruments with in-situ manipulation now allows a variety of nanoscale thin films with site-specific features to be prepared and analyzed using atom probe tomography. This talk will address the fabrication and atom probe analysis, as well as comparison to other characterization techniques, of the microstructure of a variety of nanomagnetic thin film devices including multilayers, spin valves and tunnel barriers, and quantum well layered features.

2:50pm **F1-1-6 Three-Dimensional Atom Probe Tomography of Nanocomposite Diamond-like Carbon Films**, ***T.W. Scharf*** *(thomas.scharf@unt.edu), M.C. Romanes, R. Banerjee,* The University of North Texas*, R.D. Evans, G.L. Doll*, Timken Company

Three-dimensional atom probe tomography (3DAP) studies have been carried out on hydrogenated diamond-like carbon (DLC) films. Nanocomposite tungsten carbide (predominately cubic β-WC_{1-x} phase) in mixed amorphous DLC sp^3/sp^2 C-H matrices were characterized using the combination of HAADF STEM imaging and local electrode atom probe (LEAP) techniques. Alternating ~25 nm thick carbon-rich and tungsten-rich DLC layers were observed by HAADF STEM imaging while HRTEM in the tungsten-rich layers revealed well dispersed, nanocrystalline WC (~2–4 nm) precipitates throughout the amorphous C-H matrix. Excellent correspondence in the chemical layering was observed with 3DAP which also revealed atomic-scale composition containing carbon and hydrocarbon-rich fragments (C_3^{2+} and $C_2H_4^+$) and tungsten and elemental carbon fragments (W^{2+}, WC^{2+}, C^+ and C^{2+}) in the carbon-rich and tungsten-rich layers, respectively. 3D morphology and chemical partitioning of the WC nanoprecipitates in the tungsten-rich layers will also be presented. The role of structure, phase distribution, and chemistry at the atomic and nanometer scales in controlling the tribological and mechanical properties at the macroscale will also be discussed.

3:10pm **F1-1-7 Tip Enhanced Raman Spectroscopy for High Resolution Assessment of Strained Silicon Devices**, ***L. Sanderson*** *(lisa.sanderson@ncl.ac.uk), P. Dobrosz, S.H. Olsen, S.J. Bull,* Newcastle University, United Kingdom*, S. Mantl, D.M. Buca*, FZ-Juelich, Germany

Due to aggressive scaling of semiconductor devices, strained silicon technology is an attractive option in the semiconductor industry in order to keep technology improving at a steady rate. Introducing a strained layer into the channel region of a device enhances transport mechanisms by improving hole or electron mobilities. The amount of strain induced in a device requires close monitoring as a function of processing since relaxation processes may occur. Raman Spectroscopy is a well-known technique for characterising the strain in strained Si/SiGe devices but the spatial resolution of this technique is limited by the laser spot size to between 0.5 - 1µm. Typically current strain measurement requirements are at the scale of about 50nm. Tip Enhanced Raman Spectroscopy (TERS) is a technique which exhibits large signal enhancements as well as a greatly improved spatial resolution allowing much smaller device features to be measured with some accuracy. TERS makes use of the localised surface plasmons produced by a metallised AFM tip to create a large electric field enhancement in the vicinity of the tip apex, improving the spatial resolution to the nanometre range (or that of the tip's diameter). This paper reports the strain profiling of Strained Silicon on Insulator (SSOI) stripes of various widths in the nanometre range using TERS. The stripes were fabricated in such a way that pseudo-uniaxial strain was induced to improve hole mobility. Finite Element simulation results show good agreement to the experimental work. This work illustrates the potential of TERS to become a powerful submicron in-situ characterisation technique.

3:30pm **F1-1-8 Depth Profile Analysis of Thin Layers on Glasses and Ceramics by RF GD OES**, ***P. Hunault*** *(philippe.hunault@jobinyvon.com),* Horiba Jobin Yvon Inc.*, P. Chapon, C. Tauziede, A. Tempez,* Horiba Jobin Yvon Inc., France*, M. Ganciu,* NILPRP - Bucharest, Romania*, P. Guillot,* University JF Champollion - Albi, France*, P. Belenguer*, Laplace CNRS, France

Radio Frequency Glow Discharge Optical Emission Spectrometry (RF GD-OES) is an established technique capable of Ultra Fast Elemental Depth Profiling of thin films down to the nanometre. The application of the technique to the characterisation of thin layers on glasses and ceramics is investigated. Issues related to heat and thermal damage as well as coupling efficiency are shown and solutions to overcome these issues are presented resulting from theoretical and experimental characterisations of the RF GD plasma and of its interaction with the material surface. The new RF coupler that is now available allows sputtering of up to 5mm thick glasses or ceramics and provides adequate performances for valuable analysis of such materials as examples from solar cells and coated ceramics will illustrate.

3:50pm **F1-1-9 Local-Order Information of the Decomposition of Nanocrystalline Supersaturated TiAlN by X-ray Absorption Spectroscopy**, ***J. Endrino*** *(jlendrino@icmm.csic.es), R. Gago,* Instituto de Ciencia de Materiales de Madrid, Spain*, G. Fox-Rabinovich,* McMaster University, Canada*, A. Gutierrez*, Universidad Autonoma de Madrid, Spain

Even though the decomposition of supersaturated metastable TiAlN with NaCl-type structure (c-TiAlN) upon annealing has been the subject of considerable research effort in the last decade, the evolution of the local electronic structure with annealing temperature is largely unknown. Thus far, phase transformation into coherent cubic domains has been explained by the spinodal decomposition of the metastable c-TiAlN phase upon annealing, although Al content and distribution, together with structural disorder, can play a significant role in the compound evolution and stability upon annealing. Synchrotron radiation spectroscopic techniques offer an opportunity of providing local-order information of the phenomena with site-selective information. In this study, we present X-ray absorption near edge structure (XANES) spectra of the Ti-K, Al-K, and N-K edges of supersaturated TiAlN samples annealed between 700ºC and 900ºC. Thermal treatments in this temperature range lead to the formation of coherent cubic nanocrystallites with no detrimental hexagonal phase segregation, as observed by high-resolution transmission electron microscopy and electron diffraction patterns. XANES corroborates this structural information at a local order. Further, the origin of the XANES spectral features is further investigated by using resonant inelastic X-ray scattering (RIXS) near the N K-edge.

4:10pm **F1-1-10 Testing Thin Films by Microcompression: Benefits and Limits**, *D. Kiener, H.P. Woergoetter,* Erich Schmid Institute of Materials Science, Austria*,* ***G. Dehm*** *(gerhard.dehm@mu-leoben.at)*, University of Leoben, Austria

Coatings are very important for numerous industrial applications such as increasing the wear resistance of tools, providing thermal conductivity to components in engines or electric conductivity in microelectronic devices. The mechanical properties of the coatings are of prime concern. The

practicability of performing miniaturized compression tests in order to determine mechanical properties of various thin films was analysed. Four coatings, a polycrystalline tungsten coating, a single crystal copper coating and two single crystal hard coatings (vanadium nitride and titanium nitride) were tested. The compression samples were fabricated using a focused ion beam microscope. The compression tests were executed with a micro-indenter installed in a scanning electron microscope. The deformation of the sample was observed and in-situ recorded by scanning electron microscopy. From the measured load-displacement data true stress-true strain curves were calculated. The limits and benefits of the microcompression technique are discussed.

4:30pm **F1-1-11 Contact Angle Analysis of Sol-Gel Derived Zirconia Based Hybrid Coatings on 304 Stainless Steel Substrates**, ***S. Ashokkumar*** *(sas@bio.dtu.dk), J. Hinke,* Accoat A/S, Denmark*, J.A. Nissen, P. Moller*, Technical University of Denmark

Sol-gel derived hybrid coatings were prepared from 3-Glycidoxypropyltrimethoxysilane (GPTMS), tetraethoxysilane (TEOS) and tetra-n-propoxy zirconium (TPOZ) precursors on 304 stainless steel substrates by dip and spray coating methods. The influencing parameters such as precursor ratio, number of dippings, stirring time and temperature on the morphology, chemical composition and contact angle of the coatings were studied. The chemical composition and surface morphology of the coatings have been analyzed by XPS and SEM.

The contact angle measurement were made manually by delivering a 4µmlitre drop of triple distilled water from a microsyringe onto the surface of the sample plate (10x10x1mm) placed on a horizontal stage. The photo of the static water droplet was captured by a digital video camera of high resolution equipped with TECHSPEC Silver series Telecentric measuring lense. The measurement of the contact angle was made with a program called DropSnake. Mea surements were repeated five times with new drops at different positions on the sample. The reported contact angles are the average value of the five measurements. Contact angles have an estimated accuracy of +-2 degrees.The coating prepared by dip coating method with an inorganic precursor containing tetra-n-propoxy zirconium (TPOZ) and ethylacetoacetate volume ratio of 1:1 at 55 degree celsius with a stirring time of 2 hours exhibited a higher contact angle of 79.4° when compared to coatings prepared with other different conditions.

New Horizons in Coatings and Thin Films
Room: Tiki Pavilion - Session H1-2

Nanotube, Nanowire and Nanoparticle Thin-Films and Coatings
Moderator: R. Martel, Université de Montréal

1:30pm **H1-2-1 Temperature Dependent Conduction in ZnO Nanowires**, ***J. Lu*** *(jialu@usc.edu)*, University of Southern California **INVITED**

ZnO nanowire has been intensively studied due to its excellent optical property, relative high conductivity, and versatile applications as electro-optic and electro-mechanical devices. Thin nanowires with diameters ranging from 20 to 50 nanometers were synthesized via chemical vapor deposition. The as-grown ZnO nanowires were then configured into field effect transistor (FET) devices through a series of fabrication processes and measured in a 4 K helium cryostat with quartz optical view port. From the transport measurement of ZnO FET, the Arrhenius behavior of the DC conductivity curve exhibits two regions. For T > 50 K, the conductivity show thermionic emission conduction in non-degenerate semiconductor and can be expressed as $\sigma = \sigma_0 \exp(-E_a/kT)$, where E_a is the activation energy, and k is the Boltzman constant. Without gate voltage, the activation energy value extracted from multiple samples falls in the range between 30-60 meV, correspond ing to shallow donor levels below the conduction band edge that are mainly contributed by oxygen vacancies. Under applied gate voltages, band bending occurs and lowers the activation energy, and the reduction in Ea is extracted experimentally. On the other hand, the activation energy at T < 50K is all below 1 meV. It was found that in this regime, 3D Mott's variable range hopping model governs the transport, with conductivity $\sigma = \sigma_0 \exp(-AT^{-1/4})$, where A and σ0 are material parameters. Furthermore, it is observed that the ZnO nanowires under continuous UV irradiation shows a metallic behavior for temperature above 210 K. This phenomenon is attributed to the reduction in mobility as a result of significantly increased charge carriers.

2:10pm **H1-2-3 Growth and Characterizations of ZnO Nanorod/Film Structures**, ***W.Y. Wu*** *(wanyu@mail.mse.ncku.edu.tw), M.D. Chen, J.M. Ting*, National Cheng Kung University, Taiwan

1D nanostructure materials such as nanowires, nanotubes, and nanorods attract much attention due to their unique properties to bulk materials. Among them, ZnO nanorods, representing one of the most important low-dimensional materials, find its applications in many different fields. There are a number of methods that are used for the growth of ZnO nanorods and a common mechanism of the so-called vapor-liquid-solid (VLS) mechanism has been used widely to explain the growth of ZnO nanorods. However, we have recently reported a new mechanism for the growth of ZnO nanorods using a sputter deposition process[1]. In this paper, we further examine the characteristics of the sputter deposited ZnO. The deposits were characterized using x-ray diffractometry, scanning electron microscopy, and atomic force microscopy. The deposited ZnO was found to consist of ZnO nanorods and the roots of the nanorods were embedded in a ZnO layer that was co-deposited with the nanorods at the beginning of the process. Effects of processing conditions are presented and discussed. [1] [1]Ming-Ta Chen, Jyh-Ming Ting, Thin Solid Films v.494 (2006) p.250 – 254.

[22:30pm] **H1-2-4 Sputter Deposited ZnO Nanowire/Thin Film Structures**, *T.L. Chou, W.Y. Wu,* ***J.M. Ting*** *(jting@mail.ncku.edu.tw)*, National Cheng Kung University, Taiwan

Integrated ZnO nanowires/thin film structures were obtained on amorphous glass substrates having an electroless plated copper surface layer using an rf magnetron sputter deposition technique. The growth took place under different O_2/Ar ratios. For ZnO nanowires, a higher O_2 partial pressure makes their averaged diameters decrease slightly but the area densities increase. The growth and the characteristics of the integrated ZnO nanowires/thin film structures are presented. X-ray diffraction and transmission electron microscopy characterizations show that the as-grown nanowires and the thin films are both polycrystalline with a (002) preferred orientation. No catalyst was found at the ends of the nanowires, indicating the vapor-liquid-solid mechanism is not applicable in the sputter deposition process. It was also found that the growth of nanowires competes with that of thin film. The length of the ZnO nanowires is nearly proportional to $time^{0.5}$, indicating a diffusion controlled growth; however, the thickness of the ZnO thin films increases linearly with the growth time. Furthermore, a higher oxygen partial pressure leads to a lower deposition rate for the ZnO thin films; while a higher oxygen partial pressure gives a smaller average diameter and lower area density for the ZnO nanowires.

2:50pm **H1-2-5 Formation of Titanium Germanosilicide Nanocrystals for Nonvolatile Memory Application**, ***L.-W. Feng*** *(b8823048@student.nsysu.edu.tw),* National Chiao Tung University, Taiwan*, T.-C. Chang,* National Sun Yat-Sen University, Taiwan*, C.-H. Tu, P.-S. Wang,* National Chiao Tung University, Taiwan*, D.-S. Gan, N.-J. Ho, H.-J. Huang, M.-C. Chen,* National Sun Yat-Sen University, Taiwan*, C.-Y. Chang*, National Chiao Tung University, Taiwan

Titanium Germanosilicide nanocrystals fabricated by co-sputtering titanium silicide and germanium were investigated in this paper. The characteristics of Titanium Germanosilicide composition were analyzed by X-ray photon-emission spectra and the formations of nanocrystals were observed by high resolution transmission electron microscopy under the various post-annealing temperatures. It was found that the post-annealing temperature of Titanium Germanosilicide nanocrystals for memory application can be lowered which was compared to those proposed in $TiSi_2$-based papers. However, the pre-capped silicon dioxide before thermal annealing was needed due to the out-diffusion of the germanium at relatively higher annealing temperature. Electrical measurements of the flat band voltage shift and retention characteristics are exhibited for nonvolatile memory application.

3:10pm **H1-2-6 Fabrication of Molybdenum Oxide Nanowires Grown by Thermal CVD**, *H.C. Hsih (hcshih@mx.nthu.edu.tw),* Chinese Culture University, & National Tsing Hua University, Taiwan*,* ***C.C. Chang***, National Tsing Hua University, Taiwan*, M.W. Huang,* National Chung Hsing University, Taiwan*, Y.T. Hsieh,* National Tsing Hua University, Taiwan*, B.J. Wei*, National Chung Hsing University, Taiwan

Molybdenum oxide (Mo_xO_y) nanowires were prepared successfully by thermal chemical vapor deposition through a two-step evaporation process and distributed on a p-type Si (100) substrate from Molybdenum powders (purity: 99.99%). The growth of the Mo_xO_y nanowires was made in a vacuumed quartz tube at a pressure of 6 x 10-5 torr and with heating rate of 15 degree centigrade per minute using a mixture gases of Ar and oxide at the first stage to preheat the substrate at 450 degree centigrade, the reheating sample to synthesis temperature at 850 degree centigrade. The as-synthesized Mo_xO_y nanowires were characterized by high-resolution transmission electron microscopy, field emission scanning electron microscopy, x-ray diffraction, and Raman scattered. The morphologies of

Molybdenum oxide nanowires were rod-like shape and the diameter were range from 10 to 500 nanometer and length up to several hundreds nanometers. The optical properties of the Mo_xO_y nanowires were also inves ted by photoluminescence and cathodluminescence spectra. The effects of the Mo_xO_y nanowires on the fluorescence properties were also investigated in detail.

3:30pm **H1-2-7 Thermally Evaporated Sb-Doped SnO_2 Nanobelts for Ethanol Gas Sensors**, ***P.-S. Lee*** *(g9731581@oz.nthu.edu.tw), Y.-H. Lin, C.-C. Kuo,* National Tsing Hua University, Taiwan*, J.-M. Wu,* Feng-Cha University, Taiwan*, Y.-S. Chang, H.C. Shih*, National Tsing Hua University, Taiwan

One-dimensional (1-D) tin oxide nanostructures including nanowires, nanorods and nanobelts have attracted much attention owing to their extraordinary application in nanoscale devices@super 1-3@. In order to enhance and modulate the various properties, SnO@sub 2@ thin films and nanostructures are frequently doped with an amount of appreciate elements, such as indium (In) and antimony (Sb)@super 4-5@. In this study, single crystalline tetragonal rutile Sb-doped SnO@sub 2@ nanobelts were synthesized on the Al@sub 2@O @sub 3@substrate using a thermal evaporation method. The morphology and microstructure of Sb-doped SnO@sub 2@nanobelts were characterized by a field emission scanning electron microscope (FESEM) and a transmission electron microscope (TEM). The composition and chemical bonding nature were investigated by x-ray photoelectron spectroscopy (XPS) and nano-Auger analysis (nano-AES). At room temperature, the Sb-doped SnO@sub 2@ nanobelts exhibit a high sensitivity towards eth anol gas of concentrations ranging from 50 to 500 ppm. Comparative gas sensing results reveal that the prepared Sb-doped SnO@sub 2@ nanobelts sensors exhibit a much higher sensing sensitivity and recovery property in detecting ethanol gas at room temperature than the pure SnO@sub 2@ nanowires sensor. This study shows that doping with Sb can improve the sensitivity of SnO@sub 2@ nanostructures significantly.

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@super 3@Y. Zhang, A. Kolmakov, S. Chretien, H. Metiu, and M. Moskovits: Nano Lett. 4 (2004) 403.

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3:50pm **H1-2-8 Fabrication and Characterization of ZnO Nanowire Arrays Prepared by a Simple Hydrothermal Method**, ***S.-N. Bai*** *(snbai@ctu.edu.tw)*, Chienkuo Technology University, Taiwan

A hydrothermal method has been used for synthesizing well-aligned crystalline ZnO nanowires with hexagonal structure on silicon substrates (100). The sol-gel deposition method was applied to prepare a ZnO film on the substrate and serve as seed layer for the subsequent growth of ZnO nanowires. On the ZnO seed-layer film, highly oriented nanowire arrays were successfully synthesized by solution growth process using zinc nitrate and hexamethylenetetramine in aqueous solution. The orientation and morphology of the ZnO nanowires vary with changing the concentration of reaction precursors in the range from 0.01 to 0.25M. The length of the ZnO nanowires is also controlled by the concentration of reaction precursors in the aqueous solution. The crystalline structure and orientation of the nanowires was characterized by the X-ray diffraction (XRD) using CuKa (l = 1.5418 Å) radiation. The morphology and the microstructure of the ZnO nanowires over the silicon substrates wer e investigated by using field-emission scanning electron microscopy (FE-SEM) and transmission electron microscopy (TEM), respectively. The optical properties were analyzed by using the cathodoluminescence (CL) with an electron beam as an excitation source (15 kV) at room temperature. In the CL spectra, a sharp near band edge exciton emission peak and a broad defected-related peak were observed. This low-cost hydrothermal growth method provides a mass-production synthesis of ZnO nanowires for many important applications in nano-scale devices.

4:10pm **H1-2-9 Hybrid Manganese Oxide Films for Supercapacitor Application Prepared by Sol-Gel Technique**, ***C.-K. Lin*** *(cklin@fcu.edu.tw), C.-H. Cheng, Y.-H. Tien, C.-Y. Chen,* Feng Chia University, Taiwan*, S.-C. Wang,* Southern Taiwan University, Taiwan*, W.T. Tsai*, National Cheng Kung University, Taiwan

Hybrid films were prepared by adding various concentrations of Meso-Carbon Microbeads (MCMB) during sol-gel processing of manganese oxide films. The as-prepared and heat-treated films were characterized by scanning electron microscopy, X-ray diffraction, and synchrotron X-ray absorption spectroscopy. In addition, electrochemical performance of the hybrid manganese oxide films was evaluated by cyclic voltammetry (CV) and compared with its pure counterpart.

Experimental results showed that manganese oxide films exhibited a mixture of Mn_2O_3 and Mn_3O_4 phases. The higher is the heat-treating temperature, the more the Mn_2O_3 can be observed. The specific capacitance of the pure manganese oxide electrodes is 209.2 F/g. A significant improvement, however, can be noticed by adding MCMB. The 350 oC heat-treated hybrid manganese oxide films (MnOx:MCMB = 5:1) exhibit the highest specific capacitance of 456.7 F/g (~120% increase). After 1200 repetitive charging-discharging cycles, the specific capacitance of pure MnO_x film decreases to ~50 % of its maximum value. While that of the hybrid MnOx/MCMB film can still have 407.4 F/g, ~90% of its maximum value. Not only specific capacitance increases but chemical stability enhances with the addition of MCMB.

Atomistics of Thin Films Growth
Room: Sunset - Session TS1

Computational & Experimental Studies of Thin Film Growth: An Atomistic View

Moderator: S. Kodambaka, University of California - Los Angeles, J. Rosén, Linköping University

1:30pm **TS1-1 In Situ, Real-Time Observation of Thin Film Deposition: Roughening, Zeno, Grain Boundary Corssoin Barrier, and Steering**, ***M.J. Rost*** *(rost@physics.leidenuniv.ml)*, Leiden University, Netherlands **INVITED**

Thin polycrystalline metal films are becoming increasingly important, as is reflected in the multitude of applications in nanotechnology, nanooptics, microelectronics, vacuum coating, catalysis, medical science, sensor elements, wear protection layers, decorative coatings, and the synthesis of new materials.

As thin film properties are intrinsically linked to the precise film structure, one would like to control the overall film morphology down to the nanometer scale. This clearly demands fundamental research that links well-known atomic processes, such as diffusion and nucleation, with the mesoscopic film evolution during film growth.

Applying video-rate Scanning Tunneling Microscopy (STM)[1], we succeeded in visualizing film growth with atomic-scale resolution in real-time[2]. We evaporated several tens of monolayers of gold on top of a well-annealed polycrystalline gold film, while continuously observing the evolving surface with the microscope. These measurements directly visualize atomic processes that take place during film growth.

Analyzing the evolving film structure, we observe a significant increase in the film roughness, which we explain by considering both “well-known”, single crystalline growth modes in combination with additional polycrystalline effects[2,3]. The grain boundaries play a crucial role in the evolution, as they initiate mound formation, thereby significantly increasing the total film roughness. A possible additional roughness contribution comes from atom steering, which also can delay the film closure in the early stages during film growth.

[1] M.J. Rost et al.; Rev. Sci. Instr. 76, 053710-1 (2005)

[2] M.J. Rost; Phys. Rev. Lett. 99, 266101 (2007)

[3] M.J. Rost et al.; Phys. Rev. Lett. 91, 026101 (2003)}

2:10pm **TS1-3 Evolution of Residual Stress During Thin Film Growth: Effect of Competing Kinetic Processes**, ***E. Chason*** *(Eric_Chason@brown.edu)*, Brown University **INVITED**

During deposition, thin films go through a range of stress states, changing from compressive to tensile and back again. This evolution can be understood as a kinetic competition between different mechanisms of stress generation and relaxation. The balance between them shifts as the microstructure evolves from isolated islands, through coalescence and finally into a steady state uniform film. Real-time measurements of stress using wafer curvature enable us to monitor these changes while the film is growing. We will review some experiments of stress evolution in thin films grown by different processes (sputtering, evaporation, electrodeposition). A kinetic model will be described that balances the different stress generation processes and show how it can explain the effect of changing the growth conditions on the resulting stress.

2:50pm **TS1-5 Stress and Phase Transformation During Growth of Metal Films on Silicon: an in Situ Study**, ***A. Fillon***, Université de Poitiers-CNRS, France, *G. Abadias (gregory.abadias@univ-poitiers.fr), A. Michel, C. Jaouen*, Universite de Poitiers-CNRS, France

The origin of stress in polycrystalline thin films has gained recently a renewed interest with the potentiality offered by real-time techniques to measure stress evolution during growth. Such in situ measurements not only yield the magnitude of the growth stress, but also provide insights into the growth mechanism itself. Stress models have been proposed in the literature to explain the complex behaviour of high-mobility materials grown by evaporation. Stress evolution during sputtering of metal has been less investigated, although stress producing or relieving mechanisms like intermixing, phase stabilization and defect formation may be particularly favored due to the energetics of the growth process. In the present work, we investigated the stress evolution during growth of metallic thin films on silicon, a system which is also of technological relevance

$Mo_{1-x}Si_x$ alloy thin films, with x ranging from 0 to 0.5 and film thickness up to 200 nm, were grown at 300 K on amorphous Si (a-Si) seed layers by magnetron sputtering under low Ar pressure (0.2 Pa). The stress evolution during growth was monitored in-situ with a multiple-beam optical stress sensor. Different stages of stress evolution could be evidenced and related to specific physical processes thanks to additional ex situ complementary observations, such as transmission electron microscopy and atomic force microscopy. It is shown that pure Mo and $Mo_{1-x}Si_x$ alloys initially grew in the amorphous phase, due to the stabilizing effect of the interfacial free energy (required to create interfaces to the a-Si seed layer). Then, a polymorphous glass-to-crystal transformation occurred at a concentration dependent critical thickness, followed by the development of a large tensile stress during subsequent crystalline growth. Increasing the Si content reduced the driving force for crystallization and the amorphous state was stabilized in the volume above a critica l solute concentration x = 0.19 and characterized by a constant compressive stress attributed to surface stress effects.

3:10pm **TS1-6 Atomistic Simulations of Plasma Sprayed Thin Film Growth**, ***E. Webb*** *(ebwebb@sandia.gov)*, Sandia National Laboratories

Atomistic simulations of metallic droplet impingement onto a metal substrate have been performed to model thin film growth via plasma spray processing. The plasma spray process involves injection of solid material into a heat source, or plasma, melting of the constituent particles, and acceleration of the molten particles towards a substrate. Upon impact with the substrate, molten particles deform and flatten onto the substrate and then solidify as they cool. Due to the typically high kinetic energy of impacting particles, their final solidified shape is highly asymmetric as they form disks or splats on the substrate. The shape of individual solidified particles manifests itself in the microstructure of deposited films as a lamellar morphology often emerges. This morphology is associated with many of the properties inherent in plasma sprayed coatings including beneficial thermal, chemical, and mechanical resistance so it is of use to understand how droplet properties ma nifest in final splat morphology. While conventional plasma spray processes incorporate feedstock materials (e.g., powders) with particle sizes on the order of tens to even hundreds of microns, recent work[1] has demonstrated that nanoscale particles can be sprayed and that the nanoscale feature size is retained in the solidified coating. Studying the impact and solidification behavior of nanoscale particles is well suited to molecular dynamics simulations where the fundamental entities are atoms of the constituent materials. Furthermore, the Weber number, or ratio of inertial to interfacial energy, of nanoscale particles is significantly smaller than that for conventional plasma sprayed particles. As such, it is uncertain how well existing engineering relations between the droplet and final splat diameters apply to nanoscale particles. Using MD simulations, we explore the flattening of nanoscale droplets as a function of their impingement velocity and size and rev e al that recently proposed relations[2] between flattening and droplet properties do not readily extend to nanoscale plasma sprayed particles.

[1] - J. Gang, et al.; Scripta Mater., v.48, pp. 1599-1604 (2003).

[2] - C-J. Li, et al.; Surf. & Coat. Technol., v.191, pp. 375-383 (2005).

3:30pm **TS1-7 Tunable Molecular Beams: A New Frontier in Vacuum Deposition of Organic Semiconductors**, ***A. Amassian*** *(aa359@cornell.edu)*, Cornell University, Ithaca **INVITED**

Organic electronics are widely believed to be the most viable platform to manufacture pervasive and disposable electronics on flexible substrates cheaply and with a lesser environmental impact than conventional electronics. The performance of organic electronic devices is closely tied to the packing structure, morphology and interfaces in organic semiconductor thin films, which in turn are intricately linked to molecular processes operant during their assembly. Typically, vacuum sublimation/evaporation is used to fabricate ordered molecular films (e.g., pentacene). While the simplicity of thermal deposition processes makes them attractive, they provide few knobs in way of process control. Supersonic molecular beams have emerged as a way to tailor the assembly of complex molecular building blocks by manipulating the state of incident molecules (e.g., kinetic energy, vibro-rotational states, molecular clustering). Using a powerful combination of in situ real-time synchrotron X-ray scattering, scanning probe microscopy, rate equation modeling and molecular dynamics simulations, we investigate molecular-scale processes of adsorption, assembly, and molecular crystallite formation during supersonic molecular beam deposition of small-molecule thin films of pentacene and diindenoperylene. Our research shows that tunable supersonic molecular beams can tailor the mode and kinetics of growth, resulting in a control of the morphology and packing structure of organic small-molecule semiconductors in unprecedented ways. These changes can influence the field effect mobility of organic semiconductors and offer a pathway to control the performance of organic electronic materials and devices thereof. Our findings indicate that molecular-scale control of interfaces and thin films is achievable; it is contingent upon the development of adequate processing strategies.

4:10pm **TS1-9 A First-Principles Investigation of the Phase Stability of Ti_2AlC Upon Oxygen Incorporation**, ***M. Dahlqvist*** *(madah@ifm.liu.se), B. Alling, I.A. Abrikosov, J. Rosén*, Linköping University, Sweden

The phase stability of Ti_2AlC upon oxygen incorporation has been studied by means of first-principles calculations. Experimental observations of this so called MAX-phase (M = early transition metal, A = A-group element, X = C or N) show that the characteristic nanolaminated structure is retained upon oxygen incorporation, with strong indications of O substituting for C. Therefore, a solid solution of C and O on the carbon sublattice has been simulated by the so called Special Quasi-random Structure (SQS) method. The energy of formation of $Ti_2Al(C_{1-x},O_x)$ has been compared to those of all known competing binary and ternary phases, and has been found favourable for all C to O ratios at the composition of the MAX-phase. A negative isostructural formation enthalpy have also been predicted for $Ti_2Al(C_{1-x},O_x)$. Moreover, the energy of the alloy was lower than what has been calculated for a corresponding mixture of different Ti(C,O)-ternaries and TiAl. Altogether this indicates that a mixture of C and O in the MAX-phase is thermodynamically stable for a wide range of oxygen content, x, which is consistent with experimental observations. Furthermore, the effect of oxygen incorporation on electrical and mechanical properties is discussed, and compared to what is observed experimentally. These results are of importance for an increased fundamental understanding of phase formation and material properties tuned by the incorporation of oxygen.

4:30pm **TS1-10 A Formula for Increased Hardness and/or Ductility in TiN-Based Thin Films**, *D.G. Sangiovanni,* ***V. Chirita*** *(vio@ifm.liu.se), L. Hultman*, Linköping University, Sweden

TiN-based thin films, such as $Ti_{1-x}Al_xN$ and their alloys, are known to have excellent mechanical and thermal properties. In this paper we report the initial results of our ab-initio investigations of four novel ternary compounds, $Ti_{1-x}M_xN$, obtained by alloying TiN with Nb, V, Mo and W, in concentrations of up to 50%. The elastic constants as well as bulk, shear and Young's moduli of these compounds were evaluated using density functional theory calculations within the generalized gradient approximation, and compared with the corresponding properties of TiN and $Ti_{1-x}Al_xN$. Significantly, we find that the addition of all these elements, but primarily Mo and W, results in a substantial increase in bulk modulus values compared to TiN (up to 15%) and $Ti_{1-x}Al_xN$ (up to 30%). At the same time, we observe a dramatic decrease (up to 70%) in the values of C_{44}, and a reversal in the Cauchy pressure, C_{12}-C_{44}, from negative to positive values, results which are indicative of significantly increased ductility in all of these compounds. These trends are in total contrast to what is known for $Ti_{1-x}Al_xN$, which exhibits increased brittleness and lower bulk modulus values as the Al content is increased. We analyze the electronic structure of these compounds and observe a significant shift in the ratio of ionic-to-covalent bonding, as compared to TiN and/or $Ti_{1-x}Al_xN$. This information can be used to understand the mechanisms which promote the transition from strong directional to more metallic bonding, and as it will be shown, is essential in designing compounds with mechanical properties tailored to a variety of applications.

4:50pm **TS1-11 DFT Studies of Graphene Thin Films**, ***C.V. Ciobanu*** *(cciobanu@mines.edu)*, Colorado School of Mines **INVITED**

We present a theory of Moire patterns that are generated by superposing graphene layers on substrates with triangular or rectangular symmetries.

While these quasi-periodic patterns are determined solely by the lattice constants of graphene and substrate and their relative orientation, the structure, interfacial stability, and electronic properties are determined by the atomic-scale interactions. The interactions include the rehybridization of

the carbon atoms into sp3 states, which can induce a bandgap in graphene and thus result in distinct signatures in the STM measurements. We will exemplify with the case of graphene on noble metals and transition-metal oxides, and explore the possibilities and the limitations of engineering the bandgap in graphene by using various substrates.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B1-1

Sputtering Coatings and Technologies
Moderator: C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University

10:40am **B1-1-9 Composition-Property Relations of Reactive Magnetron Sputtered VC_xN_{1-x} Coatings**, ***N. Fateh*** *(nazanin.fateh@mu-leoben.at), C. Mitterer*, University of Leoben, Austria

There is a growing technological interest in the use of transition metal nitrides and carbides due to their unique properties such as high hardness and wear resistance. The aim of this work was to investigate the structural and mechanical properties of VC_xN_{1-x} films with x ranging from 0 to 1 by varying the flow ratio between the reactive gases. The coatings were grown on silicon and high-speed steel substrates using dc magnetron sputtering from V targets in an $Ar/N_2/C_2H_2$ atmosphere at a substrate temperature of 500°C. According to X-ray diffraction analysis, VN and VC coatings exhibit a polycrystalline fcc structure where the VC film shows a (200) preferred orientation. XRD patterns of VC_xN_{1-x} coatings indicate broad peaks positioned between VN and VC giving evidence for the formation of a single-phase VC_xN_{1-x} solid solution with no preferential orientation. Cross-sectional scanning electron microscopy analysis shows a dense columnar structure for all investigated coatings. Nanoindentation yielded a hardness increase with increasing C content reaching a maximum at x ~ 0.4 while for higher carbon content a decrease in hardness was found. Dry sliding ball-on-disc tests against alumina balls in ambient air showed promising friction and wear behaviour, indicating high application potential of coatings within the V-C-N system.

11:00am **B1-1-10 Experimental and Computational Study on the Phase Stability of Cubic Al Containing Transition Metal Nitrides**, ***F. Rovere*** *(rovere@mch.rwth-aachen.de), M. to Baben, S. Ershov,* RWTH Aachen University, Germany*, H.-G. Fuss,* CemeCon AG, Germany*, P.H. Mayrhofer,* Montanuniversität Leoben, Austria*, D. Music, J.M. Schneider*, RWTH Aachen University, Germany

Al-containing cubic transition metal (TM) nitrides, where Al substitutes for TM (i.e., $TM_{1-x}Al_xN$), exhibit remarkable chemical and physical properties. To tailor the performance of such complex multi-component systems for industrial demands the identification of the physical and chemical mechanisms defining the phase stability of cubic $TM_{1-x}Al_xN$ is crucial. As obtained by x-ray diffraction analysis, the maximum AlN (x) solubility in single phase cubic $Ti_{1-x}Al_xN$, $V_{1-x}Al_xN$, and $Cr_{1-x}Al_xN$ magnetron sputtered films is 0.52±0.02, 0.58±0.02, and 0.71±0.02, respectively. During annealing in Ar atmosphere to 1600°C with a heating rate of 20 K/min, precipitation onset of hexagonal AlN is observed at ~900, 1100, and 1200 °C for $Ti_{0.5}Al_{0.5}N$, $V_{0.5}Al_{0.5}N$, and $Cr_{0.5}Al_{0.5}N$, respectively. Consequently, we find that the overall phase stability of the cubic $TM_{1-x}Al_xN$ increase s to higher Al-contents and higher temperatures with increasing TM valence electron concentration (VEC). However, the resistance against nitrogen-release decreases with increasing TM VEC, as confirmed by thermo gravimetric data indicating increased mass loss due to nitrogen release of ~2, 5, and 13 wt% for $Ti_{0.5}Al_{0.5}N$, $V_{0.5}Al_{0.5}N$, and $Cr_{0.5}Al_{0.5}N$, respectively, after annealing to 1600°C. These experimental findings are supported by complementary first principle investigations of the individual contributions of electronic configuration and lattice mismatch on the phase stabilities of ternary cubic $TM_{1-x}Al_xN$ compounds with TM = Sc, Ti, V, Cr, Y, Zr, Nb, and Mo.

11:20am **B1-1-11 Combinatorial Sputter Deposition for Thin Film Materials Selection and - Design**, ***J.M. Schneider*** *(schneider@mch.rwth-aachen.de)*, RWTH Aachen University **INVITED**

Combinatorial techniques are well established in pharmaceutical research for rapid screening and assessment of drugs. During the last decade combinatorial techniques were also used in materials science searching for new materials with attractive magnetic, ferroelectric, and catalytic properties. The assessment of structural materials has also been reported. Combinatorial magnetron sputtering was applied to create materials libraries for the rapid investigation of the correlation between chemical composition, structure, and properties of the as deposited thin films. These correlations are the basis for knowledge based materials selection and - design. Examples discussed in this paper include Cr_2AlC, V_2AlC and Ge-Sb-Te based materials. Furthermore the effect of composition on the stability of CrAlN is presented. To investigate the feasibility of a recently suggested synthesis pathway for MAX phases based on intercalation of sub-stoichiometric transition metal carbides by Al, bi-layers consisting of Ti-C layers with a composition gradient and Al layers have been deposited by combinatorial sputtering. The as deposited bi-layers were then annealed and the formation of a phase mixture containing Ti_2AlC was observed. These examples underline the potential of combinatorial thin film synthesis techniques for the rapid assessment of composition-structure-property relationships enabling efficient materials selection and - design.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B4

Laser and Ion Beam Surface Engineering
Moderator: C. Muratore, Air Force Research Laboratory/UTC, Inc., W. Waldhouser, Johanneum Research

8:00am **B4-1 Mechanical Properties of Pulsed Laser Depositied ZrC Thin Films**, ***V. Craciun*** *(valentin.craciun@inflpr.ro)*, National Insitute for Laser, Plasma and Radiation Physics

ZrC thin films were grown on (100) Si substrates by the pulsed laser deposition (PLD) technique using a high-repetition rate excimer laser working at 40 Hz. The substrate temperature during depositions was set at 300°C and the cooling rate was 5 C/min. X-ray diffraction investigations showed that the films were crystalline. Films deposited under residual vacuum or 2x10E-3 Pa of CH_4 atmosphere exhibited a (200)-axis texture, while those deposited under 2x10E-2 Pa of CH_4 atmosphere were found to be equiaxed. The surface elemental composition of as-deposited films, analyzed by Auger electron spectroscopy (AES), showed the usual high oxygen contamination of carbides. Once the topmost 3-5 nm region was removed, the oxygen concentration rapidly decreased, being around 3-4 % only in bulk. Scanning electron microscopy (SEM) investigations showed a smooth, featureless surface morphology, corroborating the roughness values below 1 nm (rms) obtained from simulations of the x-ray reflectivity (XRR) curves. From the same simulations we also estimated films mass density values of around 6.30-6.65 g/cm^3 and thicknesses that correspond to a deposition rate of around 8.25 nm / min. Nanoindentation results showed a hardness up to 30 Gpa and a reduced elastic modulus of 230 Gpa for the best quality ZrC films deposited under an atmosphere of 2x10E-3 Pa CH_4.

8:20am **B4-2 Excimer and Femtosecond Laser Microstructuring of Superhard Pulsed Laser Deposited ta-C Films - Effects on Friction and Wear Resistance**, ***S. Weissmantel*** *(steffen@htwm.de), G. Reisse, M. Nieher, R. Boettcher, A. Engel, K. Guenther*, University of Applied Sciences Mittweida, Germany

Recently, we developed a novel method for the preparation of several micrometer thick super-hard tetrahedral amorphous carbon (ta-C) films with low internal stress. The method is a combination of excimer laser ablation for film deposition and excimer laser irradiation of as-deposited sub-layers for the reduction of the high stresses. Film hardness measured by a dynamic indentation method was found to be in the range of 55 to 65 GPa and the Young´s modulus in the range of 700 - 900 GPa. Good film adhesion on hard metal and steel was obtained by using tungsten carbide intermediate layers, which are also deposited by excimer laser ablation immediately prior to the ta-C deposition. The ta-C films have been microstructured using (1) excimer laser pulses of 248 nm wavelength and 30 ns pulse duration and (2) femtosecond laser pulses of 775 nm mean wavelength and 130 fs pulse duration. By using the excimer laser, well defined gratings with the width and depth of the grooves as well as the grating spacing in the range of 1.0 to a few micrometer were produced. By using the femtosecond laser, ripple structures with some 0.5 µm spacing, were produced in the films. The influence of different microstructures on wear resistance and friction coefficient measured by using a commercial tribotester under dry and lubricant conditions will be presented. For example, the friction coefficient measured under dry conditions decreases significantly from some 0.12 – 0.15 for unstructured ta-C down to below 0.05 and in some cases even below 0.01 for microstructured ta-C films.

8:40am **B4-3 Control of Femtosecond Pulsed Laser Deposited Thin Films through Plasma Optimization by Temporal Shaping Based on Genetical Algorithm**, *M. Guillermin, C. Liebig,* ***F. Garrelie*** *(garrelie@univ-st-etienne.fr), R. Stoian, A.S. Loir,* Université Jean Monnet, France*, S. Valette,* Ecole Centrale de Lyon, France*, E. Audouard*, Université Jean Monnet, France

Time resolved emission spectroscopy of the plasma induced by a Ti:Sapphire laser (150 fs, 800 nm) irradiation coupled to the temporal shaping of the femtosecond laser pulses is carried out on pure aluminum samples (99.9%) in ultra high vacuum (~10-5 Pa). The emission lines ratio of the laser-induced plasma plume are modified by the use of a numerical adaptive loop with a feedback on the spectrally analyzed optical emission of the plasma. A line associated with Al-II ions is enhanced with respect to two transitions of neutral aluminum in a high fluence regime F=5.8 J/cm2 (~ 10 Fth). The result of the optimization is an optimized temporal shape for laser pulses consisting in series of femtosecond pulses separated by ~ 1 ps and distributed on a Gaussian envelop of 6 ps duration. Simplified temporal shapes of the femtosecond laser pulses are extracted from the optimized shape and their corresponding laser induced plasma emissions are discussed. This study is completed by an optimization of same lines with a lower fluence (low F=1.16 J/cm2 ~ 2 Fth) and an other one on the emission of an Al-III transition with respect to the line of Al-II used during the two first optimizations in the high fluence regime. The plasma optical emissions induced by the optimized and initial temporal shapes of the femtosecond pulses are spectrally analyzed and compared, evidencing modification of the ionization degree of the plasma plume. Thin film depositions are realized using the different temporally optimized and femtosecond distribution. Morphological and structural characterizations of the films are reported.

9:00am **B4-4 Deposition of Nanocomposite DNA Thin Films by Through Thin Film Ablation**, ***S. Fairchild*** *(Steven.Fairchild@wpafb.af.mil),* Air Force Research Laboratory*, P. Murray,* University of Dayton Research Institute*, O. Shenderova,* International Technology Center*, F. Ouchen*, University of Dayton Research Institute

There is increasing need for sensors in applications with lightweight and low power requirements and in thin film transistors with high effective charge carrier mobility for switching and communications. Thin films of biopolymers may be well suited for both applications. We have used a recently-developed, novel form of laser ablation, denoted through thin film ablation (TTFA), to deposit thin films of deoxyribonucleic acid (DNA). In addition, we have incorporated other materials into the ablation process to form nano-composite films. These include nano-diamond, onion like carbon, and metal (Ag, Pt) nanoparticles. We have characterized the films by XPS, Raman spectroscopy, SEM, AFM, and circular dichroism. The capacitance and conductivity of the nano-composite films were also measured. In addition to forming pin-hole free films, TTFA has potential advantages over other more conventional processing techniques such as spin coating. These advantages include controlled depos ition and uniformity of thinner films as well as the ability to create and tailor in-situ DNA-nanoparticle matrices suitable for optimized device properties.

9:20am **B4-5 Broad-Area Ion Beam Systems for Materials Processing: An Overview of Technology and Applications**, ***L.J. Mahoney*** *(lmahoney@veeco.com), D.E. Siegfried, B.A. Knollenberg, V. Kanarov, A.J. Devasahayam*, Veeco Instruments, Inc. **INVITED**

Over the last several decades broad-area ion sources have continued to be used in numerous materials processing applications where tailored ion energy and current flux are required and where material properties and substrate fixturing prohibit the use of other plasma processing techniques. In this presentation we survey existing and emerging ion beam processing applications for both broad-beam gridded ion sources and for gridless end-Hall and anode layer sources (ALS). We first review the use of gridded source systems which provide a well controlled ion energy and ion beam divergence for process substrate widths that range from 5 to 110 cm. These systems are widely used for ion beam milling and deposition of data storage structures and films, patterned LCD devices, mechanically structured optics and well-controlled deposition of high-performance optical filters. Gridless ion sources (end-Hall and ALS) are used when process requirements favor high ion current fluxes and yet allow for relatively broad distributions of ion energy and high ion beam divergence. These systems have been scaled to process widths from 10 to 150 cm wide. Gridless ion source systems are most commonly used in high-rate ion beam assisted deposition of low and high index optical coatings, direct deposition of tribological coatings, surface treatment and nano-texturing for improved adhesion over various materials and growth of anti-reflection coatings.

10:00am **B4-7 Ion Beam Irradiation Effects in Polymers Confined at Nanometer Scale and Nanomaterials**, ***M. Chipara*** *(mchipara@utpa.edu), K. Lozanon, M.D. Chipara, A. Adhikari, M. Mihut*, The University of Texas Pan American **INVITED**

Ion-beam irradiation effects in polymeric materials are rather complex ranging from physical and structural modifications up to chemical modifications. A feature of radiation-induced modifications in polymers and biologic materials is the presence of degradation processes characterized by very long lifetimes (larger than 105 s). This lecture focuses on ion-beam irradiation effects in ultra thin polymeric films and polymeric structures confined at nanometer scale. The lecture includes a brief analysis of the glass transition phenomenon in polymeric materials, an in-depth analysis of the effect of nanometer scale confinement/thickness of the polymeric film, and of the average molecular mass of the polymer on the glass transition temperature. An in-depth discussion aiming to understand from a theoretical standpoint the complex effect of ion beam irradiation on ultrathin polymeric films (with a thickness smaller than 1,000 nm) or confined polymeric structures will conclude the first part of the lecture. Theoretical predictions will be supported by preliminary experimental data reported elsewhere and tentatively connected to additional experimental difficulties (in ion beam lithography) expected to occur when the size of ion beam characteristic features or the thickness of polymeric films are dropped below 102 nm. While there are few theoretical models and experimental data regarding the effect of electron beams on copolymers and or block-copolymers confined at submicron scale (or with a thickness smaller than 1 micron), the lecture will provide a qualitative analysis of the expected radiation-induced modifications in such polymeric nanostructures.

The second part of this lecture will critically review the modifications induced in the radiation behavior of materials due to their nanometer-sized confinement, from both theoretical and experimental standpoints. The analysis will include conducting, semiconducting, and magnetic nanostructures. The lecture will end with a brief analysis of some potential applications of such materials confined at submicron scale for microelectronics, spintronics, space missions, and radiation environments (nuclear plant environment).

Optical Thin Films
Room: Royal Palm 4-6 - Session C2-1

Optical Thin Films for Active Devices and Microstystems
Moderator: M. Cremona, PUC-Rio, T. Miyata, Kanazawa Institute of Technology

8:00am **C2-1-1 Conjugated Polymer Based Organic Solar Cells: State of the Art and Future Challenges**, ***G. Dennier*** *(gdennler@konarka.com), C.J. Brabec*, Konarka Technologies Inc. **INVITED**

Low-cost photovoltaic cells, which are light and flexible, could open up many new applications for solar cells, from self-powered electronic newspapers to self sufficient buildings. Among the various thin film techniques, solution processed organic solar cells have the highest potential for a true low cost technology since production requires only low temperature solution coating with low resolution. Such photovoltaic elements based on donor acceptor composites, being manufactured by printing and coating techniques from reel to reel, are not only scientifically interesting but highly attractive from a cost standpoint. One of the most critical issues to ensure efficient charge separation is the optimization of the interface between donor and acceptor phases, that is, the nanostructure of the composite film. By mixing the two components, an interpenetrating network of donor and acceptor materials is created inducing a 3 dimensional "bulk" interface photovoltaic cells. Morphology studies of these active blends revealed that the solvent used to prepare them plays a critical role for the quality of the "bulk heterojunction". But several other approaches have recently been employed to enhance the structure of the composite and control its nanoscopic arrangement. Although the absorption of the conjugated polymers available now do not perfectly fit the solar emission spectrum, efficiencies higher than 6 % have been reported, paving the way to the production of low-cost, flexible, light-weight solar cells.

8:40am **C2-1-3 Electrochromic Performance of PECVD-Synthesized WO_xC_y Thin Films on Flexible PET/ITO Substrates for Flexible Electrochromic Devices**, ***Y.-S. Lin*** *(yslin@fcu.edu.tw)*, Feng Chia University, Taiwan

Electrochromic performance of WO_xC_y films deposited onto 60 Ω/□ flexible PET (polyethylene terephthalate)/ITO (indium tin oxide) substrates by low temperature plasma-enhanced chemical vapor deposition (PECVD) was investigated. It was proven that extraordinary electrochromic performance was provided when the precursor [tungsten carbonyl, W(CO)6] vapor,

carried by argon gas, was mixed with air gas and synthesized by radio frequency (r.f.) power at room temperature (23 oC). Cyclic voltammetry switching measurements found that only low driving voltages from -1 V to 1 V were needed to provide reversible Li+ ion intercalation and deintercalation. The light modulation with up to 62.3 % of transmittance variance at a wavelength of 650 nm was obtained for 150 cycles of Li+ intercalation and deintercalation in a 0.1 M $LiClO_4$-PC electrolyte.

9:00am **C2-1-4 Flexible IZO Thin Film Transistor with Hybrid Structure Fabricated by Solution Processes and Microwave Heating**, ***H.-C. Cheng*** *(Hua_Chi_Cheng@itri.org.tw), , C.-Y. Tsay,* Feng Chia University, Taiwan*, C.-F. Chen*, Mingdao University, Taiwan

The top gate thin film transistors with organic and inorganic hybrid structure were demonstrated on a flexible polyimide substrate. The amorphous indium zinc oxide and cross-linked poly-4-vinylphenol, used as active channel layer and gate insulator respectively, were fabricated by solution processes and microwave heating under ambient air conditions especially. The aluminum metal was used as the conducting electrodes (source, drain, and gate). These transistors operated at depletion mode showed the optimized device performance with a field-effect mobility as high as 6.9 cm^2/V s, a threshold voltage of 2 V, an on/off current ratio greater than10^6.

9:20am **C2-1-5 Large, Room-Temperature Magnetoresistance in Organic Light-Emitting Diodes**, ***M. Wohlgenannt*** *(markus-wohlgenannt@uiowa.edu), T.D. Nguyen, J. Rybicki, Y. Sheng*, University of Iowa **INVITED**

We report on the extensive characterization of a recently discovered large, room-temperature magnetoresistive effect in organic semiconductor thin film devices[123]. The discovery of this effect came as a surprise, since it has generally been believed that large magnetoresistance at room-temperature requires the presence of ferromagnetic materials. To the best of our knowledge, the mechanism causing this magnetoresistive effect is not currently known with certainty. However, it has been established that it is caused by interactions between paramagnetic species, in particular by the effect of the hyperfine interaction on the spin-selection rules of these interactions. However, the specific nature of the interaction and the involved paramagnetic species is heavily debated in the community. Three different models have been proposed: (i) the electron-hole pair model[4], (ii) the triplet exciton-polaron model[5], and (iii) the bipolaron model[6]. We introduce these models and discuss the bipolaron model in more detail . Experiments that can distinguish between these three directions are therefore needed. We report on several different approaches to such experiments including measurements in unipolar and bipolar organic devices, and spectroscopic experiments that measure the density of singlet and triplet excitons[7].

[1]T. L. Francis, O. Mermer, G. Veeraraghavan, and M. Wohlgenannt, New J. Phys. 6, 185 (2004). [2]O. Mermer, G. Veeraraghavan, T. Francis, and M. Wohlgenannt, Solid State Commun. 134, 631 (2005). [3]J. Kalinowski, M. Cocchi, D. Virgili, P. D. Marco, and V. Fattori, Chem. Phys. Lett. 380, 710 (2003). [4]J. D. Bergeson, V. N. Prigodin, D. M. Lincoln, and A. J. Epstein, Physical Review Letters 100, 067201 (2008). [5]P. Desai, P. Shakya, T. Kreouzis, W. P. Gillin, N. A. Morley, and M. R. J. Gibbs, Phys. Rev. B 75, 094423 (2007). [6]P. A. Bobbert, T. D. Nguyen, F. van Oost, B. Koopmans, and M. Wohlgenannt, Phys. Rev. Lett. 99, 216801 (2007). [7]T. D. Nguyen, J. Rybicki, Y. Sheng, and M. Wohlgenannt, Phys. Rev. B 77, 035210 (2008).}

10:00am **C2-1-7 Improved Multi-layer OLED Architecture Using Evolutionary Genetic Algorithm**, ***M. Cremona*** *(cremona@fis.puc-rio.br),* PUC-Rio, Brazil*, W.Q. Gianini, C. Legnani, K.C. Teixeira,* CeDO - Inmetro, Brazil*, B. Messer, O.P. Vilela Neto, M.A.C. Pacheco*, ICA - DEE - PUC-Rio, Brazil

Organic light emitting diodes (OLED) constitute a new class of emissive devices, which present high efficiency and low voltage operation, among other advantages over current technology. Multilayer architecture (M-OLED) is generally used to optimize these devices, specially overcoming the suppression of light emission due to the exciton recombination near the metal layers. However, improvement in recombination, transport and charge injection can also be achieved by blending electron and transporting layers into the same one. Graded emissive region devices can provide promising results regarding quantum and power efficiency and brightness, as well. The massive number of possible model configurations, however, suggests that a search algorithm would be more suitable for this matter. In this work, multilayer OLED devices were simulated and fabricated using Genetic Algorithms as evolutionary strategy to improve their efficiency. Genetic Algorithms are stochastic algorithms ba sed on genetic inheritance and Darwinian strife to survival. In our simulations, it was assumed a 50nm width graded region, divided into five equally sized layers. The relative concentrations of the materials within each layer were optimized to obtain the lower $V/J^{0.5}$ ratio, where V is the applied voltage and J the current density. The best M-OLED architecture obtained by genetic algorithm presented a $V/J^{0.5}$ ratio four times lower than the value reported in the literature. In order to check the experimental validity of the improved results obtained in the simulations, M-OLEDs with different architectures were fabricated by thermal deposition in high vacuum environment. By using a specific deposition system it was possible to produce multilayer OLEDs with highly controlled deposition rate as low as 0.3Å/s, with an accuracy of 2%. The results of the comparison between simulation and experiments carried out in controlled environment and at room temperature ar e presented and discussed.

10:20am **C2-1-8 The Effects of Post Annealing on Electrochromic Nb-Doped WO_3 Films**, *J.L Huang (jlh888@mail.ncku.edu.tw),* ***C.K. Wang***, National Cheng Kung University, Taiwan*, S.-C. Wang,* Southern Taiwan University, Taiwan*, D.R. Sahu*, National Cheng Kung University, Taiwan

The Nb-doped WO_3 films are deposited by e-beam co-evaporation method using ceramic WO_3 targets and Nb slugs. The films are analyzed by GIXRD, UV/Visible spectrophotometer, electrochemical cyclic voltammetry, XPS and nanoscratch tests. The as-prepared Nb-doped WO_3 film is amorphous structure and low transmission in optical visible region. The XPS results indicate that the valence state of W atoms is +4 rich and that of Nb atoms is +5. The electrochromic behaviors are studied via cyclic voltammetry method for 1000 cycles in Li^+ organic electrolyte show that the enclosing area of I-V curve decreases after 1000 cycles. The optical modulation between coloration and bleach states is also small. The low electrochemical reaction current and low optical modulation is ascribed to the under-stoichiometry of as-prepared Nb-doped WO_3 film and the low cycling stability is related to worse film adhesion. The electrochromic properties of Nb-doped WO_3 films can be improved by post annealing treatment at 100-300 $^{\circ}C$ in oxygen atmosphere. The annealing electrochromic films are transparent in optical visible region, and the optical modulation between coloration/belach states is also improved. There is an increase in electrochemical reaction current, as compared to as-prepared films, after annealing. But the reaction current decreases with increasing of annealing temperature. This is related to Nb-doped WO_3 which is more stoichiometric and the partial crystallization of film after annealing. On the other hand, the higher annealing temperature of Nb-doped WO_3 films possess better cycling stability and higher film adhesion.

10:40am **C2-1-9 Suppressing Coherent Thermal Transport of Photons by Stacking Multi-layer Photonic Crystals**, ***W. T. Lau*** *(wlau@stanford.edu), J.-T. Shen, S. Fan*, Stanford University

We will show that in a multi-layer photonic crystal composed of a lossless dielectric and vacuum, where all heat is coherently transported by photons, the presence of photonic band gaps would suppress the thermal conductance below the corresponding vacuum value. Furthermore, the conductance can be driven to extremely low values by stacking several crystals of different layer thicknesses, such that there are minimal overlappings of the photonic bands, and large impedance mismatches between different crystals.

11:00am **C2-1-10 Effect of Power Density on ITO Thin Films by Facing Targets Sputtering**, ***Y.J. Kim*** *(tennis0828@skku.edu), S.B. Jin, S.I. Kim, Y.S. Choi, I.S. Choi, J.G. Han*, Center for Advanced Plasma Surface Technology, Korea

High quality indium tin oxide (ITO) films with a thickness of 450 nm were deposited on glass substrates at low temperatures using a Facing Targets Sputtering (FTS) system. The crystallinity of the ITO films was investigated using high resolution X-ray diffraction (HRXRD) and field emission scanning electron microscopy (FESEM). The ITO films deposited at a sputter power density < 3^1 were amorphous, whereas they were polycrystalline at power density^2 3^1. This indicates that the crystallinity of ITO films is strongly affected by the power density. The surface morphology of the films was observed by atomic force microscopy (AFM). The resistivity (ρ), carrier concentration (n), and mobility (μ) of the films were measured using Hall-effect. The resistivity of the ITO films depended on the power density. The specific resistivity value reaches a minimum value of 4.26 x^{34} at a power density of 3^1. As the power density increases, the substituted Sn ions in the lattice can produce a free electron carrier to increase the electron carrier concentrations from 2.0 x^5 to 4.6 x^{56}. However, ionized impurity scattering centers can simultaneously trap the free electron carriers and decrease the carrier mobility from 48 to 21^7. The optical properties of the films were evaluated using UV-VIS-NIR spectrophotometry. A maximum transmittance of approximately 81.2 % was achieved in the visible spectral region at a power density of 1^1. The transmittance decreased with increasing power density due to the rough surface morphology caused by the increased optical absorption and surface scattering. $^{1\ 1}$: W/cm^{22}: ≥^{3}: 10^{-44}: Ωcm^5: 10^{206}: cm^{-37}: $cm^2V^{-1}s^{-1}$.

Room: California - Session E1-1

Friction and Wear of Coatings: Lubrication, Surface Effects and Modeling

Moderator: E. Broitman, Carnegie Mellon University, A. Fernandez-Camacho, CSIC-University Sevilla, O.L. Eryilmaz, Argonne National Laboratory

8:00am **E1-1-1 Low-Friction and Wear Mechanisms for Sputtered Ti-C-N Coatings**, ***M. Rebelo de Figueiredo*** *(marisa.figueiredo@unileoben.ac.at), G.A. Fontalvo,* University of Leoben, Austria*, C. Muratore,* Air Force Research Lab/UTC, Inc.*, R. Franz,* University of Leoben, Austria*, A.A. Voevodin,* Air Force Research Lab/University of Dayton*, M. O'Sullivan,* Plansee Composite Materials GmbH, Germany*, M. Lechthaler,* OC Oerlikon Balzers AG, Liechtenstein*, C. Mitterer*, University of Leoben, Austria

It is known that ceramic coatings with substantial amounts of structurally incorporated carbon (C) show low-friction behavior, in particular at intermediate temperatures. However, the mechanisms behind activation, formation and modification of the required free C in the friction contact are still not fully understood. A previous study focused on the investigation of tribological properties of reactive arc-evaporated Ti-C-N coatings. Dry ball-on-disc tests against alumina were performed at room temperature in different atmospheres in order to elucidate the tribo-chemical mechanisms. A low steady-state coefficient of friction of 0.18 was obtained in moist air, which increased to 0.9 under dry and inert conditions. Only for the test in moist air a significant contribution of C-H oscillations was detected, leading to the conclusion that water absorption and subsequent reactions with C must play a crucial role for the formation and activation of low-friction tribolayers. Since arc-evaporated coatings exhibit macroparticles, which are an obstacle for the study of surface phenomena, TiCN was deposited by dc sputtering from a Ti_2CN target in an industrial deposition plant. The coating is stoichiometric ($Ti_{51}C_{33}N_{15}$) with a smooth surface. XRD analyses indicated peaks positioned between TiN and TiC giving evidence for the formation of a single-phase TiCN. The synthesized coatings were tested in a tribometer with controlled environment equipped with in-situ Raman and X-ray photoelectron spectrometers, which provided for the analyses of wear track surfaces and detection of the onset of the lubricious tribofilm formation.

8:20am **E1-1-2 Study of Self-Lubricated Coatings on Radial and Angular Bearings**, ***N.M. Renevier*** *(nrenevier@uclan.ac.uk),* University of Central Lancashire, United Kingdom*, D.G. Teer*, Teer Coatings Limited, United Kingdom

Bearings are widely used in the automotive industry (gear box, wheel, steering power, crankshaft, etc) and play an essential role in the automotive reliability and performance. Extensive studied have been carried previously out on hard coatings and Me-CH coatings[1], this paper will report new results obtained with self-lubricated coatings that have shown exceptional results on other automotive parts. An experimental study was performed to assess the effect of self-lubricated coatings on the behaviour of radial and angular bearings that are designed to support thrust and/or radial loads. The bearings having large contact angles can support heavier thrust loads. They are suitable for a wide range of applications including high speed machine tool spindles or gear boxes. Self-lubricated coatings were deposited onto AISI 52100 bearing steel for laboratory tests and onto bearings for industrial tests. A laboratory pin on disc test rig was used and the tribological propertie s (friction-wear) were measured in various controlled environmental conditions (dry and lubricated). The surfaces were analysed by SEM-EDAX as well as STM/ AFM. The 4 best candidates were retained for the next industrial bearing test. Parameters such as vibration, friction/wear and temperature have been used to monitor bearing performance and the results are reported in the paper.

[1]P. W. Gold and J. Loos Wear, 253(2002)465-472.

8:40am **E1-1-3 Tribology of the Head/Disk Interface**, ***F.E. Talke*** *(ftalke@ucsd.edu)*, Univ. of California, San Diego **INVITED**

Tribology of the Head/disk Interface Frank E. Talke Center for Magnetic Recording Research and Department of Mechanical and Aerospace Engineering University of California, San Diego In presently available hard disk drives, the head/medium spacing has decreased to less than 10 nm. To achieve a recording density of 1 Tb/inch 2, it is anticipated that the head/medium spacing has to decrease to less than 3 nm. As the spacing between slider and disk decreases, increasingly more emphasis has to be given to the design of the head/disk interface to guarantee reliable tribological performance. At a head/medium spacing of 3 nm, contacts are likely to occur between slider and disk during load/unload and continuous flying. Contacts lead to lubricant migration and wear between slider and disk, which, in turn, is likely to cause failure of the disk drive. In this paper, the tribology of the head/disk interface is discussed. First, the materials and surface characteristics of currently used thin film disk are presented, including the characteristics of the carbon overcoat and the lubricant film used. Then, an overview of currently used magnetic recording sliders is given, including a discussion of thermally activated flying height control heads. The tribology and flying characteristics of the head/disk interface at extremely small spacing will be analyzed for discrete track recording and bit patterned media. In the last part of the talk, the tribological requirements for achieving recording densities of 10 Tbits/inch 2 will be discussed, including the possibility of carbonless magnetic disks; heat assisted magnetic recording; improved lubricants and contact recording.

9:20am **E1-1-5 Comparative Study of the Friction Coefficient and Wear Volume with Silane and Carbonitrided Interlayer in 316L Stainless Steel Samples in Hybrid Lubrication Conditions**, ***R.P.C.C. Statuti*** *(romina@las.inpe.br),* INPE - Instituto Nacional de Pesquisas Espaciais, Brazil*, P.A. Radi, L.V Santos,* INPE - Instituto Nacional de Pesquisas Espaciais and Instituto Technologico da Aeronautica - ITA, Brazil*, V.J. Trava-Airoldi*, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil

This paper reports the results of the tribological study that compares the friction coefficient and wear volume of 316L stainless steel samples, coated and no coated with diamond-like carbon (DLC) films, with silicon and carbonitrided interlayers[1] with and without synthetic oil and distilled water interfaces. The DLC films were grown by using a Pulsed DC Plasma Enhanced Chemical Deposition (PE-CVD) technique[2]. The tests were carried out by using a UMT-CETR ball-on-plate tribometer in the reciprocating mode at 10.0mm.s-1 sliding speeds under 2N and 4N applied load. The sphere wear was measured using a very small pin approach, in agreement with the ASTM G99 norm. The results showed that the friction coefficient and wear dependence with the composition of the fluid and the lubrication regime. Also, the film atomic arrangements and graphitization level before and after tribotests were analyzed by Raman scattering spectroscopy. The surface analyses were studied using WYKO Surface Profilers, NT1100.

[1]L.F. Bonetti, G. Capote, L.V. Santos, E.J. Corat And V.J. Trava-Airoldi, Adhesion studies of diamond-like carbon films deposited on ti6al4v substrate with a silicon interlayer, Thin Solid Films, In Press, Available online 20 March 2006

[2]Trava-Airoldi, J. V., Santos, V. L., Bonetti, L. F., Capote, G., Radi, A. P., and Corat, E. J., "Tribological and mechanical properties of DLC film obtained on metal surface by an enhanced and low cost pulsed¬ – DC discharge", (2007).

9:40am **E1-1-6 How Can H Determine the Tribological Behavior in Lubricated Contact of Cu/W – C:H Sputtered Coatings**, ***M. Evaristo*** *(manuel.evaristo@dem.uc.pt),* SEG-CEMUC, University of Coimbra, Portugal*, T. Polcar,* SEG-CEMUC University of Coimbra & Czech Technical University - Prague, Portugal*, A. Cavaleiro*, SEG-CEMUC University of Coimbra, Portugal

C-based coatings are being used in many mechanical contacts without any type of lubrication, however, in many applications the presence of liquid lubricants is required. Tribological studies carried out under oil lubrication conditions for DLC coated contacts showed that the final results are better neither than uncoated materials nor with non-lubricated coated contacts. In this study, carbon coatings were doped with W or Cu, elements with different affinities for C. A Teer Coatings sputtering device with four magnetron was used for the depositions in reactive ($Ar+CH_4$) and non reactive (Ar) atmospheres. W-C:H coatings were deposited by co-sputtering form individual carbon targets and a carbon target with W pellets placed in the preferential erosion zone. The coatings were with (~10 at.%) and without W, with (~25 at.%) and without H contents. Similar procedure was carried out for the Cu-C:H system but in this case co-sputtered was performed using individual C and C targets and a Cu target. Similar Cu and H contents were introduced in the C-based films. In all cases, prior to the deposition a Ti interlayer of approximately 100 nm was deposited for improvement the adhesion of the coatings. All depositions were done with substrates rotation with an angular speed high enough to avoid the formation of a multilayer structure. A negative substrate bias of -50V was applied to the substrates during deposition. After basic characterization of the coatings, the influence of the W and H contents on the tribological behavior in dry and lubricated contacts will be studied. The lubricants are commercial oils with and without additives. Selected worn samples will be further studied by Raman spectroscopy and scanning electron microscopy in order to understanding the sliding mechanisms influencing the friction coefficient and the wear rate.

10:00am **E1-1-7 In Orbit Tribological Tests of a Novel Solid Lubricating Film at the International Space Station**, ***M. Brizuela*** *(mbrizuel@inasmet.es), A. Garcia-Luis,* INASMET-Tecnalia, Spain*, J.I. Oñate,* Fundacion INASMET-Tecnalia, Spain*, I. Garmendia,* INASMET-Tecnalia, Spain*, C. Martinez, R. Fernandez-Sanz*, INTA, Spain **INVITED**

MoS_2 is a broadly accepted solid lubricant for space mechanisms. However, a drawback of MoS_2 is its tribo-sensitivity to atmospheric water vapor which renders the film unsuitable for use under high humidity levels at air conditions. This recommends precautions during ground qualification testing and storage of solid lubricated space mechanisms. Recently, coupling a need of extending space mechanisms' life with advances made in PVD technology, efforts have been made in developing more wear resistant MoS_2 and low friction films capable of both: vacuum and atmospheric application. Alloying the MoS_2 films with metals has been reported by several researchers with varying success. This contribution reviews the work carried out at INASMET-Tecnalia on the development of novel WC alloyed MoSx solid lubricant films. Pin on disk vacuum tribology tests of these films, at 0.75 and 0.95 GPa contact stress, have shown friction coefficients similar to those obtained for conventional MoS_2 films, but with a significant improvement in durability up to about 1 million wear cycles. Tribo-performance of these films under atmospheric conditions, at various humidity levels (40 to 75 % RH) has also been very good, with average friction coefficients as low as 0.07 and a durability as high as 220,000 wear cycles. XPS analyses have shown that the WC-MoS_x films consist mainly of a MoS_x lubricating matrix in which a carbidic wear resistant WC phase is embedded. This combination ensures a low friction behaviour while providing a higher wear resistance. Finally, these novel WC-MoS_x coatings are being tested in orbit at TriboLAB, in the International Space Station (ISS). TriboLAB is a tribology laboratory installed in the EuTEF (European Technology Exposure Facility), fixed to Columbus laboratory from ESA. Tests in progress within the TriboLAB experiment at the ISS have shown that the solid lubricating film maintain very low friction levels of 0.04 and reach a maximum of 1,011,000 wear cycles when tested on a ball on disk geometry at 0.75 Gpa, demonstrating a similar behaviour to that experienced on ground under laboratory conditions.

10:40am **E1-1-9 Low Earth Orbit Space Tribometer**, ***B. Krick,*** University of Florida*, J.G. Jones (john.jones@wpafb.af.mil),* Air Force Research Laboratory/RXBT*, J.K. Lenoff,* Air Force Research Laboratory/RXBN*, A.A. Voevodin,* Air Force Research Lab/University of Dayton*, W.G. Sawyer*, University of Florida

The requirements for increased reliability, minimization of friction losses, and extending lifetime of space deployed and operated mechanisms in both commercial and military systems place stringent demands for solid lubricant solutions. The challenge is multiplied by a need for lubricant survivability over harsh environments both in space (vacuum, atomic oxygen, radiations, particle bombardment, temperature spans) and on ground during system tests, storage and launch. Gold and molybdenum disulfide based coatings are traditional solid lubricants for space with performance data accumulated over decades of their use. However, they do not meet the increased design challenges, hindering new concepts for large space deployed antennas, solar panel structures, fast response re-positioning telecommunication and sensor platforms. A number of new materials have been recently developed which show considerable benefit over traditional lubricants in laboratory tests, but they were not flown in space to create a confidence of their use. To resolve this, a set of 8 portable pin-on-disk sliding tribometers was designed and built to space qualify such candidate materials in the frame of Materials International Space Station Experiment (MISSE) program. In collaboration with personnel from Boeing, NASA, NRL, Air Force Research Laboratory (AFRL), and the University of Florida (UF) the tribometers have been integrated in a Passive Experimental Carrier (PEC) and are awaiting shuttle delivery in October, 2009 to the International Space Station (ISS). Suited up astronauts will place the PEC mounted tribometers outside the ISS where it will remain for more than 12 months, allowing space exposure in front and back of ISS (different degree of atomic oxygen and radiation exposure). The new coatings based on PTFEE composites, adaptive chameleon solid lubricants, and low friction carbon originated from recent fundamental studies in the Extreme Environment Tribology programs supported by the Air Force Office of Scientific Research will be space tested against the base line standards of Au and MoS2 materials. This paper provides information on the MISSE tribological experiment design and compares laboratory data for the selected coatings scheduled for space experiments. Seventh in a series, MISSE 7B will re-utilize existing PEC hardware, refurbished for flight. With the retirement of the shuttle program, this is expected to be the last MISSE project for at least 5 years. The miniature and yet fully controllable design by UF with control and data acquisition developed by AFRL allow the tribometers to also be used in a variety of vacuum chamber tests on the ground, including in-situ SEM, XPS, and Auger surface analyses experiments.

11:00am **E1-1-10 Role of Transfer Phenomena on Tribological Behaviour of of Al-Cu Thin Films**, *M. Ruet,* Ecole Centrale de Lyon, France*, T. Duguet,* Ecole des Mines de Nancy, France*,* ***J. Fontaine*** *(julien.fontaine@ec-lyon.fr),* Ecole Centrale de Lyon, France*, V. Fournee,* Ecole des Mines de Nancy, France*, K. Ito,* Ecole Centrale de Lyon, France*, J. Ledieu,* Ecole des Mines de Nancy, France*, M. Belin*, Ecole Centrale de Lyon, France

Adhesive phenomena are controlling friction and wear of metallic contacts, which are usually poor tribological materials without lubrication. In this study, thin films of Aluminum-Copper alloys have been deposited by magnetron sputtering followed by annealing. Regular alloys (Al, θ-Al_2Cu...) as well as approximant phases (ζ-Al_3Cu_4, γ-Al_4Cu_9) were obtained, as observed by XRD. The tribological behaviour of these different coatings has been evaluated against steel ball on a linear reciprocating tribometer allowing electrical contact measurements.

Two different behaviours can be distinguished. Pure metal and regular alloys exhibit high friction (> 0.3) and evidence of strong adhesive phenomena with massive transfer to the counterface, finally leading to removal of the coating. Approximant phases exhibit lower friction (< 0.3), with less material transfer to the ball, but coating failure arises from brittle fracture when coefficient of friction is higher than about 0.2. Nevertheless, surprisingly low friction (< 0.1) was observed for ζ-Al_4Cu_9 phase, with mild wear and build-up of insulating tribofilm on the ball. When the insulating character of the tribofilm is lost, friction starts to increase, leading to tensile cracks on the coating surface.

The role of transfer phenomena and adhesive interactions between sliding surfaces will be discussed, attempting to account for such significant differences in tribological behaviour of Al-Cu alloys.

Characterization: Linking Synthesis Properties and Microstructure
Room: Sunrise - Session F1-2

Advances in Characterization of Coatings and Thin Films
Moderator: P. Schaaf, TU Ilmenau, M. Baker, University of Surrey

8:00am **F1-2-1 Thin Film Studies by the Application of Complementary Techniques in Real Time**, ***A. Vantomme*** *(andre.vantomme@fys.kuleuven.be), D. Smeets, J. Demeulemeester,* Katholieke University Leuven, Belgium*, C. Detavernier,* Universiteit Gent, Belgium*, C.M. Comrie,* University of Cape Town, South Africa*, C.C. Theron,* iThemba Labs, South Africa*, C. Lavoie*, T.J. Watson Research Center **INVITED**

Advanced coatings and thin films often exhibit a complex composition of several atomic species. The distribution, redistribution, diffusive and reactive properties of the individual species as well as their cooperative motion upon thermal agitation, has a distinct influence on overall thin film properties.

X-ray diffraction (XRD), Rutherford backscattering spectrometry (RBS) and sheet resistance measurements have proven most valuable in the study of thin film properties as a function of thermal treatment. Conventionally, several specimens are subjected to different heat treatments and subsequently analyzed one by one for a complete overview of the response of a thin film to thermal annealing. Because of the discrete character of this approach, however, critical stages for the understanding of the development of thin film properties are easily overlooked. This problem is avoided by determining the specimen properties in real time, i.e. during annealing. Additionally, real-time measurements drastically decrease the workload, as kinetic parameters, for example, can be obtained from a single ramped annealing, while numerous specimens have to be analyzed in the conventional approach.

To illustrate the strength and complementarity of these real-time techniques, we will present some examples of (ternary) silicide and germanide thin film growth by thermal annealing. We will illustrate that (i) the combination of real-time XRD, RBS and sheet resistance measurements allows to investigate the initial (nucleation controlled) as well as the advanced (diffusion controlled) stages of $Co_{1-x}Ni_xSi_2$ formation, (ii) the real-time determination of Pt (re)distribution during Ni(Pt)Si formation reveals the influence of impurities on texture development and (iii) a complete understanding of the Pd-germanide formation process on different Ge surfaces can only be obtained by combining real-time XRD and RBS results.

8:40am **F1-2-3 Control of Hard Protective ZrN Coatings by Modulated IR Radiometry**, *J. Gibkes,* Ruhr-University Bochum, Germany, *F. Vaz, A.C. Fernandes,* ***F. Macedo*** *(fmacedo@fisica.uminho.pt),* Minho University, Portugal, *K. Puchong, J. Pelzl, B.K. Bein*, Ruhr-University Bochum, Germany

In the last three decades, transition metal nitrides (MeN), deposited by vapour deposition techniques, have been implemented as hard coatings on industrial devices and components to increase their lifetime and performance, mainly due to such properties as high hardness, good wear resistance, and chemical stability. In cutting, casting or hot forming, these coatings are submitted to high temperatures giving rise to microstructural changes affecting application-oriented properties. Thus, detailed knowledge about thermal properties and behavior under transient heat loads is fundamental for the successful application of such coatings. Based on Modulated IR Radiometry (MIRR), a non-destructive thermal wave technique appropriate for remote detection in industrial applications@super 1@, and based on an analytical inverse solution of the two-layer thermal wave problem@super 2@, hard protective magnetron sputtered ZrN thin films and coatings have been analyzed with respect to their therma l effusivity, the relevant parameter for time-dependent surface heating processes. For a second objective of this work, namely to control the film thickness by means of the MIRR technique, a set of ZrN films has been prepared keeping constant the deposition parameters, except the deposition time, in order to obtain films of approximately equal composition, structure, and thermal effusivity, but different film thickness. The results obtained by MIRR have been compared with the coating thickness determined by destructive measurements and confirm the reliability of MIRR for this kind of applications on ZrN films and coatings above 1 micrometer.

@super 1@ F. Macedo, A. Gören, A. C. Fernandes, F. Vaz, J. Gibkes, K. H. Junge, J. L. Nzodoum-Fotsing, B. K. Bein, Potential of Modulated IR radiometry for the On-Line Control of Coatings, Plasma Proc. Polym., 4, (2007) S857-64. @paragraph2@super 2@J.L. Nzodoum Fotsing, J. Gibkes, J. Pelzl, B.K. Bein, Extremum method: Inverse solution of the two-layer thermal wave problem, J.Appl.Phys. 98 (2005), 063522.

9:00am **F1-2-4 Cathodoluminescent Properties and Phase Identification in Kappa- and Gamma-Alumina Coatings**, ***G. Pozina*** *(galia@ifm.liu.se), F. Giuliani, L. Hultman,* Linköping University, Sweden, *H. Blomqvist, M. Collin, I. Reineck*, Sandvik Tooling AB, Sweden

We have studied cathodoluminescence (CL) properties of alumina coatings commercially grown on cemented carbide substrates with an intermediate TiCN layer. Typical films of a PVD sputtered gamma-Al_2O_3 or a CVD produced kappa-Al_2O_3 have been characterized and compared before and after annealing. Heat treatment of alumina at temperatures exceeding 900°C results in phase transformation to the α-Al_2O_3. Practical use of CL for phase identification has been demonstrated for both γ- and κ-alumina. All phases have revealed a strong room temperature CL with different peak energies depending on the phase. Spatial resolution of phases on microscopic level is especially demonstrative for the annealed κ-Al_2O_3 coating with mixed alpha- and kappa-alumina. We have also characterized by mean of CL the flank wear cutting tools with the gamma-alumina coating and we have found that the CL spectra for the worn fac e demonstrated a high-energy shift, i.e. towards alpha-phase. We have observed an appearance of a new luminescent band centered at 2.4 eV, which is similar to the emission from a Ti-doped alpha-alumina film, which suggests diffusion of Ti in alumina under thermal conditions at the interface during cutting.

Characterization: Linking Synthesis Properties and Microstructure
Room: Sunrise - Session F3

Characterisation by Electron and Ion Beam Microscopy
Moderator: M. Watanabe, Lawrence Berkeley National Laboratory, J.-G. Wen, University of Illinois

9:20am **F3-5 Characterization of Interfaces Using Scanning Transmission Electron Microscopy Techniques**, ***S. Stemmer*** *(stemmer@mrl.ucsb.edu), J.M. LeBeau*, University of California, Santa Barbara **INVITED**

In the first part of this presentation several examples will be presented that demonstrate the utility of advanced scanning transmission electron microscopy (STEM) techniques in determining the atomic scale structure of interfaces between thin films and substrates. We show that interface structures in dissimilar materials systems can be determined directly, without the need for preconceived structure models, using Z-contrast imaging in STEM. Examples will include interfaces between thin dielectric layers and semiconductors, interfaces in complex oxides and metal/semiconductor interfaces. In the second part of the presentation we will discuss the need for a quantitative understanding of the Z-contrast imaging technique and the need for image simulations in the characterization of more complex structures and interfaces. We will present a practical approach for obtaining quantitative Z-contrast images. We compare experiments and image simulations and show that near perfect agreement is achieved.

10:00am **F3-7 Thermal Stability of PVD Nitride Multilayer Coatings: Effect of Nitrogen Inter-Diffusion**, ***I. Ross*** *(i.ross@shef.ac.uk), W. Rainforth,* University of Sheffield, United Kingdom, *P.Eh. Hovsepian*, Sheffield Hallam University, United Kingdom

High performance nitride based multilayer PVD coatings show excellent potential for the environmental protection of technologically important light alloys such as gamma titanium aluminide. Previous oxidation studies in air have revealed that uncoated gamma titanium aluminide exhibits a strong affinity for nitrogen. In this study, we report the effect of nitrogen inter-diffusion between the coating and substrate and its impact on the coatings thermal stability. We compare two multilayer coating architectures: CrAlYN/CrN and TiAlYN/CrN deposited on gamma titanium aluminide (Ti-45Al-8Nb-0.17C) via a combined unbalanced magnetron / high power impulse magnetron sputtering technique. Samples of the as-deposited coatings were subjected to isothermal thermal annealing at 750°C - 850°C for times up to 1000 hours in ultra pure argon (300mbar). We have applied advanced transmission electron microscopy, scanning transmission electron microscopy and associated analytical technique s to elucidate the structure of the bulk coating and diffusion layers as a function of annealing time. Site specific cross-sections for transmission electron microscopy were prepared by focused ion beam sectioning. In both the coatings studied, extensive inward nitrogen diffusion from the nitride coating to the near surface region of the substrate was observed at 850°C. The resulting interface region was identified as polycrystalline TiN. The subsequent depletion on Al and Nb from this region results in the formation of an Al_3Nb interlayer between the TiN and the unaffected substrate. An approximate parabolic diffusion rate was observed in each instance. The impact of the TiN diffusion layer on the coating performance and the thermal stability is discussed.

10:20am **F3-8 Raman Multi Wavelength and Energy Filtred Tem Study of Diamond-Like Carbon Films Deposited by Femtosecond and Nanosecond Pulsed Laser Ablation**, ***A. Sikora*** *(Aurelien.Sikora@univ-st-etienne.fr),* Université Jean Monnet, France, *B. Vacher,* Ecole Centrale de Lyon, France, *A.S. Loir, F. Garrelie, C. Donnet*, Université Jean Monnet, France

Diamond-Like Carbon (DLC) films deposited by femtosecond and nanosecond pulsed laser ablation have been investigated using multi wavelength Raman spectroscopy. The a-C films have been deposited at room temperature in high vacuum conditions, by ablating graphite target either with a femtosecond pulsed laser (800 nm, 150 fs, ~5 J/cm², femto-DLC), or with a nanosecond pulsed laser (248 nm, 20 ns, ~40 J/cm², nano-DLC). The thin films have been analyzed by Raman spectroscopy at 3 different wavelengths (325, 488 and 633 nm). This allows the study of the value and dispersion of several parameters such as the G peak position, the D peak position and the I(D)/I(G) ratio. Other parameters are also discussed, including the full width at half maximum of the G peak, the T peak position and the I(T)/I(G) ratio. By coupling Raman with Energy Filtred Transmission Electron Microscopy (EFTEM) performed on thin DLC layers prepared by Focused Ion Beam (FIB), the structure of both films are discussed and compared, depending on the laser used during the ablation process. The degree of disorder, the sp^2/sp^3 ratio, the sp^2 clustering and bonding of the two types of thin films are compared. We show that the nano-DLC is more disordered than the femto-DLC. Moreover, the nano-DLC seems to be richer in sp^3 bonding. The D band does not appear for the nano-DLC or should be very low. This means that the sp^2 bonds are more organized as aromatic cycles in the femto-DLC. The comparison of nanosecond and femtosecond pulsed laser ablation on the DLC film structure will thus be highlighted.

10:40am **F3-9 The Stability of Thin Metal Films in Contact with Oxides**, ***W. Kaplan*** *(kaplan@tx.technion.ac.il), M. Baram*, Technion, Israel **INVITED**

Due to the relative interface/surface energies at metal-oxide interfaces, thin metal films on oxide substrates are intrinsically unstable. As a result, thermal annealing will result in the break-up (or dewetting) of thin films, resulting in small droplets if the process is conducted above the melting point of the metal, or in small solid particles if the process is conducted below the melting point of the metal. The process can be kinetically slowed by engineering the relative interface/surface interfaces via equilibrium segregation. Grain boundary grooving of the thin film was thought to be the mechanism for dewetting, but recent experiments have shown that

dewetting initiates via the formation of voids at the metal-oxide interface, via vacancy diffusion.

These issues will be used for the comparison to the concept of stable equilibrium films at Au-sapphire interfaces. The films were formed using a novel experimental approach, in which thin sputtered films of Au were dewetted on a sapphire substrate which was previously partially wetted with drops of anorthite glass. The process resulted in equilibrated metal particles residing on glass drops and on the sapphire substrate adjacent to the glass drops. Aberration corrected transmission electron microscopy and analytical transmission electron microscopy were used to confirm the existence of the ~1nm thick stable films at the Au-sapphire interfaces, and to explore the degree of order within the film.

11:20am **F3-11 Characteristic Analysis of Supporting Precious Metal Nanoparticles on Carbon Nanotubes for Electrocatalysts**, ***M. Chen*** *(chenmi@must.edu.tw), S.-C. Lu, H.-Y. Chen,* Minghsin University of Science and Technology, Taiwan*, H.-W. Yu, C.-M. Chen*, National Chiao-Tung University, Taiwan

This article introduce an efficient method for achieving high dispersion and loading amount of precious metal nanoparticles on carbon nanotubes supports for electrocatalysts. Pt-Ru nanoparticles with uniform diameter about 4.3/2.5 nm were efficiently disperse and supported on carbon nanotubes (CNTs) by microwave assisted polyol method. Temperature and additives are important factors that affects the reduction rate of metals particle from metal chloride salt. In microwave system, metal chloride salts rapidly dissociate and reduce metal catalyst particles then deposite on the CNTs. An addition of additives in metal chloride salts/ ethylene glycol (EG) solution can efficiently disperse catalyst particles homogeneously on CNTs and enhance the adsorption and reduction metal particles. In this article, the mechanism of supporting precious metal nanoparticles on carbon nanotubes supports for electrocatalysts will also Investigate. The characterization analyses will be determined by SEM, TEM, XRD, XPS, and TGA.

11:40am **F3-12 The Effect of Surface Mechanical Attrition Treatment on Phase Transformation in Surface Layer of NiTi Alloy**, ***T. Hu***, *W. Zhang, S. Wu, Y. Xin,* City University of Hong Kong*, C.W Chu,* Southeast University, Taiwan*, R.K.Y. Fu,* Plasma Technology Limited, Hong Kong*, P.K. Chu (paul.chu@cityu.edu.hk),* City University of Hong Kong, China*, K.W. Yeung,* City University of Hong Kong*, J. Lu*, The Hong Kong Polytechnic University, Hong Kong

Surface mechanical attrition treatment (SMAT) is an effective technique to create localized plastic deformation, resulting in grain refinement in the surface layer of metallic materials. In this study, the surface layer of NiTi alloy with martensite as initiate state has been treated with SMAT method. DSC, XRD and TEM were employed to characterize the phase transformation and microstructure features in the deformed surface layer of NiTi alloy. It was found that martensite stabilization and parent B2 phase were introduced after SMAT process. After the occurrence of the first reverse martensitic transformation of B19'→B2, the martensite stabilization vanished. TEM results indicated that the deformed martensite structures and deform-induced dislocations and/or other defects were responsible for the martensite stabilization and parent B2 phase. The decrease of heat of transformation, ΔH, in the first reverse martensitic transformation has also been discussed.

Applications, Manufacturing, and Equipment
Room: Royal Palm 1-3 - Session G1-1

Innovations in Surface Coatings and Treatments
Moderator: B.J. Wei, National Chung Hsing University, M. Rodmar, Sandvik Tooling

8:00am **G1-1-1 High Ionization Plasma for Advanced Coatings: ANew Approach for Coating Deposition on Temperature Sensitive and Complex-Shaped Substrates**, ***J. Alami*** *(jones.alami@sulzer.com), G. Erkens, J. Vetter, J. Mueller, T. Rasa, W. Jung*, Sulzer Metaplas GmbH, Germany

The success of PVD techniques for deposition of hard and dense coatings on Steel tools and components has initiated the interest of many end-users to develop new coatings for other substrate materials such as plastics and polymers. This, however, gives rise to a number of process difficulties as the substrates are often sensitive to high deposition temperatures. Another concern related to PVD is the deposition flux which, because of the line of sight, results in a low thickness homogeneity of the coating. The present work treats these concerns by using the highly ionized plasma for advance coatings (HIPAC) technology in order to deposit highly adhesive, flat and dense CrN coatings in temperature sensitive substrates. HIPAC is a new further development of the HPPMS (high power pulsed magnetron sputtering) deposition technique and is characterized by a highly ionized target material as well as a highly ionized sputtering gas. Plasma analyses show that the high plasma dens ities during the HIPAC process result n a high ionization of the sputtered material as well as a full ionization of the sputtering gas. The energy distribution in the HIPAC plasma shows a smaller energy distribution compared to other pulsed magnetron depositions. This is a unique feature of this deposition technique and is shown to lead to a deposition flux consisting, to a large extent, of low-energy ions. The CrN coatings were deposited on complex-shaped plastic substrates. The substrate temperature on the substrate was lower than 100°C, while the coating analysis showed a dense and flat surface with minimal variation of the coating thickness. HIPAC is shown thus to be an excellent tool for the control of the deposition process energetics and the substrate temperature during growth.

8:20am **G1-1-2 Industrial Scale Deposition of Oxides**, ***L.P. Nielsen*** *(Lars.Pleth.Nielsen@teknologisk.dk), K.P. Almtoft, I.H. Andersen,* Danish Technological Institute, Denmark*, P. Eklund, I. Hansen-Bruhn, B. Thomsen, J. Bottiger*, Aarhus University, Denmark

Industrial scale synthesis of advanced oxides is of outmost importance when bringing small-scale state-of-the-art university research results onto a commercial production platform. In the present content we will illustrate optimized and highly reproducible large-scale reactive sputter deposition of TiO_2, Al_2O_3, and $Ce_{0.9}Gd_{0.1}O_2$ oxides on a commercial CC800/9 CemeCon Sinox unit equipped with the newest HIPIMS technology. For the TiO_2 system it will be shown that reproducible growth of dense, as well as highly porous TiO_2 structures can be synthesised and grown in the anatase as well as the rutile crystal phase. The photocatalytic activity and the mechanical properties (hardness) of the deposited TiO_2 coatings will be addressed.

Bias ramping enabled control of the morphology and density of the alumina coatings, with columnar growth as evidenced by SEM for negative bias of 30 to 65 V. A dense, featureless, insulating alumina was obtained for bias above 70 V. For application in Solid Oxide Fuel Cell (SOFC) the reactive deposition of a $Ce_{0.9}Gd_{0.1}O_2$ diffusing limiting reaction barrier will be shown and the crystal morphology will be discussed as a function of applied growth parameters.

8:40am **G1-1-3 High Power Pulsed Sputtering and Plasma-Assisted Hybrid Processes for Industrial Scale Hard Layer Deposition**, ***H. Klostermann*** *(heidrun.klostermann@fep.fraunhofer.de), F. Fietzke, R. Labitzke, T. Modes, O. Zywitzki*, Fraunhofer FEP, Germany **INVITED**

During the past few years, hard material film development has made advances in tailoring film structure and properties towards sophisticated ends. Multilayer and nanocomposite structures exhibiting superhardness are one example, crystalline layers with single phase composition of high purity another. The benefit of such development for practical application relies not only on basic research but also on appropriate equipment that allows transfer of processes to an industrial scale with guaranteed long-term stability and reproducibility. A key factor in tailoring film composition and structure is the control of particle energies and densities. These are affected by the mode of plasma generation in physical vapor deposition processes. Pulse power supplies with high flexibility for adaptation to different discharge conditions are a powerful tool for the achievement of specific film structures in a variety of coating materials. However, good adhesion is a further prerequisite for an outstanding performance of a coating. The combination of recently developed pulse current supplies and a hollow cathode plasma source open up a new level of coating development in large scale coating equipment. The versatility of these tools will be shown for different hard layer materials. As one example, the deposition of $Zr_{1-x}Nb_xN$ hard protective layers by reactive co-sputtering of zirconium and niobium will be presented. In this process the choice of pulse mode, namely single bipolar or pulse package mode, and pulse times allows tailoring composition and structure of the coatings. Layers with hardness in the range 24 GPa to 35 GPa are deposited without additional substrate bias. Application of substrate bias can increase layer hardness up to 41 GPa, still with good adhesion even for coatings of thickness 5 µm, for which cohesive failure becomes predominant over adhesive failure. As a second example, the deposition of crystalline alumina coatings will be presented. This reactive sputter process poses a special challenge with regard to arc handling and rate optimization. The influence of the pulse mode on the one hand and of additional hollow cathode plasma activation on the other on crystallinity and deposition rate will be presented. Again, a hollow cathode etching process is the key to excellent adhesion of these fully crystalline coatings.

9:20am **G1-1-5 Study of AL Optical Emission in a Reactive Sputtering PVD Process for AlN Deposition**, ***J. Acosta***, *J. Oseguera (joseguer@itesm.mx),* ITESM-CEM, Mexico, *E. Grosse,* Ecole des Mines, France, *O. Salas*, ITESM-CEM

The deposition of AlN films has been analyzed and the optical emission spectrum of reactive sputtering DC plasma process has been obtained for different plasma discharge electrical currents. The film morphology and its chemical composition have been studied by SEM and EDS. Through the behavior of the 396.15 nm peak of the Al, from current variations in the reactive sputtering plasma, a correlation between these Al intensity peak and the plasma electrical current has been obtained. From this study a relation between plasma current control and AlN film morphology is shown.

9:40am **G1-1-6 Nanocomposite PVD Coatings to Prevent Failure of Molten Aluminium Forming Dies**, ***E.A. Torres Miranda*** *(eloy.torres@polito.it), Z. Brytan, D. Ugues,* Politecnico di Torino, Italy, *M. Perucca*, Clean NT Lab - Environment Park S.p.A., Italy

Forming processes of aluminium parts from liquid state are highly diffused. Both traditional (e.g. gravity and die casting) and recently developed techniques (e.g. liquid forging) are continuously growing in terms of productivity and, as a consequence, in severity of stresses applied on the forming tools surfaces. Chemical aggression of molten aluminium, abrasion by hard solid particles and thermal fatigue are typical damages occurring in such operating environment.

In this paper a design process of PVD multilayer coating specifically developed for such complicate operating environment is presented. Nanocomposite $(Cr, Al)_{1-x}N_x/a\text{-}Si_3N_4$coatings were optimally developed and applied on hot a typical working tool steel. Analysis of the coating morphology, adhesion level, thickness, hardness and crystallographic structure are given. Results of alternate immersions in molten aluminium alloy and Taber wear measurements are reported. X-ray diffraction patterns on coatings before and after the immersion cycles in molten aluminum alloy are given so as to identify the corrosion by-products formed on to the surface.

The main results of such study are that the higher is the Al content in mixed nitrides coatings the best is the resistance to the chemical attack and the higher is the hardness and the resistance to abrasion. Furthermore a direct demonstration of efficiency of the multilayer structure in deviating thermal fatigue cracks was recorded by SEM imaging. Finally a relation between the structure and the ultimate properties of the coating is drawn.

10:00am **G1-1-7 Magnetron Sputtering of Free-Standing Aluminium-Scandium Alloys for Use in Cold Forming Processes**, ***I. Eisbrecher*** *(eisbrecher@iwt-bremen.de), H.-R. Stock*, Stiftung Institut fuer Werkstofftechnik, Germany

Thin sheets of aluminium alloys with thicknesses below 30 µm are very interesting for cold forming processes. However, high strength aluminium alloys cannot be rolled down to such a low thickness. Magnetron sputtering of such films followed by separating the substrate from the films can provide an alternative method. To do so, we manufactured a target of aluminium master alloy with a scandium content of 1.8 %. D.C. magnetron sputtering was carried out in a high vacuum equipment with a base pressure below 10^{-4} Pa. As substrate a preoxidized steel foil was used. The maximum temperature during the process was kept below 200 °C measured with a thermocouple mounted behind the substrate. After cooling down the substrate was dissolved in a mixture of H_2SO_4 and H_2O_2. The resulting free-standing sheets had a scandium content of 1.5 % and showed a significant columnar morphology. As these columns decrease formability and cause an unsatisfying tensile strength we applied cold isostatic pressing 400 Mpa for about one hour. The properties of the resulting sheets were examined with analysis by electron microscopy, ultra micro hardness testing, atomic force microscopy and glow discharge optical emission spectrometry.

10:20am **G1-1-8 Effect of Dry Micro-Blasting Conditions on Pvd Films' Properties and on Coated Tools Cutting Performance**, ***K.-D. Bouzakis*** *(bouzakis@eng.auth.gr), G. Skordaris, S. Gerardis, G. Katirtzoglou, S. Makrimallakis, N. Michailidis,* Aristoteles University of Thessaloniki, Greece, *F. Klocke, E. Bouzakis*, Technical University of Aachen, Germany

The conduct of micro-blasting on PVD films has been documented as an efficient method to improve the cutting performance of coated tools. The increase of tool life is affected significantly by the applied conditions during micro-blasting. Therefore the determination of appropriate process conditions during micro-blasting is of crucial importance. In the paper, the effect of various blasting conditions such as of pressure and process duration on the film properties and cutting performance are investigated. With the aid of nanoindentations, the film superficial hardness growth by increasing the micro-blasting pressure and duration was documented. Ball cratering tests at the flank and rake close to the cutting edge and confocal measurements of the tool wedges were conducted to display the change of coating thickness and cutting edge radius respectively induced by micro-blasting. In this way the occurring film distribution along the cutting edge after micro-blasting was det ermined, demonstrating that the substrate was revealed, under certain process conditions. These results were verified by EDX micro-analyses. Furthermore milling investigations were carried out to monitor the cutting performance of coated tools, subjected to films' micro-blasting. The results showed that there are restrictions concerning the increase of pressure and the process duration, with regard to the tool wear. Beyond these limits, tool life deterioration occurs.

10:40am **G1-1-9 Oxidizing Pretreatment of TiN Capping Layer for Al-Based Metallization**, ***A. Yoba*** *(ayoba@us.ibm.com)*, IBM Systems & Technology Group

Aluminum patterning by lithography and reactive ion etching (RIE) is commonly used for integrated circuit fabrication. A common metal stack consists of Ti/Al(Cu)/Ti/TiN, with the Ti and TiN providing redundant conductors for improved reliability. In some cases, residual metal defects are observed after RIE, due to oxidation of Al by the developer[1]. The formation of these defects can be eliminated by oxidizing the TiN surface prior to lithography.

In this study, oxide layer growth on TiN surfaces is characterized. Wafers with an Al(Cu)/TiN metal stack were run through an ozone ash tool, a microwave plasma ash tool (O_2 chemistry), and an RF inductively coupled plasma ash tool (O_2 chemistry). Analysis showed that the surface of the TiN was highly oxidized for all three cases, with an oxidized layer thickness of ~ 4.0 nm. Elemental depth profiling suggests the oxidation penetrates the surface, perhaps along grain boundaries.

[1] E.G. Colgan et al., VMIC Conf. Proc., p. 284, 1994.

11:00am **G1-1-10 Characterization and Antibacterial Properties of TaON–Ag Nanocomposite Thin Film**, ***J.H. Hsieh*** *(jhhsieh@mail.mcut.edu.tw), C.C. Chang*, Mingchi University of Technology, Taiwan

TaON–Ag nanocomposite thin films with Ag nano-particles dispersed in TaON matrix and surface were prepared by reactive co-sputtering of Ta and Ag in the plasma of (O2+ N2)/Ar, followed by rapid thermal annealing (RTA) at 800°C for five minutes. These films were then characterized by a variety of techniques including X-ray diffraction, EDAX, UV-Vis photospectrometer and spectroscopic ellipsometry. In addition, FE-SEM (field-emission scanning electron microscopy) were applied to characterize the surfaced Ag nano-particles on TaON–Ag thin films. Addition of Ag leads to a beneficial change of the film properties. The amorphous structure of the as-deposited TaON films would transform to crystalline state. The results show that annealed TaON-Ag films can have an enhanced antibacterial behavior under the irradiation of visible light due to the synergistic effect of Ag and photocatalytic behavior of TaON.

11:20am **G1-1-11 Structure Effect on the Improvement of the Corrosion Resistance in a 4140 Nitrided Steel by Post-Discharge**, ***A. Medina,*** UMSNH-Instituto de Investigaciones Metalúrgicas, Mexico, *J. Oseguera (joseguer@itesm.mx),* TESM-CEM, Mexico, *H. Carreón,* UMSNH-Instituto de Investigaciones Metalúrgicas, Mexico, *L. Béjar-Gómez*, UMSNH-ngenieria Mecanica, Mexico

The influence of the post-discharge microwave nitriding time on surface properties and corrosion resistance of AISI 4140 low-alloy carbon steel has been investigated. The steel samples were nitrided at 810 K between 5 and 25 minutes. The microstructure and the nitride layer were analyzed by X-ray diffractometry, scanning electron microscopy (SEM) as well as microhardness measurements. The corrosion resistance of untreated and nitrided samples was evaluated in several potentiodynamics tests such as Polarization curves and electrochemical noise. The results showed that as the nitriding time is increased the compound layers changed from a dual phase (ε - $Fe_{2-3}N$ and γ' - Fe_4N) to a monophase γ' - Fe_4N. On the hand, the corrosion resistance and corrosion rate were improved on the samples nitrided during 25 min where the surface is composed by the γ' - Fe_4N phase.

11:40am **G1-1-12 Influence of Residual Stresses on the Growth Kinetics of Boride Layers**, ***M. Ortiz-Domínguez,*** *I. Campos (icampos@ipn.mx), A. Meneses-Amador, J. Martínez-Trinidad*, Instituto Politécnico Nacional, México

This study evaluates the effect of thermal residual stresses on the growth kinetics of Fe@sub 2@B boride layers. The Fe@sub 2@B phase was created by developing the paste boriding thermochemical process on AISI 1018 steels surface. The treatment was carried out at temperatures of 1173, 1223 and 1273 K with 2, 4, 6 and 8 h exposure times for each temperature using a constant 4 mm boron carbide paste thickness (B@sub 4@C) over

the material surface. On the other hand, the XRD method was used to determine the thermal residual stresses profile of the Fe@sub 2@B after the boriding treatment.

In order to determine the growth kinetics of the iron boride layers with the influence of the thermal residual stresses it was necessary to consider, in first instance, the free-stresees boron diffusion coefficient D@sub Fe@sub 2@B@, obtained by the mass balance equation and the boride incubation time, assuming that the boride layers obeys the parabolic growth law@super 1@.

Likewise, according to A. Einstein@super 2@ and L. Darken@super 3@ it is possible to establish a new relationship between thermal residual stresses and boron concentration profile on the growth interfaces, to obtain the boron effective diffusion coefficient D@sub eff@ on the Fe@sub 2@B boride phase@super 4-5@@.

@super 1@ I. Campos-Silva, M. Ortiz-Dom@aa i@nguez, C. VillaVel@aa a@zquez, R. Escobar, N. L@aa o@pez, Defect and Diffusion Forum, 272 (2007), 79.

@super 2@A. Einstein, Ann. Phys., 17 (1905), 549.

@super 3@L. S. Darken, Trans. AIME, 175 (1948), 184.

@super 4@J. C. M. Li, Metall. Trans., 9A (1978), 1353.

@super 5@J. L. Chu, J. Appl. Phys., 75 (1994), 2823.

12:00pm **G1-1-13 Improvement on Low-Temperature Deposited HfO_2 High-Pressure Oxygen/Ozone Treatment**, ***P.-C. Yang*** *(d953050008@student.nsysu.edu.tw), T.-C. Chang*, National Sun Yat-Sen University, Taiwan

In this study, high-pressure oxygen (O2 and O3) technologies were employed originally to effectively improve the properties of low-temperature-deposited metal oxide dielectric films. In this work, 5 nm ultra-thin HfO_2 films were deposited by sputtering method at room temperature. Then, the low temperature high-pressure oxygen treatments at 150 ^{o}C were used to replace the conventional high temperature annealing. According to the XPS analyses, the absorption peaks of Hf-O bonding energies apparently raise and the quantity of oxygen in HfO2 film also increases from XPS measurement. In addition, both the leakage current density of 5nm HfO_2 film can be improved to 10^{-8} A/cm^2 at |Vg| = 3 V. The proposed low-temperature and high pressure O2 or O3 treatment for improving high-k dielectric films is novel and applicable for the future flexible electronics.

New Horizons in Coatings and Thin Films
Room: Tiki Pavilion - Session H3

Surface Engineering of Coatings: Tribo, Bio and Nano-Corrosion Effects

Moderator: M.M. Stack, Strathclyde University, E. Meletis, University of Texas at Arlington

8:00am **H3-1 Potentiodynamic and Potentiostatic Characterization of CVD Alumina Coating for Orthopaedic Implant Wear Reduction**, ***R. Overholser*** *(roverhol@its.jnj.com), E. Gulley, B. Smith*, DePuy, a Johnson & Johnson Company

As orthopaedic implant manufacturers improve longevity of hip and knee prostheses, new coatings are being developed to minimize in-vivo wear against a polyethylene bearing. The rationale for development of a CVD alumina coated Co-28Cr-6Mo implant is driven by the manufacturers desire to combine the wear resistance of an alumina surface with the fracture toughness and damage tolerance of a conventional orthopaedic structural alloy. Physical and mechanical properties that are important in designing a material for use as an implant articular wear surface include scratch resistance, fatigue strength, hardness, fracture toughness, aqueous wettability, biocompatibility and corrosion resistance. In this part of the investigation, a multilayer coating consisting of chemical vapor deposition CVD Al_2O_3 with a TiCN-containing interlayer on a Co-28Cr-6Mo implant substrate is evaluated in terms of corrosion resistance in as-deposited and scratch-damaged conditions using potentiodynamic/potentiostatic testing and scanning electron microscopy. Oxidized Zr-2.5Nb, similar to Oxinium material by orthopaedic implant manufacturer Smith & Nephew, is selected for comparison because of its similar oxide surface/metal substrate composite structure, which consists of a 4-5 μm layer of ZrO_2 grown into a polished Zr-2.5Nb surface by thermal oxidation.

Single sweep potentiodynamic scans performed in non-deaerated Hanks solution at 37^{o}C followed by SEM surface inspection suggest that at potentials up to +2.2V (vs. Ag/AgCl), the as-coated TiCN and Al_2O_3 components exhibit lower corrosion currents and better resistance to dissolution with increasing anodic potential than the Co-28Cr-6Mo substrate. Cyclic scans performed in non-deaerated Hanks solution with 25% bovine serum on scratched multilayer CVD alumina coating to reverse potentials of +1.1V and +2.2V (vs. Ag/AgCl) followed by SEM cross section inspection, exhibit no hysteresis characteristic of crevice corrosion or pitting. Potentiostatic testing of scratched CVD alumina coating and scratched oxidized Zr-2.5Nb at +0.5V and +1.0V (vs. V_{oc}) for 72 hours reveal current decrease to approximately 1 nA, suggesting that spontaneous passivation occurs in the scratched CVD alumina coated Co-28Cr-6Mo at these potentials in these conditions.

8:20am **H3-2 Corrosion Behavior of Magnetron Sputtered Al-Mn Coatings in Neutral Saline Solution**, ***M. Reffass*** *(mreffass@univ-lr.fr), C. Berzioub, C. Rebere,* University of La Rochelle, France*, A. Billard,* University of Technology Belfort Montbeliard, France*, J. Creus*, University of La Rochelle, France

The aim of this work is to develop new Al-Mn sacrificial coatings with good mechanical properties for the protection of steel in saline environments. Al-Mn monolayer coatings with different contents (0-40.94 at %) were deposited on glass slides by magnetron co-sputtering. These coatings were characterized, before and after immersion tests, by X-ray diffraction, scanning electron microscopy (SEM) and electron pr obe microanalysis. The electrochemical measurements in a neutral saline solution showed that the pitting corrosion resistance of Al-Mn coatings increased with increasing Mn content as the pitting corrosion potentials are progressively shifted towards positive values. The immersion tests reveal that for all Mn contents, Al-Mn coatings keep a sacrificial character compared to steel. For Mn content below 25.98 at %, XRD analysis showed the formation of a Mn-rich phase. This structure would be responsible for the high increase of the hardness of Al-Mn coatings and of the stabilisation of their open circuit potentials during the 48 h immersion tests.

8:40am **H3-3 Tribocorrosion Mechanisms: Materials and Coatings for Bio-Implants**, ***M.T. Mathew*** *(mathew_t_mathew@rush.edu),* University Medical Centre (RUMC)*, L.A. Rocha,* Universidade do Minho, Portugal*, A Fischer,* University of Duisburg-Essen, Germany*, M.A. Wimmer*, Rush University Medical Centre (RUMC) **INVITED**

The significant progress made in the area of tribocorrosion, generated another band of studies called as “bio-tribocorrosion”, where the research is focused on the simultaneous tribological and corrosive nature of the bio-implant materials and coatings and resulted degradation mechanisms, basically in the areas of dentistry and orthopedic science. The subsequent effect of partial or complete failure of dental implants or artificial joints (hip and knee joints) on an affected individual and several “unknowns” about the failure mechanisms/modes involved in those failures draw the attention of researchers and medical professionals to the above area. In particular, the complex nature of the bio-tribocorrosion system, due to the combined effect of the corrosive behaviour of the biological fluids, (e.g., saliva and synovial fluid in the joints) and the dynamics of the tribological properties of the material couples in the contact zone still need to be unders tood. Recent advancements, in the area of thin films and coatings with improved material properties as an alternative to current implant materials; also underline the need of a standard and appropriate methodology for bio-tribocorrosion studies, to characterize such new material systems, before the practical usage. Hence, the aim of the current paper is to provide an overview of the recent work in the area of bio-tribocorrosion, considering different materials and coatings. From the bio-medical point of view, a comparison study is also made on the currently employed bio-tribocorrosion test set-up and methodologies by various research laboratories, such as microabrasion-corrosion, fretting-corrosion and sliding wear- corrosion etc. Further, the attempt made by a few researchers in using new techniques to understand the complex bio-tribocorrosion system (e.g., synergism), such as mathematical and software based models and developing maps, are explained and also highlighted the usefulness and limitations of such outcomes. Finally, some thoughts are made on the future perspectives and directions of this attractive research area.

9:20am **H3-5 Functionalization of Ti for Dental Implants Applications**, *A.C. Alves,* ***L.A. Rocha*** *(lrocha@dem.uminho.pt)*, Universidade do Minho, Portugal

Titanium spontaneously forms a compact and protective oxide layer (mainly TiO_2) in the atmospheric environment, which provides anti-corrosive properties. When used as a bone implant, cells will be in direct contact with this surface layer, covered with proteins which nature are also dependent on both the environment and the surfaces properties of the material.

In this study oxide films were created combining two different techniques, anodic treatment followed by a thermal oxidation. By the anodic treatment specific Ca/P ratios were created in the oxide film, while the thermal treatment was aimed at creating a compositional/structural transition across

the film, which may influence the tribological and corrosion behavior of the material. A β-GP + calcium acetate solution was used as electrolyte in the anodic treatment performed at different cell voltages.

The thickness, composition, structure and topography of the films were investigated by FE-SEM, XRD, Raman Spectroscopy and AFM. Electrochemical impedance spectroscopy (EIS) was used before and after anodic treatment in order to characterize and compare the original native oxide film with the anodic oxide film. AFM and SEM analysis demonstrate substantial differences on film topography, both at the micro and nano-scale, with an enhancement of the overall surface roughness. Adhesion of the films was evaluated by the scratch test associated to acoustic signal measurements. Furthermore, the tribocorrosion behavior of the material was evaluated by combining tribological and electrochemical tests (AC and DC).

Results show that a wide range of surface states (from the chemical composition, structural and topographical point of views) can be obtained, some of them showing good performance in terms of biocompatibility. The combination of both treatments strongly enhances the tribocorrosion behavior of the material.

9:40am **H3-6 Characterisation and Evaluation of the Erosion-Corrosion Resistance of HIPIMS-HIPIMS Deposited Nanoscale CrN/NbN Multilayer Coatings**, ***Y.P. Purandare*** *(Y.Purandare@shu.ac.uk), A.P. Ehiasarian,* Sheffield Hallam University, United Kingdom*, M.M. Stack,* Strathclyde University, United Kingdom*, P.Eh. Hovsepian*, Sheffield Hallam University, United Kingdom

Nanoscale CrN/NbN multilayer PVD coatings have proven their mettle in offering combined erosion-corrosion resistance. However growth defects (under-dense structures and droplets) in the coating reduces the ability to offer combined erosion-corrosion resistance. In this work a novel High Power Impulse Magnetron Sputtering (HIPIMS) technique has been utilised to pre-treat substrates and deposit dense nanoscale CrN/NbN PVD coatings (HIPIMS-HIPIMS technique). This new technique, rich with metal ion plasma, deposits very dense structures and offers virtually defect free coatings (free of droplets as observed in Cathodic Arc technique and under-dense structures observed in standard dc sputtering)

Plasma diagnostic studies revealed a high metal ion to gas ion ratio (Cr:Ar) of 3:1 for HIPIMS pre-treatment conditions with the detection of 14%Cr^{+2} and 1%Cr^{+3} ions and $J_s$of 155 $mAcm^{-2}$. For coating deposition conditions the metal ion to gas ion rat io was around 1:4 which is significantly higher compared to DC at 1:30. Characterisation of coating on HSS revealed a high adhesion of L_c75 N (scratch tests), high hardness of 34 GPa and Young's modulus of 381 GPa. Friction coefficient was 0.78 and dry sliding wear resistance was very high with wear coefficients (K_c) values of 1.22 x 10^{-15}. The effect of superior microstructure (droplet defect and intergranular void free) on erosion-corrosion resistance has been evaluated by subjecting the coatings to a slurry impingement (Na_2CO_3+ $NaHCO_3$buffer solution with Al_2O_3particles of size 500-700µm) at 90°with a velocity of 5 ms@supe r-1@. Experiments have been carried at -1000 mV, +300 mV and +700 mV representing 3 different corrosion conditions. The development in the coating technology and in the performance of nanoscale CrN/NbN PVD coatings due to the novel HIPIMS technique is presented by comparing results with coatings deposited by standard UBM technique.

10:00am **H3-7 Nanoscale Properties of In-Situ Formed Tribofilms from Thiophosphate Compounds**, ***P. Aswath*** *(aswath@uta.edu)*, University of Texas at Arlington **INVITED**

In-situ formed tribofilms are the first line of defense in triblogical contacts under boundary lubrication. These tribofilms are formed by decomposition of antiwear additives and their reaction with the underlying substrate. The presence of other additives in oil such as detergents, antioxidants corrosion inhibitors etc. influence the formation of these tribofilms. The level of protection offered by these tribofilms depends not only on their chemistry but also on their mechanical properties. In this study the morphology and chemistry of the tribofilms will be examined using XANES spectroscopy, Scanning electron microscopy, Auger electron microscopy and energy dispersive spectroscopy. In addition, the wear debris formed from these tribological events will be examined by transmission electron microscopy. The mechanical properties of the tribofilms including their resistance to wear and indentation are explored by nanoindentation. The anti-wear additives examined include ZDDP, F-ZDDP and a number of ashless antiwear additives .

10:40am **H3-9 Nanostructured Aluminium Based Coatings Deposited by Electron-Beam Evaporative PVD**, *F. Sanchette, C. Ducros,* CEA de Grenoble, France*, A. Billard,* University of Technology Belfort Montbeliard, France*, C. Rebere,* University of La Rochelle, France*, C. Berziou,* Université de la Rochelle, France*, M. Reffass,* ***J. Creus*** *(jcreus@univ-lr.fr)*, University of La Rochelle, France

Aluminium based alloys were deposited on glass substrates by electron-beam evaporative PVD technique. Alloying elements (Cr, Ti, Gd, Mg) are added in order to improve both mechanical and corrosion behaviours of aluminium. This approach aims to develop sacrificial protection of steel flat products. Because of the components vapour pressures difference, aluminium and alloying elements are co-evaporated from two separate sources. Nanohardness measurements and electro-chemical characterizations in saline solution were carried out. It's shown that aluminium can be mechanically reinforced with preserving sacrificial behaviours. Nanocomposite alloys are compared to nanolayered films in terms of mechanical and corrosion performances.

11:00am **H3-10 Superhydrophilic Textured-Surfaces on Stainless Steel Substrates**, *H. Wang,* ***M. Zou*** *(mzou@uark.edu),* University of Arkansas*, R. Wei*, Southwest Research Institute

Water-assisted flow, such as core-annular flow shows promise for heavy oil transportation. A thin water film lubricates the walls of the pipeline and thereby confining the oil to the central portion of the pipe. Possible oil adhesion to the pipe wall, however, will prevent the effective implement of this method. Coating the internal wall of the pipe with hydrophilic and/or oleophobic material will provide a solution to this problem. Here we report a surface-texturing technique to produce superhydrophilic surface on stainless steel substrates, a commonly used material for oil pipeline. The surface-texturing technique is based on aluminum-induced crystallization (AIC) of amorphous silicon (a-Si), which has been extensively studied to produce large continuous poly-Si grains for electronic and photovoltaic applications, such as thin-film transistors, sensors, solar cells, and display panels. In this study, it was investigated to produce various textured surfaces on stainles s steel substrates. The surface topographies of these textured surfaces were characterized using scanning electron microscope. The effects of amorphous silicon thickness, annealing temperature and time on the resulting surface-topography were investigated. It was found that the surface topography can be controlled by changing the amorphous silicon thickness and the annealing conditions. The wetting property of the textured surfaces were also studied and compared to a smooth stainless steel surface. The results show that the textured surfaces are hydrophilic/superhydrophilic with the smallest water contact angle of less than 5°, while the water contact angle of the smooth stainless steel surface is about 90°.

11:20am **H3-11 Influence of Substrate Microstructures of Squeeze Cast Magnesium Alloy AJ62 on Corrosion Resistance of Plasma Electrolytic Oxidation Coating**, *L. Han (hanb@uwindsor.ca), P. Zhang, J. Ni,* ***X. Nie****, H. Hu*, University of Windsor, Canada

Due to chilling effect, AJ62 magnesium alloy cast by high-pressure squeeze casting exhibits a fine-grained microstructure in the thin skin layer close to the surface of the castings while there is a coarse-grained microstructure in the interior region of the casting. In an attempt to correlate grain sizes to coating corrosion resistance, a layer of ceramic coating was individually deposited on the fine and coarse grained AJ62 magnesium alloys, which were cut from the skin and the interior respectively, in KOH and NaAlO2 solution using plasma electrolytic oxidation (PEO) technology. The potentiodynamic polarization and the electrochemical impedance spectroscopy (EIS) experiments were performed using EC-LAB SP-150 electrochemical apparatus to investigate the corrosion resistances of the coated AJ62 alloys in a salt solution and a commercial automotive engine coolant. The electrochemical behavior of the coating on fine microstructure was compared with that of the coating o n the coarse-grained AJ62 alloy. The surface morphologies of the coatings before and after electrochemical tests were examined using scanning electron microscopy (SEM). The EIS results showed that the substrate microstructures of magnesium alloy AJ62 play a certain role in coating corrosion resistance.

11:40am **H3-12 Corrosion Behavior of Nickel Doped Diamond-Like Carbon Thin Films in NaCl Solution**, *E. Liu (MEJLiu@ntu.edu.sg),* ***N.W. Khun***, Nanyang Technological University, Singapore

Nickel doped diamond-like carbon (DLC:Ni) thin films were deposited on p-Si substrates using a DC magnetron sputtering process by varying DC power applied to a pure Ni target. The bonding structure, surface roughness, scratch resistance of the DLC:Ni films were studied with X-ray photoelectron spectroscopy (XPS), micro-Raman spectroscopy, atomic force microscopy (AFM) and micro-scratch testing in terms of DC power applied to the Ni target. The corrosion performance of the DLC:Ni films was investigated in a 0.6 M NaCl solution by potentiodynamic polarization testing at room temperature. The results indicated that the DLC:Ni films

showed a good passivation behavior in the NaCl solution though the corrosion resistance of the films decreased with increased Ni content in the films. [1] Keywords: Diamond-like carbon; Nickel doping; DC magnetron sputtering; Polarization; NaCl solution.

Room: Sunset - Session TS4

Surface Engineering for Thermal Interfaces, Heat Flow Control and Thermal Energy Management

Moderator: A.A. Voevodin, Air Force Research Laboratory, T. Fisher, Purdue University

8:00am **TS4-1 Thermal Conductivity Control with Thin Films**, ***D.G. Cahill*** *(d-cahill@uiuc.edu)*, University of Illinois Urbana-Champaign **INVITED**

The control and management of high heat fluxes is an increasingly critical issue for a wide range of technologies. Thin film materials have a vital role to play in thermal management at both ends of the spectrum: for example, improved thermal barriers are needed to raise the operating temperatures of heat engines and high thermal conductivity coatings are needed for heat spreading and thermal interfaces. In this talk, I will discuss the basic science and current limits to the thermal conductivity of thin films and the thermal conductance of interfaces. Our experiments are enabled by recent advances in time-domain thermoreflectance (TDTR); although the equipment required for TDTR is still relatively complex, a measurement of the thermal conductivity of thin films is relatively simple and requires a level of experimental effort that is similar to using ellipsometry to measure optical constants. Carbon nanotubes may have the highest thermal conductivity of any material but the superlative thermal properties of nanotubes have not found immediate application in thermal interfaces because of difficulties in making good thermal contact with nanotubes, i.e., the thermal conductance of interfaces with nanotubes is very small. Solids that combine order and disorder in the random stacking of two-dimensional crystalline sheets, so-called "disordered layered crystals" show the lowest thermal conductivity ever observed in a fully dense solid, only a factor of 2 larger than air.

8:40am **TS4-3 Thermal Conductivity Anisotropy in Molybdenum Disulfide Thin Films**, ***R. McLaren****, D.G. Cahill, W. King (wpk@illinois.edu),* University of Illinois Urbana-Champaign*, C. Muratore,* Air Force Research Lab/UTC, Inc.*, J. Hu, A.A. Voevodin*, Air Force Research Laboratory

Hexagonal molybdenum disulfide (MoS_2) is composed of atomic lamellae. Each layer consists of two planes of sulfur atoms and an intermediate plane of molybdenum atoms, bound with covalent bonds. The layers themselves are held together by weaker van der Waal bonds. Such crystallographically anisotropic compounds offer an opportunity to study intrinsic anisotropic thermal conductivity, which may be of interest for heat spreading or thermal isolation within a compositionally homogeneous material. To this end, MoS_2 films with thicknesses of 0.3 – 1 μm were grown by pulsed magnetron sputtering of an MoS_2 target in an argon background. The deposition rate was modulated with the duty factor of the pulsed power applied to the sputter target to control the orientation of the atomic lamellae. Conditions were selected to grow the MoS_2 films with the c-axis of the hexagonal crystals oriented perpendicular (002) or parallel (100) to the substrate, as determined by X-ray diffraction and transmission electron microscopy of film cross-sections. Amorphous films were also examined. Laser thermoreflectance was used to measure the out-of plane thermal conductivity of the films. The thermal conductivity for all of the samples was in the range 0.3-1 W/m-K. The films with (002) orientation of the MoS_2 sheets had out-of-plane thermal conductivity of 0.3-0.6 W/m-K, while films with (100) orientation had thermal conductivity 0.65-1 W/m-K. The thermal conductivity was dependent on the orientation of the films and on film thickness, with thinner films having higher thermal conductivity. Discussion of conductivity changes based on crystal anisotropy, and orientation dependence on thickness will be presented.

9:00am **TS4-4 Fabrication and Characterisation of Nanoscale Heating Sources ('NanoHeaters') for Thermal Nanomanufacturing**, ***C. Rebholz*** *(claus@ucy.ac.cy), K. Fadenberger, I.E. Gunduz, M. Kokonou, C.C. Doumanidis,* University of Cyprus*, T. Ando,* Northeastern University*, J. Chen, Z Gu,* University of Massachusetts Lowell*, P. Wong*, Tufts University

Nanoheaters consist of nanoscale heterostructures of reactive multi-material systems, which upon controlled external ignition release instantaneous, locally singular exothermic heat to a substrate or surrounding material. This presentation overviews progress in fabrication of nanoheater systems based on the exothermic reaction between aluminum and nickel. Multi-layer nanoheater reaction thermodynamics and formation of nickel aluminides have been studied by differential scanning calorimetry tests, contributing to 3D simulations of the temperature and concentration distributions, validated by high-speed and infrared pyrometry experiments on a special fixture. Results on the self propagating reaction of Ni-Al thin film multilayers with various bilayer thicknesses (in the range of 20-100 nm) indicate a two-stage reaction with two different characteristic temperatures. Alternative ignitable bulk nanoheater fabrication was investigated through preheated ultrasonic consolidati on or mechanical alloying of bi-metallic micropowders, and also in the form of Ni-Al nanorods, deposited by electron gun evaporation in nanoporous anodized aluminium oxide membranes. Applications of nanoheaters in rapid thermal processing of semiconductors, self-heated materials, MEMS actuators, thermal nanobatteries, etc. are explored, along with nanoheater fabrication and operation safety.

9:20am **TS4-5 Steady State Microscale Thermal Conductivity Measurement Technique**, ***S. Ganguli*** *(sabyasachi.ganguli@wpafb.af.mil),* University of Dayton Research Institute*, R. Wheeler,* UES Inc.*, A. Roy*, Air Force Research Laboratories/RXBT

The importance of thermal conductivity measurement at microscale has gained importance even more with the development of microelectronics and materials in the submicrometer regime. The knowledge about the local thermal conductivity with lateral resolution of sub-micrometer scale is indispensable. As characteristic length scale of the structures tend to be comparable to the mean free path of the phonon and/or electrons, conventional heat transfer laws (Fourier or continuum laws) fails. A microscale steady state thermal conductivity measurement technique has been developed. The technique consists of an identical pair of heater and platinum RTDs. Platinum heater lines have been synthesized by photolithography on a 400 micrometer sapphire substrate. The sapphire chip has been packaged in a ceramic chip package and wire bonded. A 200 nm platinum film has been deposited on a freshly cleaved rock salt and has been placed in between the two RTDs. The sensitivity of the RTDs has been enhanced by making finer serpentine loops using a focused ion beam machining technique. The platinum film has also been machined to accommodate samples of different lengths ranging from 10 micrometer to 50 micrometer. A Keithley Semiconductor Characterization System, 4200 SCS has been used to supply power to the heaters and also measure the change in resistance in the RTDs. The placement of the sample and also imaging the specimen while undergoing the measurement would be achieved by performing the test inside an SEM. The samples would be welded on the platinum film by the focused ion beam platinum deposition. Thermal conductivity would be measured from the values of the power supplied to the heater and the temperature differential across a known cross sectional area of the sample.

9:40am **TS4-6 Processing and Characterization of Carbon Nanotubes on Diamond/Si Substrates for Thermal Management Applications**, ***C. Varanasi*** *(chakrapani.varanasi@wpafb.af.mil),* University of Dayton Research Institute*, J. Petry,* Air Force Research Laboratory*, J. Burke, L. Brunke,* University of Dayton Research Institute*, J. Bulmer, K. Yost,* Air Force Research Laboratory*, W. Lanter,* Innovative Scientific Solutions, Inc.*, J. Scofield, P. Barnes*, Air Force Research Laboratory

Carbon nanotubes (CNTs) with a high thermal conductivity (~3000 W/m-K) are very attractive for their potential use in a variety of thermal management applications. Diamonds films deposited by chemical vapor deposition with a thermal conductivity >1800 W/m-K are useful to act as heat sinks in high power devices. A structure consisting of both CNTs and diamonds is explored in the present study for thermal management. Diamond films were initially grown on Si wafers at 1100°C in a 5KW microwave plasma enhanced chemical vapor deposition (PECVD) reactor using CH_4 as the carbon precursor. Diamond films on the Si as well as free standing diamond films obtained after etching off the Si substrate were used to subsequently grow CNTs. Substrates were decorated with nanoparticles of various metals (Ni, Ni-Mo, Ni-W-Fe) as catalysts for the CNT nucleation and growth. Both magnetron sputtering and pulsed laser ablation were used to deposit the catalyst nanoparticles. CNTs were then gr own on the catalyst-decorated diamond films using thermal chemical vapor deposition technique. Ni- W- Fe was found to be the most suitable to grow a high density of CNTs on the diamond. Processing parameters to deposit catalyst particles as well as the CNT growth parameters such as pressure, temperature, gas flow rate, etc. were optimized to obtain the high density. Processing details and characterization of the CNT/Diamond nanostructures, including microstructural and Raman spectroscopy data, will be discussed.

10:00am **TS4-7 Strain, Electronic Structure, Phonons and Thermal Properties of ScN:ZrN Superlattices: A First-Principles Study**, ***U.V. Waghmare**** *(waghmare@jncasr.ac.in)*, J Nehru Centre for Advanced Scientific Research, India **INVITED**

We use first-principles density functional theory-based simulations to determine electronic structure and vibrational spectra of ScN:ZrN superlattices, aiming at (a) understanding the role of interfaces and epitaxial strain in controlling their thermal and thermoelectric properties, and (b) developing models that can be employed in determination such properties of superlattices with periodicity of longer length-scales. We first obtain electronic structure and vibrational spectrum of ScN and ZrN crystals with rocksalt structure, and use them in construction of lattice dynamical models and statistical thermodynamic analysis to estimate their specific heat and thermal conductivity. Through comparison of the vibrational and electronic structure of bulk ScN and ZrN with that of their superlattices, we develop a strategy of modeling the interface between them. We use the resultant model in predicting properties of ZrN:ScN super-lattices of different periodicities and epitaxial strain.

* Work done in collaboration with Bivas Saha, Jagaran Acharya and Timothy Sands.

10:40am **TS4-9 GaN-on-Diamond Wafers for HEMT, the Diamond Side of the Story**, ***C. Engdahl*** *(cengdahl@crystallume.com), E. Francis*, Crystallume

HEMT devices fabricated on GaN-on-diamond wafers have demonstrated greatly improved thermal management properties, and have the potential to increase temperature of operation, power and RF performance. GaN-on-Diamond up to 2" in diameter have been fabricated as a thin (~ 100 μm), both freestanding and bonded on silicon wafers.

GaN-on-diamond wafers present unique challenges for device fabrication. First, since diamond wafers are extremely difficult to thin post-process, the initial thickness of the wafer must equal the final desired thickness. Second, the bow of freestanding diamond wafers could be several hundred microns, which can preclude the use of standard photolithography techniques. Third any attempt to improve and adjust any physical properties of diamond should not impact the thermal conductivity of the material negatively.

This paper presents the latest progress in hot filament assisted CVD diamond hardware and process necessary for integration of diamond with GaN. In this investigation, we study the successful demonstration of GaN on diamond and review and compare the results with that of GaN on silicon. The effects of process conditions on the characteristics such as stress and thermal conductivity are described. The as-deposited diamond films were characterized by stress and morphology. The GaN on diamond stacks were evaluated optically and electrically. A steady-state technique was used to measure the thermal conductivity of the deposited and free standing diamond and the GaN/Diamond layers.

11:00am **TS4-10 Diamond V-Grooves for Thermal Management**, ***S.Z. Rotter***, *J.C. Madaleno (jcmadaleno@ua.pt), J. Gracio*, University of Aveiro, Portugal

It is well known that chemical vapor deposited (CVD) diamond thin films have excellent characteristics such as high hardness, high heat conductivity and high chemical stability. This makes diamond the material of choice for high power applications that require an efficient heat transfer capability. With appropriate growth conditions, diamond can be deposited on different substrates and used for a variety of special applications. However, diamond deposition usually takes place at high temperatures (higher than 600ºC), and this limits the materials that can be used as substrates. Silicon has been widely used as a substrate for diamond CVD. The novel nucleation process (NNP) is a method that has been successfully used to deposit diamond on a variety of 3-D substrates with a high conformal capability.

The heat transfer efficiency increases with the area exposed to the liquid cooler or to the air. A wide variety of approaches are used to increase the area of heatsinks at a macro scale, for example. If the same approach is to be used in different electronic scenarios, efficient techniques to increase the exposed area at a micro-scale are needed. In this work, we propose a chemical etching of a flat silicon substrate, followed by the deposition of a diamond film.

The increase of the exposed area of a flat silicon substrate was achieved by a proper chemical etching that allowed the patterning of the surface with nice micron-sized V-grooves. The silicon was then seeded by the NNP and the grooves were successfully coated with a thin diamond film. This approach can be easily used to coat large V-grooved silicon substrates, since the deposition of diamond on silicon is a mature technique. In addition, if diamond is allowed to grow thick enough (a few microns), the silicon can be further removed by etching and the resulting structure is a V-grooved self-standing diamond membrane that can be used for a variety of microscale heatsinks with a very high thermal conductivity. The possible applications of this kind of structure are enormous and a few scenarios are discussed.

11:20am **TS4-11 Modeling of Anisotropic Thermal Transport Behavior in Molybdenum Disulphide (MoS_2)**, ***V. Varshney*** *(vikas.varshney@afmcx.net),* Air Force Research Laboraotry/RXBT, *S. Patnaik,* Air Force Research Laboratory, *A. Roy,* Air Force Research Laboratories/RXBT, *A.A. Voevodin,* Air Force Research Lab/University of Dayton, *B. Farmer*, Air Force Research Laboratory

In this study, we present an investigation of the anisotropic nature of heat transport in Molybdenum Disulphide (MoS_2) crystallites using atomistic molecular dynamics simulations. The motivation for this work originates from recent findings of variation in the in-plane (parallel to the substrate) thermal conductivity of MoS_2 coating depending on the relative orientations of basal planes of MoS_2 crystallites at the surface (ratio of parallel and perpendicular orientation of basal planes). The objective of this modeling work is to provide deeper insight into the thermal transport behavior of MoS_2 . The anisotropic thermal conductivity along the basal plane and normal to the basal plane will be shown followed by a systematic study of thermal conductivity estimates of crystallites with different concentration of parallel and perpendicular basal planes. A model parameter set from literature, based on empirical force-fields, has been developed to reproduce crystal structure o f MoS_2.The parameter set will be further verified and refined to reproduce experimentally known thermal properties of MoS_2 such as CTE, heat capacity etc. Equilibrium molecular dynamics simulations based on Green-Kubo formalism to estimate thermal conductivities of MoS_2 and its dependence on possible orthogonal orientations of basal planes will be presented. This will provide further insight into interfacial thermal resistance at the grain boundaries of the crystallites.

11:40am **TS4-12 Thermal Transport Considerations in Soft Matter Thermoelectrics**, ***D.S. Dudis*** *(douglas.dudis@wpafb.af.mil),* Air Force Research Laboratory/RXBT, *J.B. Ferguson, J.E. Schmidt,* Air Force Research Laboratory, *J.A. Shumake*, University of Dayton Research Institute

The development of new thermoelectric materials based on nanostructured materials requires a complex optimization including minimizing thermal conductivity while maximizing electrical conductivity and thermopower (Seebeck coefficient). Gains the thermoelectric performance, as measured by ZT, have almost all been won by lowering the thermal conductivity. Over the last few years a number of reports of substantial reductions in thermal conductivity have been reported in hard, inorganic materials. In our laboratory our emphasis is developing molecular, soft-matter based thermoelectric materials. We report a review of some concepts in soft-matter thermal conductivity as well as measurements on potential low thermal conductivity building blocks.

12:00pm **TS4-13 Models of Thermal Condutivity of Multilayer Coatings for Cutting Applications**, *L. Braginsky,* ***V. Shklover*** *(valery.shklover@mat.ethz.ch)*, ETH Zürich, Switzerland

The applicability of two earlier elaborated by us models of thermal conductivity to the multilayer coatings for cutting applications is discussed. First is the hopping model, which has been elaborated to consider the phonon hopping between the grains in the nanocrystalline and grained coatings. The model uses the structure parameters of the coating: the grain size, the size of the necks between the grains, coordination number, and cohesion. Transparency of the grains for the phonon hopping is the only adjustable parameter used in the model. Good agreement with experimental data for nanocrystalline metal oxide coatings, where the phonon mean free path is larger or comparable to the grain size has been shown.

The second model was suggested for the porous coatings, where the phonon mean free path is much smaller than the pore size. The model permits us also to estimate the thermal conductivity of the coating using its optical or SEM image. Two-dimensional image of the coating can be used to consider the three-dimensional problem of the heat transport.

Interesting results were obtained for the use of the model, combining both models. This approach is particularly important for modeling thermal conductivity of the coatings where the defects of two or more different scales are present. Temperature deviations in the multilayer coatings due to roughness of the layers or local thermal gradients will be considered. Being large, such deviations can destroy the coating owing to the temperature stress. We discuss also some other aspects related to thermal conductivity of multilayer coatings, which can be used to ensure anisotropic heat spreading in the cutting instruments.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B1-2

Sputtering Coatings and Technologies
Moderator: C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University

1:30pm **B1-2-1 Development of Multi-Element Nitride/Oxide Coatings**, ***F.-S. Shieu*** *(fsshieu@dragon.nchu.edu.tw), D.-C. Tsai, R.-S. Yu*, National Chung Hsing University, Taiwan **INVITED**

Multi-element/high entropy alloys have emerged as a new class of high temperature materials over the years. Unlike conventional alloys with only one or two major components, multi-element alloys usually contain more than three principal elements of equal fraction. One of the distinct characteristics of the high entropy alloys is that the tensile strength of the alloys increases with temperature. The crystal structure of the alloys also exhibits a simple form, such as body-centered cubic, face-centered cubic, and hexagonal closed-packed. Research on the properties and microstructure of multi-element alloy thin films prepared by magnetron sputtering has been carried out intensively in recent years. An overview of the current research on nitride/oxide thin films of the multi-element alloys that contain Al, Si, Ti, V, Cr, Fe, Co, Ni, Cu, Zr, Nb, Mo, and Ta, is given. Attempt is made to correlate among deposition parameters, microstructure, and mechanical properties of the multi-element nitride/oxide films either from literature or our experiments.

2:10pm **B1-2-3 Preferential Sputtering of Oxides and Target Design for Stable Reactive Magnetron Deposition of Oxides**, ***T. Kubart*** *(Tomas.Kubart@angstrom.uu.se), T. Nyberg, S. Berg*, Uppsala University, Sweden

In reactive magnetron sputtering, hysteresis effect often limits the deposition rate and achievable compositions of the coatings. Hysteresis is of particular concern in the deposition of oxides due to the large difference between metal and compound sputtering yields and the high reactivity of oxygen.

In this work we have focused on the understanding of sputtering from different oxides. The overall aim is to avoid hysteresis and increase the deposition rate. High sputtering rate has previously been observed from TiO_x target. This was attributed to the sputter reduction of TiO_2 to lower oxides which have substantially higher sputtering yields. To investigate this effect for other materials, a study of sputtering from metal and oxide targets of Al, Ti, V, Nb, and Ta has been carried out. Sputtering was performed by means of RF magnetron reactive sputtering and x-ray photoelectron spectroscopy (XPS) with ion beam sputtering. The evolution of the surface composition during ion bombardment, as measured by XPS, was related to the corresponding hysteresis behaviour in reactive sputtering. Computer modelling of the sputtering process using the Tridyn code was employed in order to describe the preferential sputtering effects and extract the values of surface binding energy

Preferential sputtering could be described using Tridyn. The results have shown an anomalous behaviour of Ti oxide which differs from all the other materials. Even though the formation of lower oxides and preferential sputtering was observed for other oxides as well, the rate increase was substantially lower. Mixed targets consisting of a metal and its compound have been suggested as an alternative to ceramic targets for high rate stable reactive sputter deposition.

2:30pm **B1-2-4 Low Temperature Deposition of Anatase TiO_2 Film by Dual Magnetron Sputtering**, *J. Sicha,* ***P. Baroch*** *(pbaroch@kfy.zcu.cz), M. Meissner, R. Cerstvy, J. Musil*, University of West Bohemia, Czech Republic

A formation of thin crystalline photoactive TiO_2 films at low deposition temperatures belongs to important topics whose solution would open wide utilization of TiO_2 films on plastics and as a functional barrier coating in microelectronics. To reach this goal, the surface temperature during the deposition must not exceed 150°C and at these low temperatures crystalline films must be formed. For this purpose, a pulsed dual magnetron sputtering system operated at high repetition frequencies and at interruptive deposition mode was proposed. To reduce maximum surface temperature of the substrate and deposited film, the deposition process was periodically interrupted. The deposition process consisted of 1 to 7 deposition cycles with a 60 min pause between each cycle. It was found that the interruption of the deposition process reduced maximum surface temperature, however had no influence on the final crystallographic structure of the TiO_2 films of the same thickness. Likewise, no significant differences in the photocatalytic activity and hydrophilicity of anatase TiO_2 films were observed. The photoactive nanocrystalline TiO_2 anatase film can be deposited at the surface temperature T_{surf} below 130°C and at a relatively high deposition rate a_D ~ 10 nm/min. Results indicate that i) the formation of crystalline TiO_2 films with the anatase phase requires a certain amount of energy to be delivered to the growing film independently on the surface temperature and ii) the energy deliver to the growing TiO_2 film by bombarding ions has a dominant effect on its nanocrystalization. The structure, optical and photocatalytic performance of TiO_2 films are reported in detail.

2:50pm **B1-2-5 Photocatalysis and Phase Transformation of Oxygen-Deficient Carbon-Containing Titania Films**, *C.C. Lin,* ***Y.S. Wang***, *P.W. Chou, M.S. Wong (mswong@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

A series of pure and carbon containing titania films of varying oxygen content were prepared by either sputtering or evaporation by increasing the oxygen flow rate during film deposition. The films were annealed at different temperatures from 300 to 800℃ to study the effect of carbon and oxygen content on the phase transformation of titania films and their photocatalysis. The results indicated that the phase of the as-deposited films with increasing oxygen flow changed from amorphous, rutile, mixed phase of rutile and anatase, and finally anatase. Upon annealing the films deposited at higher oxygen flow maintained anatase with improved crystallinity up to 800℃, while the films deposited at lower oxygen flow transform to rutile phase at low temperature at 300℃ and maintained rutile phase with improved crystallinity with increasing temperature. The photocatalytic performance is strongly related to the content and quality of anatase phase in the films. The more conte nt and the better crystallinity of anatase in the films, the better the phtocatalytic degradation of methylene blue.

3:10pm **B1-2-6 Nucleation and Crystal Growth Behavior of α-Alumina by Reactive Magnetron Sputtering**, ***H. Tamagaki*** *(tamagaki.hiroshi@kobelco.com), Y. Ikari,* Kobe Steel, Ltd., Japan, *H. Fujii, K. Yamamoto,* Kobe Steel Ltd., Japan, *T. Kohara*, Kohara Ltd., Japan

Alumina as top-coat of hard coatings is considered as the best material for oxidation-resistance and heat-resistance layer for cutting tools. Particularly, α-alumina with the corundum structure is considered as the best because of its thermal stability compared with the other crystal structures of alumina. Previously, the authors demonstrated the successful deposition of α-Alumina by a reactive magnetron sputtering at temperature around 750°C in a production scale PVD system, by using oxidization layer of CrN or TiAlN as under layer of Alumina.

In this work, the nucleation and crystal growth behaviors of PVD-Alumina were studies. The films were deposited form the metallic aluminum target in Ar and O_2 mixture at the substrate temperature from 600 to 750°C on various kind of substrates and under layer, including Si wafers, Cemented Carbide, CrN, TiAlN etc. The proper process windows for the successful growth of α crystallin e was found on the temperature as well as the deposition rate. Under the optimized conditions around 750°C, the formation of single phase α-Alumina with good crystallinity was confirmed on CrN under layer and some other substrates. However, even at the identical deposition conditions, only γ phase was found on some of the substrates such as Si wafers. In the paper, the effects of the substrates and under layers on the crystal growth of α phase will be demonstrated, and possible growth mechanism of α-Alumina will be discussed.

3:30pm **B1-2-7 Low-Temperature Deposition of Alpha Alumina by Physical Vapor Deposition without a Chrome Template Layer**, ***A. Barito*** *(abarito@uark.edu),* University of Arkansas, *H.H. Abu-Safe,* Lebanese American University, Lebanon, *D.E. Spearot, M.H. Gordon*, University of Arkansas

Alpha alumina has many chemical and mechanical properties that make it an ideal candidate for cutting tool coatings and bio-medical applications. In previous work, we have deposited low-temperature (480C) alpha-alumina with a chrome template layer, and low-temperature (480C) mixed-phase alumina coatings without a chrome template layer using an AC inverted cylindrical magnetron sputtering system. SEM analysis indicated films that were predominantly alpha-phased, and TEM demonstrated pure alpha phase in some samples.

In this work we report on the systematic deposition of alumina films as a function of power (4-6kW), pressure (2-8mTorr), substrate bias (DC and pulsed), and oxygen partial pressure (35-75%). 39 total runs were performed and several substrates were used (glass, silicon, stainless steel,

and a titanium alloy). Analysis of each film included XRD, SEM and AFM. Preliminary results indicate that pure alpha films are favorable at 5-6kW, 50% oxygen partia l pressure, pulsed bias, and 2mTorr. In general, pure aluminum films are observed at lower oxygen partial pressures. At lower powers, little alpha alumina is observed as the energy at the substrate is too low. Indicating noticeable pressure dependence, films deposited at lower pressure (2mTorr) tend to exhibit phases fairly independent of power and oxygen partial pressure. Initial evidence suggests that the deposited films have a weak dependence on substrate bias.

3:50pm **B1-2-8 RF Sputtered Piezoelectric Zinc Oxide Layers for Microfluidic Applications**, ***K.F. Wätje*** *(kerstin.waetje@physik.uni-augsburg.de), A. Wixforth*, University of Augsburg, Germany

Surface acoustic waves (SAWs) on piezoelectric substrates find widespread applications for high frequency signal processing applications. Recently, SAW induced acoustic streaming for agitation and actuation of smallest volumes of fluids has attracted considerable attention in the field of microfluidics. Apart from single crystal substrates like $LiNbO_3$, low-cost, high quality alternatives are required for on-chip biosensor applications.

Here, we investigate high performance piezoelectric ZnO films produced by radio frequency magnetron sputtering on different host substrates.

We use Rutherford backscattering spectroscopy (RBS) and elastic recoil detection analysis (ERDA) to analyze the composition of the ZnO layers. Structural characterization by X-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) demonstrates preferential c-axis orientation and an excellent microstructure of our ZnO thin fil ms.

The influence of process conditions such as pressure and temperature are discussed as well as the applicability of different crystalline and non-crystalline substrates considering the advantages and disadvantages for the envisioned applications. We find high coupling factors (close to single crystals). These allow for efficient mixing as required for any microfluidic application. The effects of post annealing, film thickness, roughness, residual stress and resistivity are studied in terms of these properties.

4:10pm **B1-2-9 Phase Transition and Mechanical Properties of Zr(N,O) Thin Films on AISI 304 Stainless Steel**, ***J.H. Huang*** *(jhhuang@mx.nthu.edu.tw), T.C. Lin, G.P. Yu*, National Tsing Hua University at Hsinchu, Taiwan

Nanocrystalline Zr(N,O) thin films were deposited on 304 stainless steel substrates using unbalanced magnetron sputtering (UBMS) system. The purpose of this study was to investigate the phase transition and the accompanied mechanical properties of the Zr(N,O) thin films deposited on AISI 304 stainless steel by varying oxygen flow rate (ranging from 0 to 2 sccm). The oxygen contents of the thin films determined using X-ray Photoelectron Spectroscopy (XPS) increased significantly with increasing oxygen flow rate. From the observation of X-ray Diffraction (XRD), the dominant phases in the films were in a sequence of fcc-ZrN, c-Zr_2ON_2, and m-ZrO_2 with increasing oxygen flow rate. The characteristics of the films can be divided into three zones: Zone I (ZrN), Zone II (Zr_2ON_2) and Zone III (m-ZrO_2). Modified XRD $\sin^2\psi$ method was used to respectively measure the residual stresses of ZrN, Zr_2ON_2 and m-ZrO_2 phases. The residual stress in ZrN was relieved as the oxygen content increased. Zr_2ON_2 and m-ZrO_2 were found to be low residual stress phases. The hardness of the Zr(N,O) films decreased with increasing the oxygen content due to the formation of the soft oxide phase. AES analysis showed that there existed a ZrO_2 interlayer between the Zr(N,O) film and the substrate for those samples deposited using O_2/N_2 mixing gas. Contact angle was used as an index to assess the wettability of the film on substrate, which was associated with adhesion of the film. The contact angle was calculated from the measured surface energies of the related materials. The results indicated the poor wettability between ZrO_2 and SS304, which was correlated to the film spallation in salt spray tests.

4:30pm **B1-2-10 Preparation of Artificial $BiFeO_3/SrTiO_3$ Superlattices by rf Sputtering**, ***S.-J. Chiu***, *G.P. Yu, J.H. Huang,* National Tsing Hua University, Taiwan, *H.-Y. Lee (hylee@nsrrc.org.tw)*, National Synchrotron Radiation Research Center, Taiwan

Artificial superlattice structures consisting of multiferroic $BiFeO_3$ and paraelectric $SrTiO_3$ sublayers have been successfully grown on a $SrTiO_3$ substrate with rf magnetron sputtering. X-ray reflectivity and high-resolution diffraction measurements were employed to characterize the microstructure of these films. A symmetric sublayer structure, with the designed thickness of a sublayer varied in a range from 2.5 nm to 10 nm and the total thickness of the superlattices was fixed at 100 nm, was adopted. By changing the stacking periodicity, we introduced the several of interfacial strain in superlattice. The effect of strain resulted from difference of lattice constant of $BiFeO_3/SrTiO_3$ was investigated on the microstructures. The X-ray reflectivity curve showed the formation of superlattice structure and increasing of roughness with stacking period decreased, which are consisted with the measurements of atomic force microscopy (AFM). Increasing surface roughness of the superlattice may be resulted from the grain growth of $BiFeO_3/SrTiO_3$ superlattice by increase of periodic rotation of targets during deposition. The epitaxial relation between the $BiFeO_3$ and $SrTiO_3$ layers in the superlattice is examined by the in-plane orientation with respect to the major axes of the $SrTiO_3$ substrate. The results clearly show the four-fold symmetry of $BiFeO_3$ sublayer, and it confirmed the pseudocubic structure of the $BiFeO_3$. The analysis of crystal truncation rod spectra showed higher elongation of $BiFeO_3$ sublayer along [0 0 L] by the heteroepitaxial strain when stacking period of $BiFeO_3/SrTiO_3$ superlattice increased. The ferroelectric properties of $BiFeO_3/SrTiO_3$ superlattice could be improved by adjusting deposition conditions.

4:50pm **B1-2-11 PVD Module for High Quality AC Reactive Sputtering of Oxide and Nitride Thin Films**, ***V.V. Felmetsger*** *(vfelmetsger@tegal.com), P.N. Laptev, S.M. Tanner*, Tegal Corporation

Various electronics and surface engineering applications require deposition of thin dielectric films having certain functional characteristics. In recent years, remarkable progress has been achieved in developing new techniques for reactive sputtering. In this paper, we discuss design features and application capabilities of the S-Gun magnetron for ac (40 kHz) reactive sputtering.

Due to its dual target arrangement, the ac powered S-Gun is uniquely able to realize reactive sputtering processes free of parasitic arcing and disappearing anode effects thus enabling formation of high quality silicon oxide, aluminum oxide, aluminum nitride and other dielectric films with deposition rates up to 100 nm/min. The process module is equipped with a stress adjustment unit to reduce compressive stress in the films by controllably suppressing the flux of charged particles to the substrate by redistributing the discharge current between the targets and the internal shields of the magnetron. If film has excessive tensile stress, deposition with rf substrate bias allows reducing stress to near zero level.

The key advantages of the deposition technique we developed are the following:

- Reliable, arcless reactive sputtering at ambient and elevated temperatures;

- Precise adjustment and high run to run repeatability of the film thickness and uniformity;

- Formation of well-textured and, if required, crystal oriented films;

- Enhancement of the film adhesion using energy of accelerated neutrals and ion-assisted condensation;

- Effective stress control to produce films with either compressive, or near-zero, or tensile stresses.

In the paper, we also discuss some examples of the S-Gun reactive sputter technologies, particularly, deposition of highly c-axis oriented AlN films with low intrinsic stress at relatively low temperature (below 300°C).

5:10pm **B1-2-12 Adhesion Improvement of Reactive RF-Sputtered Zirconium Nitride Films on WC Substrate**, ***A. Rizzo*** *(antonella.rizzo@brindisi.enea.it), M.A. Signore, L. Mirenghi, L. Tapfer, D. Valerini,* ENEA, UTS FIM, CR Brindisi, Italy, *U. Galietti,* DIMEG – Politecnico of Bari, Italy, *D. Altamura*, University of Lecce, Italy

Thin films and coatings are used for a variety of purposes over a large variety of applications. According to the projected function, adequate adhesion to the underlying substrate is of fundamental importance. In this work the attention is focused on the adhesion of sputtered ZrN films on WC–wt.10 % Co substrate tuning different parameters: the assistance bias applied during the growth of the nitride (in the range −5 to 20 V), the implanted N ions flux during substrate surface treatment, the addition of TiN layers in different coating configurations. The structure and morphology of the obtained coatings were analyzed using atomic force microscopy (AFM) and x-ray diffraction (XRD) while elemental composition was deduced from x-ray photoelectron spectroscopy (XPS). It was found that the substrate bias strongly influenced the residual oxygen (impurities) incorporation that decreases until a trivial value for positive bias. A structural switch from (200) to (111) has be en detected increasing the bias voltage. The gradient composition layer formed by implantation improves the bond in the coating-substrate interface. The presence of TiN layers in the coating structure improves the adhesion decreasing the thermal and the intrinsic strain. These investigated properties have been correlated to the films adhesion studied by scratch test and to the wear resistance carried out by pin-on-disc tribometer. The maximum critical load is about 70 N and the wear rate is about 2.23*10-6mm3/Nm.

Optical Thin Films
Room: Royal Palm 4-6 - Session C2-2

Optical Thin Films for Active Devices and Microstystems
Moderator: M. Cremona, PUC-Rio, T. Miyata, Kanazawa Institute of Technology

1:30pm **C2-2-1 ZnO:Al Transparent Conducting Oxide Films of Very High Quality Synthesized by Filtered Cathodic Arc Deposition**, ***A. Anders*** *(aanders@lbl.gov),* Lawrence Berkeley National Laboratory, *S. Lim,* University of Sydney, Australia, *K.M. Yu,* Lawrence Berkeley National Laboratory, *J. Andersson,* Ångstrom Laboratory, Sweden, *J. Rosén,* Linköping University, Sweden, *M. McFarland, J. Brown*, Acree Technologies Inc.

Aluminum-doped zinc oxide, ZnO:Al, is a well-known n-type transparent conducting oxide with great potential in a number of applications currently dominated by indium tin oxide (ITO). ITO replacement projects are driven by the high cost of indium, and ZnO:Al may serve well under certain conditions. Additionally, ZnO is a material that offers the possibility of transparent electronics by synthesizing n- and p-type material. Here we focus on ZnO:Al deposited by filtered cathodic arc deposition as an interesting ITO alternative to magnetron sputtering because we can utilize ion-assisted deposition while avoiding the damage to films caused by energetic negative oxygen ions that are typical for reactive magnetron sputtering. The quality of the films strongly depends on the deposition temperature while only marginal improvements are obtained with post-deposition annealing. To date, our best films have conductivities in the low 10^{-4} Ωcm range with a transmittance better than 85% in the visible part of the spectrum. We will report about the relationships of deposition conditions and structural, optical, and electronic properties.

This work was supported in part by the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

1:50pm **C2-2-2 Influence of Thermal Annealing on Electrical and Optical Properties of Ga-Doped ZnO Thin Films**, ***H. Makino*** *(makino.hisao@kochi-tech.ac.jp), N. Yamamoto, A. Miyake, T. Yamada,* Kochi University of Technology, Japan, *Y. Hirashima, H. Iwaoka, T. Itoh,* Geomatec Co., Ltd., Japan, *H. Hokari, H. Aoki,* Casio Computer Co., Ltd., Japan, *T. Yamamoto*, Kochi University of Technology, Japan

Ga-doped ZnO (GZO) is one of potential candidates as substitute for indium tin oxide. In practical applications of GZO films as transparent electrode in liquid crystal display, thermal stability of GZO films is required. In this study, we report influence of thermal annealing on electrical and optical properties of GZO films.

150 nm thick GZO films were deposited on alkali-free glass substrates by ion plating (IP), direct current magnetron sputtering (DCMS), and radio frequency power superimposed direct current magnetron sputtering (RF/DCMS) at a substrate temperature of 150 ºC. Electrical resistivity of as-deposited films by IP, DCMS, and RF/DCMS were 2.8, 8.0, and 3.7 μm, respectively. The films were annealed in air or in nitrogen gas atmosphere for 30 min at annealing temperatures, T_{an}, between 200 and 450ºC. In the case of GZO films deposited by IP, the resistivity was stable up to 300ºC in air and up to 350ºC in nitrogen gas atmosphere, respectively. The resistivity increased with increasing the T_{an} beyond the critical temperature. The critical temperatures of GZO films deposited by DCMS and RF/DCMS were lower than that of IP.

Optical transmission and reflection spectra were measured by a spectrophotometer. The optical spectra also showed systematic change depending on the T_{an}. The optical spectra were simulated by a conventional Drude model, and optical mobility, μ_{opt}, and carrier concentration, N_{opt}, were estimated from the model fitting. The N_{opt} showed overall good correspondence with carrier concentration characterized by Hall measurements. The μ_{opt} was also nearly identical with the Hall mobility, μ_{Hall}, up to the critical temperature. However, the μ_{opt} and the μ_{Hall} showed different behavior at the T_{an} higher than the critical temperature. The μ_{opt} increased with increasing the T_{an}, while the μ_{Hall} decreased with increasing the T_{an}. The difference suggests different scattering mechanism governing the mobility. The increase in μ_{opt} suggests a decrease of some scattering centers inside grains. On the other hand, grain boundary scattering possibly contribute the decrease in μ_{Hall}.

2:10pm **C2-2-3 Attractive Potential Substitute for ITO in Flat Panel Display**, ***K. Utsumi*** *(k_utumi@tosoh.co.jp)*, Tosoh Corporation, Japan **INVITED**

Recently, markets of LCD (Liquid Crystal Display) and solar cell are expanding rapidly. In these devices, TCO (Transparent Conductive Oxide) are used. ITO (Tin doped Indium Oxide) is the most representative material of TCO. However, there are problems regarding In resources and stability of supply. From these backgrounds, demand of alternative ITO is increasing.

ITO films are prepared by sputtering, vacuum evaporation, spray, and CVD. Among these methods, sputtering is widely used for production line of LCD and solar cell because of its advantageous in good uniformity for large area deposition. So, material and deposition method of the alternative ITO must be used in present production line. ZnO based materials are one of candidate materials of alternative ITO because Zn is abundance, low price and poisonous less material.

In this report, characters of aluminum doped ZnO (AZO) film prepared by dc magnetron sputtering on glass substrate will be discussed.

Resisitivity of AZO film prepared by dc sputtering is over 1000 μmΩcm because of lower crystallinity by the bombardment of high energy particle such as neutral Ar and negative ionized oxygen. The resistivity of AZO film strongly depend on the crystallinity of the film. Therefore, to improve the crystallinity of the film, optimization of Al content was performed. As the results, the resistivity of ZAO film was reduced under 500 μmΩcm (thickness =150nm for color filter of LCD) and under 1000 μmΩcm (thickness = 50nm for TFT of LCD). These values of resisitivity are adaptive for LCD.

It is well known that ZnO based materials are very week for heat and humidity. These are significant problems for reliability of the products.. By the addition of the third element, these problems were solved.

2:50pm **C2-2-5 Thermopower and Optical Studies on Undoped and Manganese Doped Indium Tin Oxide Films**, ***S.R Kumar***, *V. Damodara Das, S. Kasiviswanathan (kasi@iitm.ac.in)*, Indian Institute of Technology Madras, India

Thermopower measurements in the range of 300-650 K along with optical studies were performed on indium tin oxide (ITO) and manganese doped indium tin oxide (Mn:ITO) films grown by DC reactive sputtering. The role of oxygen vacancies and manganese in determining the electrical properties and band gap of the films were examined by annealing the films in argon and oxygen and by the analysis of the thermopower and optical data. The Seebeck coefficient (S) of the films is negative and its magnitude increases linearly with temperature suggesting a near free-electron like behaviour, but decreases drastically beyond a temperature for all the films. This has been explained by considering the out-diffusion of oxygen from the films, upon argon annealing. The optical transparency of the films is found to be more than 90% in the visible region. A dielectric function model combining the Forouhi-Bloomer model for crystalline materials and Drude model was used to fit the optical transm ittance of the films. The complex dielectric function, thickness, plasma frequency and damping constant of the films were obtained from which the electrical properties were extracted. Argon annealed ITO film is found to have the highest carrier concentration (8.6×10^{20} /cm^3) while the highest band gap (4.41 eV) is observed in Mn:ITO film annealed in argon. The results indicate that oxygen vacancies as well as presence of manganese affect the optical and electrical properties of the films. The observed changes in band gap could be explained on the basis of Burstein-Moss widening along with electron-electron and electron-impurity scattering. Comparison of results from optical and thermopower studies indicate that ionized impurity scattering is the predominant scattering process in these films.

3:10pm **C2-2-6 Reactively Co-Sputtering Transparent Conducting Oxide Films of Nb-Doped Titania**, *K.H. Hong,* ***P.W. Lee***, *W.C. Hsu, H.C. Chang, M.S. Wong (mswong@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

Niobium-doped titania (TNO) films were deposited on glass substrates by reactive co-sputtering of titanium and niobium targets in a mixed argon/oxygen plasma. The composition of the TNO films was modulated by the Nb target power ranging from 0 to 150 Watts, resulting Nb content from 0 to ~13.9 at.%. The XRD patterns of the as-grown TNO films revealed that all the films were amorphous in nature, and the UV-visible spectra showed a high average visible transmittance above 70%. The amorphous TNO films were found to show an insulator-like behavior with high resistivity of ~1E7 ohm-cm. After post-annealing in a hydrogen atmosphere at 600°C for 1 hour, the TNO films became anatase phase. The d-spacing of the anatase (101) peak in XRD pattern almost monotonously increased with the Nb at.% due to the larger ionic size of Nb5+ (0.78 Å) than that of Ti4+ (0.75 Å). The H2-annealed TNO film with Nb of ~10.5 at.% exhibited a high average visible transmittance of 70%. In addition, Hall measurement of the TNO film revealed an n-type semiconducting behavior with a carrier density of 6.6E21 cm^{-3} and a mobility of 1.0 cm2V-1s-1, and a resistivity of 9.2E-4 Ω-cm, a dramatically ten order reduction in resistivity.

3:30pm **C2-2-7 Structural, Electrical and Optical Properties of Transparent $Zn_{1-x}Mg_xO$ Nanocomposite Thin Films**, ***A. Kaushik***, *D. Kaur (dkaurfph@iitr.ernet.in)*, Indian Institute of Technology, India

We report on the growth of $Zn_{1-x}Mg_xO$ (ZMO) thin films on quartz substrate using ultrasonic spray pyrolysis technique. AFM images of the films deposited at optimized substrate temperature clearly reveals the formation of nanorods. The influence of varying Mg composition on structural, electrical and optical properties of $Zn_{1-x}Mg_xO$ films has been systematically investigated. Increase in Mg content (in the range $0.0 \leq x \leq 1.0$), reflects the structural phase transition from wurtzite via mixed phase region to cubic one. The variation of the cation-anion bond length to Mg content shows that the lattice constant of the hexagonal $Zn_{1-x}Mg_xO$ decreases with corresponding increase in Mg content, which result in the structure gradually deviating from the wurtzite structure. Substrate temperature was also observed to have great impact on properties of these films. The optical measurements reveal a blue shift in absorption edge and increase in transmittance with increase in Mg content . Tuning of the band gap has been obtained from 3.41 eV to 6.58 eV with corresponding increase in Mg content from x = 0.0 to x = 1.0, which demonstrates that the films are useful for window layer of solar cells that improve the overall efficiency by decreasing the absorption loss. Keywords: Thin films; $Zn_{1-x}Mg_xO$; Optical band gap.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D3-1

Carbon and Nitrogen-Containing Nanostructured Composite and Nanolaminated Films

Moderator: U. Jansson, Uppsala University, Y. Pauleau, Grenoble Polytechnic Institute, J.M. Ting, National Cheng Kung University

3:50pm **D3-1-8 Applications of Nanocomposites in Organic Electronic Devices**, ***T.P. Nguyen*** *(nguyen@cnrs-imn.fr),* Institut des Materiaux Jean Rouxel, France, *C.W. Lee*, University of Chiao Tung, Taiwan **INVITED**

Organic electronic semiconductors have been developed over more than two decades and devices using them start to be commercialized in everyday applications. For instance, organic light emitting diodes (OLEDs) in small screens of cameras, cell phones, mp3 players or plastic photovoltaic cells (OPCs) cells are available on the market. Most of the materials used for these devices are conjugated polymers or small molecules, whose acute problem is their stability because degradation of materials affects the lifetime of devices. Among the different strategies proposed to remedy this drawback, the use of inorganic/organic nanocomposites is attractive by its easy processing and its ability to enhance the stability of the materials. In this talk, we present an overview of nanocomposites, which are commonly used in OLEDs and OPCs. The role of the inorganic parts, which are oxides or semiconductors, and its impact on the optical and electrical performance of devices, will be descr ibed and discussed. Some recent results on devices using nanocomposistesas active layers will be given as illustrations.

4:30pm **D3-1-10 Sol Gel Derived Hybrid Dielectric Thin films for ULSI Applications**, ***A.M. Mahajan*** *(ammnmuj@yahoo.com), B.N. Joshi*, North Maharashtra University, India

The hybrid materials with combine properties of organic and inorganic materials have attracted much attention.The number of precursors for inorganic component as metal alkoxides, organosilanes are available and they are compatible to incorporate polymerizable organic groups such as methycryloxy, vinyl and epoxy to yield wide variety of sol gel derived materials. Compare to inorganic sol gel derived hybrid films provides several advantages like better adhesion, transparency, flexibility and reduced surface roughness. These hybrid films have promising applications in optical devices, microelectronics, membranes, scratch resistant coatings etc. An important challenge for preparation of hybrid materials is to control the phase separation between the inorganic and organic moieties. @In present paper, deposition of hybrid thin films of Polymer MethylMethAcrylate (PMMA)/ SiO@sub 2@composite has been reported. These hybrid films were deposited by sol gel spin coating te chnique. Methyl MethAcrylate (MMA) monomer was polymerized by Benzoyl PerOxide (BPO) at 60@ Super O@C using water bath. This polymerised MMA were further added to sol of Tetraethylorthosilicate (TEOS), ethanol, water, acid catalyst with molar ratio 1:2:3.8:0.05. This mixture was constantly stirred for 2 hours and observed to homogenous after the stirring time. This homogenous PMMA/SiO@sub 2 @sol with optimized viscosity was coated on Si substrate at 3000 rpm for 30 seconds and then dried at 200@super O @C for 1 hour. The films have been characterized by FTIR shows the presence of hydrogen bonding at 935, 3378 cm@super-1@which supports strong chemical bonding of PMMA/ SiO@sub 2 @. The other peaks in FTIR spectra at 1071.6, 797.5, 432.39, 1729.6, 1632.5 related to Si-O-Si stretching, Si-O-Si bending, Si-O-Si rocking, C=O, C=C bonds respectively. These films have good adhesion with Si substrate. The effect of annealing temperature and detailed study of FTIR will be discus s ed in main manuscript.@

4:50pm **D3-1-11 Cermet Structured a-c:H/Pt Thin Films as High Temperature Selective Solar Absorber Coatings**, ***Y.H. Lan***, *W.Y. Wu, J.M. Ting (jting@mail.ncku.edu.tw)*, National Cheng Kung University, Taiwan

Selective solar absorber coatings with a metal-dielectric composite structure attract great attentions due to the thermal stability at high operation temperature (>400°C). In this study, a-c:H/Pt solar absorber coatings exhibiting a double cermet structure were fabricated using a magnetron sputter deposition process. Amorphous hydrogenated carbon and platinum were used as the constituents as they provide high spectral selectivity and thermal stability. Thermal stability was tested by annealing the a-c:H/Pt solar absorber coatings at different temperature from 300 to 600°C in air. The as-deposited and heat treated samples were characterized using scanning electron microscopy, glazing incident X-ray diffraction, and transmission electron microscopy. The surface chemical state was investigated using X-ray photoelectron spectroscopy. The optical performance was also examined using UV-Vis-NIR spectrophotometry and IR spectrometry.

5:10pm **D3-1-12 Microstructure and Mechanical Properties of CrAlN/Si_3N_4 Nanostructure Multilayered Coatings**, *J.-G. Duh (jgd@mx.nthu.edu.tw),* ***S.-H. Tsao***, National Tsing-Hua University, Taiwan

CrAlN and Si_3N_4 layers were deposited periodically by radio frequency reactive magnetron sputtering. In the CrAlN/Si_3N_4 multilayered coatings, the thickness of CrAlN layer was fixed at 4 nm, while the thickness of Si_3N_4 layer was adjusted from 4nm to 0.3nm. The dependence of the Si3N4 layer thickness on the preferred orientation, crystalline behavior and mechanical properties of multilayers coatings were discussed with the aid of XRD patterns and HRTEM. It was revealed that amorphous Si_3N_4 layer transformed to a crystallized one when the thickness decreased from 4nm to 0.3nm. The crystalline Si3N4 layer grew epitaxially and formed the coherent interface with the CrAlN layer and the column structure was exhibited. The critical layer thickness for the transition from amorphous Si_3N_4 to a crystallized one was 0.4nm, and maximum hardness of 33GPa was revealed.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E1-2

Friction and Wear of Coatings: Lubrication, Surface Effects and Modeling

Moderator: E. Broitman, Carnegie Mellon University, A. Fernandez-Camacho, CSIC-University Sevilla, O.L. Eryilmaz, Argonne National Laboratory

1:30pm **E1-2-1 Interfacial Scratch Adhesion Behavior of Multilayered Ti(BN):Ti(MoS_2) Based PVD Coatings**, ***I. Efeoglu*** *(iefeoglu@atauni.edu.tr),* Ataturk University, Turkey, *B. Prakash, J. Hardell*, Luleå University of Technology, Sweden

Interfacial adhesion is critical property of multilayered thin films used in micro-electromechanical systems, ceramic capacitors, wear resistant coatings. In the present investigation, Ti(BN)+Ti(MoS_2) solid multilayered-composite solid lubricant coatings were deposited by magnetron sputtering from separate Ti, TiB2, and MoS_2 target. X-ray diffraction, microhardness tester, and scratch tester were used to evaluate structural, mechanical and interfacial adhesion properties. In the work, described here changes in the adhesion of intercoat exhibited by nine different coatings deposited under variants of deposition parameters have been investigated. It is found that the crack propagates alternatively between the two interfaces with thinning of the interlayer.

1:50pm **E1-2-2 Optimization of AlN Coatings for Tribological Applications**, ***A. Rojo***, ITESM-TOL, Mexico, *J. Oseguera (joseguer@itesm.mx), O. Salas, J. Solis*, ITESM-CEM, Mexico

Several modes of injection of nitrogen during reactive magnetron sputtering of aluminum where carried out in order to optimize the AlN layer structure for tribological applications. The injection methods used were: convectional injection of Ar + N_2 mixture away from the substrate varying the amount of nitrogen, Ar + N_2 mixture away of the substrate with extra nitrogen near the substrate and direct injection of N_2 near the substrate, the resulting coatings where analyze by X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM) and tribological characterization in a pin-on disk machine. The injection method that results in a richer nitrogen atmosphere seems to produce better results. Adhesion of the coatings to the substrate is central for the tribological behavior.

2:10pm **E1-2-3 Surface Chemical Mechanisms of Lubricants Under Sliding Conditions**, ***W. Tysoe*** *(wtt@uwm.edu)*, UW-Milwaukee **INVITED**

Chlorine- and sulfur-containing compounds are commonly added to the base fluid to synthesize lubricants used under extreme pressure (EP) conditions. Analysis of the resulting tribological films on iron reveals that chlorinated hydrocarbons thermally decompose forming a layer that consists of iron chloride ($FeCl_2$) or iron carbide (Fe_3C), depending on the additive. Carbides are formed most easily when carbon tetrachloride is used as an additive. Similar analyses of films formed from sulfur-containing additives reveal that dialkyldisulfides and carbon disulfide thermally decompose to form a tribological film that consists of FeS and Fe_3C. The surface chemistry leading to the formation of these tribological films is explored on clean iron in ultrahigh vacuum using d.c. molecular beams of the reactant impinging on the surface. In order to better understand the role of thin films in reducing friction, their tribological properties were investigated in ultrahigh vacuum. This strategy eliminates contamination and allows films of known composition and structure to be grown on well-characterized substrates. It is found that a single layer of the film causes a substantial reduction in friction so that a monolayer of KCl on iron reduces the friction coefficient from its clean-surface value of ~2, to 0.27, while a layer of $FeCl_2$ reduces it to ~0.08. The friction coefficient increases once again as the film becomes thicker and this effect can be modeled using Greenwood-Williamson theory. Systematically varying the nature of the film and the substrate allows the limiting friction coefficient of the monolayer to be related to the mechanical properties of both the films and the substrate.

2:50pm **E1-2-5 Effect of Cu Content on the Tribological Performance of Cr-N Coatings at High Temperatures (840°C)**, *K. Polychronopoulou,* University of Cyrpus, *N. Demas,* University of Illinois at Urbana-Champaign, *C. Rebholz,* University of Cyprus, ***A.A. Polycarpou*** *(polycarp@illinois.edu)*, University of Illinois at Urbana-Champaign

Cu-Cr-N coatings with Cu content ≥3 at.% and Cu/Cr ratios in the 0.04-0.2 range synthesized by twin EBPVD at 450oC, were investigated and compared against substoichiometric Cr-N reference samples. The main objective of the present work was the study of the room-to extremely high temperature tribological performance of Cu-Cr-N coatings, namely 22, 500 and 840 oC. Using X-ray photoelectron spectroscopy, X-ray diffraction and scanning electron microscopy, in combination with nanoindentation mechanical property measurements and laboratory controlled ball-on-disc sliding experiments, it is shown that Cu-Cr-N coatings with low Cu contents (3 at.%) possess sufficient wear resistance for high temperature (>800°C) demanding tribological applications, such as high speed machining and aeronautical applications. Specifically for the Cu-Cr-N coatings with low Cu content a nanostructure consisting of miniature crystallites of Cu surrounding the CrxN crystallites is postulated. For the high Cu-content coatings further growth of Cu crystallites, detectable with XRD, was noticed. It was found that Cu-Cr-N coatings exhibited comparable hardness values compared to Cr-N coatings with the environmental benefit of lower Cr content (lower toxicity). Decrease of coating hardness was measured as Cu content increased due to the highly Cu (soft)-character adopted by the coating. Cu-Cr-N coatings with Cu/Cr ratios of 0.04 (Cu =3 at.%) showed superior tribological performance compared to other Cu-containing and Cu-free (Cr-N) coatings at both room and elevated temperatures. A correlation between wear behaviour/mechanism and metal oxide phases formed in situ during tribological testing was found to be the key parameter affecting the tribological performance of Cu-Cr-N coatings at elevated temperatures.

3:10pm **E1-2-7 Expanding the Range of Temperature-Adaptive Solid Lubricant Coatings Beyond 25-700°C with the Aid of In Situ Wear and Surface Analysis Techniques**, ***C. Muratore*** *(chris.muratore@wpafb.af.mil), J. Hu, J. Bultman,* Air Force Research Laboratory, *A.A. Voevodin*, Air Force Research Lab/University of Dayton

Nanocomposite coatings demonstrating multiple temperature adaptation mechanisms have been deposited with a hybrid physical vapor deposition process to provide solid lubrication from room temperature to over 700°C. These coatings generally rely on diffusion of low shear phases to the surface and tribo-oxidation mechanisms to yield moderately low friction coefficients of 0.4 over this broad temperature range. Catalyst elements can be added to the coatings to promote chemical interaction between the metal phases and produce compounds with layered atomic structures with a lower shear stress than the pure metals or their oxides, resulting in a reduction of the friction coefficient to less than 0.2 from 25-700°C and potentially higher temperatures. Multilayered coatings consisting of pure ceramic and metal adaptive lubricant layers separated by diffusion barriers to inhibit segregation and oxidation of the buried lubricant material allowed adaptation in those layers to oc cur only upon exposure by wear, which resulted in longer wear lives both at static temperatures and over multiple thermal cycles. Further studies of the multilayered coating architecture described above were carried out to demonstrate a novel in situ wear measurement and failure warning system consisting of buried layers known to produce distinctive luminescence spectra when exposed to laser illumination and located at different depths throughout the thickness of the solid lubricant coating. One example employed erbium- and samarium-doped (1 at. %) yttria stabilized zirconia (YSZ) films as sensor layers. The YSZ-Er material was placed approximately midway through an adaptive coating, and the YSZ-Sm layer was located at the coating/substrate interface. Placement of the luminescent coatings in these positions allowed detection of wear depth and provided a clear warning of impending coating failure during testing. With the wear detection system in place, luminescence spectroscop y was also performed in situ on high temperature sliding interfaces to allow identification of the onset and mechanisms of changes in surface chemistry at elevated temperature during sliding without artifacts introduced by chemical reactions and crystal growth during cooling after testing and prior to analysis. This technique also provided a means of identifying the role of sliding in altering surface chemistry.

3:30pm **E1-2-8 Optimization of the Coating Parameters for Micro Arc oxidation of Cp-Ti**, *Y. Vangolu,* ***A. Alsaran*** *(aalsaran@atauni.edu.tr), E. Arslan, Y. Totik*, Ataturk University, Turkey

TiO_2 coatings were formed on the surface of CP-Ti by AC pulse micro arc oxidation. The structural, mechanical, tribological and corrosion properties were analyzed by using XRD, SEM, microhardness tester, surface profilometer, pin-on-disk tribotester and electrochemical polarization unit. Voltage, electrolyte components, concentration, frequency and duration time were chosen as the coating parameters and three levels for each parameter were determined. By determining the wear, surface hardness and corrosion behaviors, the optimum working conditions were determined by using a Taguchi design of experiment. After micro arc oxidation, the aim is to minimize the friction coefficient, the wear rate and the corrosion resistances and to maximize surface hardness after micro arc oxidation. While the optimum conditions were determined, due to existance of more than one goal, a trade-off among goals was considered. First of all, each goal was optimized separately, and then all the goals were optimized together, considering the priority of the goals. Keyword: AC pulse micro arc oxidation, Taguchi method.

3:50pm **E1-2-9 A Pure-Silver Coatings for Electrical Contact Application Subjected to Fretting Conditions**, ***J. Jedrzejczyk***, Technical University of Lodz, Poland / Ecole Centrale de Lyon, France, *S. Fouvry (siegfried.fouvry@ec-lyon.fr),* Ecole Centrale de Lyon, France, *P. Chalandon*, Centre Technique de Belchamp, France

Materials used in the electrical contacts in cars are expected to have very low and stable electrical resistance and high resistance for corrosion. The environmental and mechanical factors strongly influence mentioned parameters. Thus, numerous coating systems consisting of pure metallic materials, noble and non-noble, doped as well as soft coatings were developed and studied. In this work the degradation mechanisms of bronze/nickel/pure-silver coating system was studied under fretting loading. Three thicknesses (1.3µm, 2µm, 3µm) of the silver coating were investigated.

The Variable

Displacement Amplitude methodology (VDA), Constant Displacement Amplitude (CDA) as well as interrupted tests were applied to distinguish the transition amplitude between Partial Slip (P.S) and Gross Slip (G.S) regime, build so called Wohler-like curves and clarify the mechanism of the wear of the coatings. The silver coating was found to delaminate from

nickel interlayer in the very beginning of the tests. Despite this, the lifetime of the electrical contact has shown to be satisfactory. The process of the wear of the coating was determined and explained. A quantitative variable, i.e. the electrical resistance threshold •R = 0.004Ω, was applied to distinguish the lifetime of studied materials and to allow the comparison between examined coating configurations.

4:10pm **E1-2-10 Effects of Sicking-Sliding Zones on the Tool Wear and Coating Delamination When Machining Hard Materials**, ***S. Bahi*** *(slim.bahi@insic.fr), A. Moufki, M. Nouari,* Ecole Nationale Supérieure des Mines de Nancy, France*, M. El Mansori,* Arts et Métiers Paristech, France*, A. Molinari*, Ecole Nationale Supérieure des Mines de Nancy, France

To minimize tool wear and coating delamination processes, it is necessary to understand the nature of interactions between chip flow, tool and coating materials. In this paper, a hybrid analytical-numerical approach is performed for the orthogonal cutting process. The modelling of the thermomechanical material flow in the primary shear zone, the tool-chip contact length and the sliding-sticking zones is obtained from an analytical approach. In addition, the Finite Element method is used to solve the non linear thermal problem in the chip. At the chip-tool interface, the friction condition can be affected by the important heating induced by the large values of pressure and sliding velocity. In spite of the complexity of phenomena governing the friction law in machining, a reasonable assumption is to consider that at the sliding zone the mean friction coefficient is primarily function of the average temperature at the tool-chip interface. The objective is to propose an ap proach which can easily be used to identify the main parameters governing tool wear and to explain the experimental trends. The effects of cutting conditions and material behaviour on the sliding-sticking zones and on the temperature distribution along the tool-chip interface are evaluated. It has been found that the sliding-sticking zones at the tool chip interface strongly control the local conditions of stress, velocity and temperature. Experimental analyses of the sticking-sliding zones and their evolution at the tool-chip interface have been used to identify tool wear modes and coating delamination process for different cutting conditions. A qualitative comparison between the model and the experimental results is also provided.

Characterization: Linking Synthesis Properties and Microstructure
Room: Sunrise - Session F2/B7

In Situ Characterization for Deposition Proceess and Film Properties Modeling
Moderator: M. Beckers, Linköping University, D. Depla, Ghent University

1:30pm **F2/B7-1 Quantification of the Incorporation Coefficient of a Reactive Gas on a Metallic Film During Magnetron Sputtering: the Method and Results**, ***W.P. Leroy*** *(wouter.leroy@ugent.be), S. Mahieu,* Ghent University, Belgium*, R. Persoons,* Flemish Institute for Technological Research (VITO), Belgium*, D. Depla*, Ghent University, Belgium

Magnetron sputtering is a widely used technique to deposit thin films. Adding a reactive gas to the discharge allows one to deposit stoichiometric compound films from a metallic (or substoichiometric) target at a high deposition rate. However, the addition of this reactive gas makes the control of the deposition process a complex task. Huge efforts are going into the understanding of the fundamental phenomena of this reactive sputter process and into the simulation of the deposition process by e.g. PIC/MC. However, one of the most uncertain parameters in this reactive sputtering process is the incorporation coefficient of the reactive gas on the growing layer, i.e. the real-time sticking coefficient during deposition. In this work, mass spectrometry is used to deliver more insights on this complex matter.

A method has been developed to determine the incorporation coefficient of the reactive gas onto the growing metal film, using mass spectrometry combined with thin film analysis. This method delivers a global, realistic incorporation coefficient and hence a correct parameter to be used in the models for the reactive sputtering process. We have determined the sticking coefficient of O_2 and N_2 on several metals during reactive magnetron sputtering, and relate these to the material properties of the different metals.

1:50pm **F2/B7-2 Experimental and Numerical Plasma Characterization in a Deep Reactive Ion Etch System**, ***S.P. Koirala*** *(skoiral@uark.edu), M.H. Gordon,* University of Arkansas*, S.L. Burkett*, The University of Alabama

Using deep reactive ion etch (DRIE) with a modified Bosch process, we create through-silicon-vias (TSVs) which have application in 3D interconnects. These interconnects allow the development of three-dimensional architectures which have several advantages including faster signal processing and overall system miniaturization. Our modified Bosch process uses alternating etch and passivation cycles with sulfur hexafluoride (SF_6)/Argon (Ar), and cyclofluorobutane (C_4F_8), respectively. Previous studies show that the coil power and pressure significantly affect the quality of the via shape. In this study, optical emission spectroscopy and a Langmuir probe are used to experimentally characterize the plasma conditions during the etch process, and numerical plasma models are used for comparison and optimization. Preliminary Langmuir probe studies show that the introduction of SF6 into an Ar plasma causes a slight decrease in the ion density and a signific ant decrease in electron density. This latter effect is attributed to the electro-negativity of SF_6. The optical emission studies provide information on the relative population of argon and fluorine excited states, species which both play an important role in the formation of TSVs. Numerically, the commercially available Boltzmann equation solver ELENDIF is used in conjunction with an Ar collision radiative model to simulate the experimental conditions. Good agreement with experimental data is obtained.

2:10pm **F2/B7-3 Initial Stages of Polycrystalline Thin Film Growth as Seen by Scanning Probe Microscopy**, ***T. Michely*** *(michely@ph2.uni-koeln.de)*, Universität zu Köln, Germany **INVITED**

The growth of Ag thin films deposited at 300 K on amorphized Si surfaces under ultra high vacuum conditions is investigated by in situ scanning tunneling microscopy (STM). The analysis of the film morphology as a function of film thickness together with additional annealing or low temperature experiments allow one to obtain a quite complete picture of the film formation processes. It is shown that the large kinetic stability of the trenches separating Ag islands strongly influences all the structural features during the initial growth stages, i. e., island density, grain size, roughness and texture evolution. Specifically it becomes understandable, why abnormal grain growth is initiated only when continuous films develop facets. Finally it is shown, how texture evolution and grain structure can be manipulated in ion assisted growth by using a grazing incidence ion beam.

The contributions to this work by Celia Polop, Christian Rosiepen, Daniel Förster and Se bastian Bleikamp are acknowledged.

2:50pm **F2/B7-5 Surface Mound Formation During Epitaxial Growth of CrN(001)**, ***X. Zhang*** *(zhangx11@rpi.edu), D. Gall*, Rensselaer Polytechnic Institute

Single crystal CrN(001) layers, 10 to 160 nm thick, were grown on MgO(001) by ultrahigh vacuum magnetron sputtering at growth temperatures T_s = 600 and 800°C. All layer surfaces exhibit mounds which evolve in both shape and size, as observed by in-situ scanning tunneling microscopy and quantified by statistical analyses using height-height correlation functions. For T_s = 600°C, the root mean square surface roughness σ initially increases sharply from 0.69±0.18 for a thickness t = 10 nm to 2.44±0.48nm for t = 20nm, but then remains constant at σ = 2.43±0.13nm for t = 40, 80 and 160 nm. The mounds exhibit square shapes with edges along <110> directions for t≤40 nm, but develop dendritic shapes at t = 80 nm which revert back to squares at t = 160 nm. This is associated with a lateral mound growth that is followed by coarsening, yielding a decrease in the mound density from 5700 to 700 μm^{-2} and an initial increase in the coherence length L from 7.18±0.6 to 16.27±0.8 to 23.87±2.87 nm for t = 10, 20, and 40 nm, respectively, followed by a drop in L to 22.22±1.97 and 16.12±1.89 nm for t = 80 and 160nm, respectively. Growth at T_s = 800°C leads to opposite trends: σ decreases from 1.98±0.45 to 0.92±0.07 and 1.03±0.07 nm and L decreases from 20.48±3.67 to 10.27±0.37 and 9.84±0.45nm, for t = 10, 20, and 40 nm, respectively, while the mound density remains approximately constant at 900 μm^{-2}. These unexpected trends are associated with mounds that elongate and join along <100> directions, yielding long chains of interconnected square mounds for t = 40 nm. At larger t, coalescence causes a rapid mound growth with σ increasing to 2.54±0.2 and 2.52±0.11nm, L increasing to 23.77±2.52 and 40.02±2.34 nm, and the mound density decreasing to 280 and 100 μm^{-2}, for t = 80 and 160 nm, respectively.

3:10pm **F2/B7-6 In Situ Stress Evolution in TiZrN and TiTaN Thin Films Grown by Reactive Magnetron Sputtering**, ***G. Abadias*** *(gregory.abadias@univ-poitiers.fr),* Universite de Poitiers, France, *Ph. Guerin,* Université de Poitiers-CNRS, Laboratoire PHYMAT, France, *L.E. Koutsokeras, P. Patsalas*, University of Ioannina, Greece

Due to their inherent physical and mechanical properties transition metal (TM) nitride thin films are commonly used in various applications, such as contact layers in microelectronics or protective hard coatings on cutting tools.[1] Extensive studies have been carried out in binary compounds (TiN, ZrN or TaN) to relate deposition conditions, stress, microstructure and preferred orientation to films' properties. In particular, it is important to understand stress development during growth to tailor thin films with reduced stress levels to enhance device's lifetime and reliability.

Current efforts are now made to synthesize functional and adaptative layers based on multicomponents TM-based systems. Among these, the TiN-ZrN and TiN-TaN systems arouse an increasing interest due to the possibility to stabilize ternary nitride solid solutions with enhanced properties. For example, conducting $Ti_{1-x}Ta_xN$ thin films ($0<x<1$) could be stabilized using various physical vapor deposition techniques.[2] However, stress development during growth in these multinary systems remains unexplored.

In the present work, we investigated the stress evolution during reactive magnetron cosputtering of TiTaN and TiZrN thin films using a real time wafer curvature measurement technique. Samples were grown at 300°C on Si wafers under an Ar/N_2 atmosphere. The influence of substrate bias voltage, deposition rate, N_2 and Ar partial pressures on stress buildup was studied. The obtained data showed the presence of stress gradients over film thickness, as a result of two competing stress producing mechanisms: atomic peening inducing compressive stress and void formation inducing tensile stress.[3] Complimentary ex situ techniques using X-ray diffraction and atomic force microscopy were performed to correlate stress evolution with texture formation and film morphology. A comparison will also be made with thin films grown by pulsed laser deposition.

[1] G. Abadias, Surf. Coat. Technol. 202, 2223 (2008)

[2]L. E. Koutsokeras, G. Abadias, Ch. E. Lekka,G. M. Matenoglou, D. F. Anagnostopoulos, G. A. Evangelakis and P. Patsalas, Appl. Phys. Lett. 93, 011904 (2008)

[3]G. Abadias and Ph. Guerin, Appl. Phys. Lett. 93, 111908 (2008).

3:30pm **F2/B7-7 In Situ AFM Investigation on Tribo-Corrosion of CrSiN Film Adherent Tool Steel**, ***H.-H. Lin***, *C.-C. Chou (cchou@mail.ntou.edu.tw),* National Taiwan Ocean University, Taiwan, *J.-C. Huang, J.-W. Lee, Y.-C. Chang, Y.-B. Lin*, Tungnan University, Taiwan

A CrSiN film was coated on SKD61 to enhance the anti-corrosion capability by a bipolar symmetry pulsed DC reactive magnetron sputtering process. A series of mechanical and electro-chemical polishing processes were implemented to obtain various surface roughnesses before the CrSiN films were built. The adhesion of CrSiN-coated samples was evaluated by scratch test. The corrosive characteristics were studied by potentiodynamic test and electrochemical impedance spectroscopy (EIS) in a 3.5 wt.% NaCl solution. An in-situ atomic force microscope (AFM) was then applied to detect their pitting corrosion in a 0.01 M NaCl solution. Two loading conditions were applied to investigate the corrosion induced by different stresses. The results demonstrate that the significant improvement of the CrSiN-coated SKD61 substrates either in tribological property and corrosion resistance as well. In the mean time, the in-situ corrosion behavior of sample's microstructures was also discussed and addressed.

3:50pm **F2/B7-8 Fast Characterization of Reaction Waves in Exothermic, Metal-Metal Multilayer Nanolaminates**, ***D.P. Adams*** *(dpadams@sandia.gov), J. McDonald, M Hobbs*, Sandia National Laboratories

Sputter-deposited, exothermic multilayer films and foils have generated a great deal of interest recently, because they exhibit rapid, high-temperature, self-sustained reactions characterized by tailorable average propagation speeds.[1] These multilayers often consist of two or more reactant layers (typically dissimilar metals) characterized by a large negative heat of formation. Layer thicknesses range from 5-300 nm and coatings may consist of thousands of individual layers. Despite much interest, a great deal of research is required to fully understand the behavior and performance of these materials. In this presentation, we describe the dynamics of reaction front propagation as these depend on multilayer design and composition. Using high-speed photography (1E5 frames per second with 5 um spatial resolution) and high-speed thermal imaging devices we show direct evidence for steady and unsteady modes. A few multilayer systems (e.g., those consisting of Al/Pt) exhibit a steady reaction mode with no evidence for unsteady behaviors. Co/Al, Ni/Ti and other lower-enthalpy metal-metal pairs exhibit various, unsteady modes - particularly when bilayer thickness is made large. We further show how reaction mode and flame morphology depends on the total thickness of a given multilayer system (varied in this study from 150 nm to 50 micrometers). In addition, the relationship of reaction mode to final foil microstructure and morphology is presented. Complimentary finite difference and finite element heat transport models explain some of the observed macroscopic behaviors.

1 U.S. Patent 5,538,795 T.W. Barbee, Jr. and T. Weihs.

4:10pm **F2/B7-9 Quantitative Measurement of Ion Energy Distributions Impinging onto Arbitrarily Biased Substrates During Plasma Deposition**, ***T. Baloniak*** *(Tim.Baloniak@rub.de), A. von Keudell*, Ruhr-Universität Bochum, Germany

Substrate biasing is an established technique to control and adjust material properties during thin film deposition from a plasma. The energy distribution function of the ions impinging onto the substrate (IEDF) is manipulated by the external bias voltage. Optimal ion bombardment can significantly improve film properties like hardness, adhesion, crystallinity, or wear resistance. In our contribution, we report about the quantitative measurement of ion energy distribution functions on arbitrarily biased substrates. The measurements are performed in a magnetically enhanced, capacitively coupled argon discharge, which is heated by 13.56 and/or 71 MHz. An aluminum target is mounted on the powered electrode. The substrates are placed on an arbitrarily biased electrode driven by RF waveforms at 1 MHz. A miniaturized, floating retarding field analyzer allows for IEDF measurements on the biased substrate holder. The energy distributions are found to be good replica of the bias waveforms applied to the substrate, eventually skewed by collisions at higher pressures. Our findings allow to design tailored waveforms for optimal ion bombardment and thus, optimal film properties.

Applications, Manufacturing, and Equipment
Room: Royal Palm 1-3 - Session G7

Advances in Industrial PVD & CVD Deposition
Moderator: R. Cremer, CemeCon AG

1:30pm **G7-1 Innovative Applications Enabled by Large Area Hot Filament Diamond Deposition**, ***J.W. Zimmer*** *(jzimmer@sp3inc.com), D. Aidala, J Herlinger*, sp3 Diamond Technologies

Hot filament diamond coatings have traditionally been used only for cutting tool applications because of perceived limitations in film characteristics. Recently however improvements in the quality, range of morphology, and electrical characteristics of hot filament diamond have allowed many additional commercial applications to be addressed. This talk will discuss some of these new applications and how large area diamond deposition has enabled their growth.

1:50pm **G7-2 Industrial Scale Production of CVD-Diamond Coated Mechanical Seals and Electrodes**, ***M. Rüffer*** *(mrueffer@diaccon.de), M. Foreta, J. Holzke, M. Nierada,* DiaCCon GmbH, Germany, *S. Rosiwall*, University Erlangen Nürnberg, Germany

The outstanding properties and advantages of crystalline CVD-diamond coatings applied onto high performance mechanical seals and electrodes are widely known. But similar to the diamond coating of tools an adopted coating set-up must be developed for industrial scale diamond coating of mechanical seals and electrodes. Mechanical seals ask for quite good flatness of sliding faces and electrodes require homogeneous, double side coating of large areas without bending or overheating of the metal substrate. For both applications different setups have been developed, by using the same platform, a CemeCon CC800/9.

For an economic industrial application a large filament array must be used to coat numerous or large parts in one batch. These filaments should perform long process times and must stand for several batch processes. Especially for coating of mechanical seals a horizontal setup assures the required flatness of the faces by providing homogeneous temperature distribution and gas composition over the complete coating area.

Since applications of diamond coated electrodes like waste water treatment usually need large coated areas and valuable substrate materials like Niobium, a double side coating of an electrode is preferred. The coating of such electrodes should be achieved in one batch without interrupting the process and turning the substrate. A vertical filament setup that is enclosing the electrode from both sides provides overall homogeneous distribution of doping elements (e.g. Boron), equal diamond quality and prevents bending at once.

We show that it is possible to coat with quite different setups different high performance products by using the same CVD-machine.

2:10pm **G7-3 Characterization of Industrial-Scale LAFAD Technology and Applications**, ***V.I. Gorokhovsky*** *(VIGASE@aol.com)*, Arcomac Surface Engineering, LLC

A unique industrial-scale Large Area Filtered Arc Deposition (LAFAD) process offers the opportunity for significant improvement in surface engineering technologies. The unidirectional LAFAD dual-arc vapor plasma source yields 100% ionized metal vapor plasma flow and more than 50% ionized gaseous plasma in the coating chamber. The dramatic increase of magnetized plasma propagating capabilities in LAFAD process produces substantially conformal coatings on complex shapes. The LAFAD technology deposits a thick ceramic and cermet coatings with multielemental nanostructured architectures, near defectless morphology and atomically smooth surface at high deposition rates. The operating range of LAFAD process allows it to combine with conventional EBPVD and magnetron sputtering in a hybrid surface engineering processes. The productivity of one unidirectional LAFAD vapor plasma source integrated in industrial scale batch coating system ranging from 3-4 μm/hr for nitride and carbide base coatings and up to 6 um/hr for oxiceramic coatings with required uniformity over large deposition areas, making it an attractive alternative to other PVD processes for wide variety of applications. The modular design of the LAFAD plasma source allows it to be easily integrated in any batch coating or in-line coating systems. The industrial applications of LAFAD process in deposition of wear and corrosion resistant coatings for forming tools and medical instruments, erosion resistant coatings for turbomachinery, tribological coatings on automotive and aerospace components, high temperature oxidation resistant coatings for SOFC and commercialization strategy of LAFAD technology will be discussed.

2:30pm **G7-4 Development of a Magnetically Steered Cathodic Arc Evaporation Source**, ***K. Yamamoto*** *(yamamoto.kenji1@kobelco.com), S. Tanifuji, S. Nakakubo, H. Fujii, Y. Kurokawa, S. Kujime*, Kobe Steel Ltd., Japan

Cathodic arc evaporation process (AIP) is nowadays well established and widely adopted in many industries involved with thin film technology, mostly for triboligical applications such as cutting tools. As already well recognized, arc plasma is characterized by high degree of ionization ratio up to 90 % which assures superior adhesion and densification of the coating. High deposition rate is also an advantage of the cathodic arc compared to sputtering or other industrial PVD process for hard coating. Most recognized drawback is emission of macro-particles (MPs) which is considered more or less unavoidable nature of the cathodic arc process. Kobe steel has been a pioneer in realization of magnetically steered arc technology for industrial use and in this paper, a novel cathodic arc evaporation source based on magnetic steering principle and coating properties are reported. A magnetically arc evaporation source based on a new magnetic field design was developed and deposition of various hard coating, such as TiN, (Ti,Al)N, (Al,Cr)N and (Ti,Cr,Al)N was conducted in a industrial coating system. Surface roughness of standard 3 μm (Ti,Al)N coating from our previous two kinds of arc source is Ra=0.2μm for random arc and Ra=0.1μm for plasma enhanced cathode[1]. Whereas the surface roughness can be as good as 0.02μm for 3 μm (Ti,Al)N coating deposited by the new arc source. Similar significant improvement in surface roughness can be observed for other major nitride coatings. The other characteristic of the coating deposited by the new source is significant reduction in residual stress. Residual stress of a few Gpa is commonly observed for arc deposited nitride coatings, whereas for the coatings deposited by the new source, the stress can be as small as 0.02GPa. A very thick (Ti,Al)N coating up to 20 μm can be grown on very sharp edge of a cutting tool without any chipping. Cutting test showed a significant enhancement in the tool life compared to a standard (Ti,Al)N coating with a few microns of thickness. Application of the new source to other coating systems will be reported.

[1]K. Yamamoto, T. Sato, K. Takahara, K. Hanaguri, Surface and Coatings Technology 174 –175 (2003) 620–626.

2:50pm **G7-7 Integration Aspects of High Impulse Magnetron Sputtering Plus (HIPIMS+) Technology in an Industrial PVD Coating Machine**, ***F. Papa*** *(fpapa@hauzer.nl), R. Tietema, T. Krug, C. Strondl, I. Kolev*, Hauzer Techno Coating BV, Netherlands

Recent developments in the field of High Power Impulse Magnetron Sputtering (HIPIMS) have instigated great interest from both the academic and industrial worlds. The promise of ionized sputtered material creates the possibility for metal ion assisted deposition. This opens a new world of possibilities for creating defect free, conformal coatings with tailored microstructures. For academic research, small cathodes requiring low average powers are used. However, in order for HIPIMS technology to have commercially interest, it must be scaled up for large scale industrial PVD coaters. This requires a reliable power supply which can deliver an average power between 10 and 40kW. In addition to the HIPIMS supply itself, considerations must also be made for the design of different parts of the machine, such as the bias supply and magnetic field design of the cathode. HIPIMS+ is a platform which encompasses all aspects which are necessary for the industrial commercialization of HIPIMS technology. The properties of HIPIMS+ deposited coatings such as Titanium Nitride (TiN), Chromium Nitride (CrN) and Aluminum Titanium Nitride (AlTiN) will be presented. Potential tool and tribological applications will also be discussed.

3:10pm **G7-8 The New Vacotec/Eifeler Alpha 400/900P PVD Deposition System as a Basis for Innovative Coatings for Industries and Research**, ***H. Hrubý*** *(hynek.hruby@eifeler.com), E. Voss, G. Keiren,* Eifeler Werkzeuge GmbH, Germany*, J. Anklam*, Vacotec S.A., Switzerland

The successful and innovative concept of Vacotec/Eifeler PVD technology based on the Alpha 400P coating system has been recently demonstrated[1].

These new Alpha 400/900P arc coaters show a broad variety of technical features to produce state of the art PVD coatings for industrial as well as R&D applications.

Highlighted are the flexibility for hard and lubricant coatings as well as the combination of in-situ plasma-nitriding with a hard coating. With these standard features of the new generation machines it is easy to create well adopted PVD coatings which are nicely fitting for many special applications.

Using the DC arc with very high plasma density offers the ability to produce films of high density and superb wear resistance features. This will be demonstrated with nanostructured AlTiN-based films and their properties.

Regarding the operating costs it is shown that the concept of multiple arc sources is a ve ry competitive way to produce high quality wear resistant films for industrial use. And the concept offers the possibility to produce PVD films out of up to 4 different cathode materials at a very high deposition rate.

A couple of applications concerning metal cutting, metal forming and component coating will demonstrate the broad flexibility of the presented PVD machine concept. Limitations of the concept and technology and some ideas how to overcome these will be discussed.

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New Horizons in Coatings and Thin Films
Room: Tiki Pavilion - Session H4

Thin Films for Photovoltaics: Synthesis and Characterization

Moderator: S. Khare, University of Toledo, S. Fairchild, Air Force Research Laboratory

1:30pm **H4-1 Manufacturing Photovoltaic Systems**, ***L. Kazmerski***, NREL **INVITED**

2:10pm **H4-3 Steady State and Transient Photoconductivity of a-$Se_{90-x}Sb_{10}In_x$ ($0 \le x \le 15$) Thin Films**, ***M. Kamboj*** *(mkamboj@ee.ryerson.ca), F. Mohammadi*, Ryerson University Toronto, Canada

Amorphous thin films of $Se_{90-x}Sb_{10}In_x$ ($0 \le x \le 15$) have been prepared by electron beam evaporation method. The steady state and transient photoconductivity measurements on the thin films of $Se_{90-x}Sb_{10}In_x$ ($0 \le x \le 15$) have been carried out at different level of light intensities (500 lx – 5000 lx) at room temperature (301 K). The plot of photocurrent (I_{ph}) versus light intensity (F) follows a power law $I_{ph} = F^{\gamma}$. The value of exponent lies between 0.5 and 1.0, which indicates there exists a continuous distribution of localized states in the mobility gap of $Se_{90-x}Sb_{10}In_x$ ($0 \le x \le 15$) thin films. For transient photoconductivity, when the samples were illuminated with light, the photocurrent quickly reaches the maximum value and thereafter, it start decreasing with the exposure time and becomes stable after 15 minutes of exposure. This kind of phenomenon is termed as photo-degradation of photocurrent. The results have been explained on the basis of charged defect model and the inter-cluster interaction model. High photocurrents are found for a-$Se_{75}Sb_{10}In_{15}$ system which is even higher than the parent system $Se_{90}Sb_{10}$. The photosensitivity shows a minimum value at 5 at% of Indium (In) concentration, which has been explained based on chemically ordered network and the topological models.

2:30pm **H4-4 Enhancing Ion Diffusion of Nanoporous TiO_2 Film in Dye-Sensitized Solar Cell by Pore Structure Controlling Through Vacuum Cold Spray**, ***G.-J. Yang*** *(ygj@mail.xjtu.edu.cn), C.-J. Li, S.-Q. Fan, C.-X. Li, J.-C. Gao, Y.-X. Xi*, Xi'an Jiaotong University, China

The ion diffusion performance of nanoporous TiO_2 film was an important factor which influences the efficiency of the dye-sensitized solar cell. Although increasing the pore size by using large primary TiO_2 particles, such as 60nm, can increase the ion diffusion performance, the smaller surface area resulting from large primary particle size will deteriorate the cell efficiency by decrease the dye molecules adsorbed on TiO_2 particle surface. In this study, nanoporous TiO_2 film was deposited by vacuum cold spray using TiO_2 powders with primary particle size at 25nm. The porosity and pore size were examined by nitrogen adsorption and desorption technique. The diffusion performance of I_3^- in acetonitrile was examined. The results showed that the pore size of the coating was significantly influenced by the structure of the spray powder. However, the porosity of the coatings was found to be the same despite of different structure of the spray powders. The I_3^- ion diffusion coefficient was found to increase from 2.2×10^{-7} cm^2/s to 5.2×10^{-7} cm^2/s when the mean pore size increased from 19nm to 30nm. Vacuum cold spray technique was proposed to be a promising approach by which the ion diffusion performance of porous film can be controlled by the pore size within the porous film.

2:50pm **H4-5 Glancing-Angle Deposited Titania Films for Dye-Sensitized Solar Cells**, *H.Y. Yang,* ***M.F. Lee****, M.S. Wong (mswong@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

A series of sculptured porous nano-columnar titanium oxide films were prepared by glancing angle deposition (GLAD) method using an electron-beam evaporation system. The films were deposited on ITO glass and used as photo-anode in the dye-sensitized solar cell (DSSC). The as-deposited TiO_2 films have anatase phase and ordered porous nano-columnar structures. Both features of the photo-anode are advantageous for electron transfer and for high surface area, resulting in the enhanced dye-absorption, thus, promote the efficiency of the DSSC. In this study, we varied glancing angle and film thickness, and focused on the structure, crystallinity, dye-absorption, and light-absorption of the films, and their effect on the performance of DSSCs. The DSSCs exhibited a high fill-factor(FF) above 0.78. The best performing solar cell incorporating an 8 μm thick TiO_2 film fabricated at deposition angle of 73° had a photoelectric conversion efficiency of 3.23 %.

3:10pm **H4-6 High Performance Polymer Soloar Cells and Optical Sensors**, ***Y. Yang*** *(yangy@ucla.edu)*, UCLA **INVITED**

In this presentation, we report the formation of polymer solar cells, with efficiency closed to 6% power conversation efficiency based on a new class of Si-containing conjugated polymer. The Si atom provides better pi-pi stacking on the polymer in the solid state format, hence improves the device performance significantly. The device with thin film with only 50% transparency reaches 5.6% efficiency. On the other hand, attempts to add quantum dots to further the efficiency has leaded to the discovery of high gain photoconductivity photo detector. By adding CdTe QDs into P3HT:PCBM blends, the polymer photo-device reaches 8000 times higher photo current.

3:50pm **H4-8 Hybrid Solar Cells Based on Poly (3-Hexylthiophene):Fullerene Blend and TiO_2 Porous Film**, ***M.K. Fung****, C.T. Yip, K.Y. Cheung, A.B. Djurišić (dalek@hkusua.hku.hk), W.K. Chan*, The University of Hong Kong

While inorganic solar cells have higher efficiency, faster electron transport and stability, organic solar cells can be fabricated by inexpensive methods on large area substrates, resulting in increasing interest in organic photovoltaics in spite of their inferior charge transport properties and efficiency compared to inorganic solar cells. One of the possible methods to combine the advantages of the two types of material is the use of hybrid organic/inorganic solar cells. However, the efficiencies of hybrid solar cells based on inorganic nanostructures and organic polymers are still relatively low. The reasons for low efficiency of hybrid cells are partially due to possible problems at the organic/inorganic interface[1], and also to the poor infiltration of polymer into the inorganic nanostructured network[2]. In spite of this, it has been reported that the performance of pure polymer solar cells was improved by employing nanostructured semiconducting materials as charge collectors[3-5]. We have previously reported solar cells based on TiO_x porous layer and poly(2-methoxy-5-(2'-ethyl-hexyloxy)-p-phenylene vinylene) (MEH-PPV)[4]. Since the absorption spectrum of MEH-PPV is not as well matched with the solar spectrum as that of poly(3-hexylthiophene) (P3HT) or P3HT:[6,6]-phenyl-C61-butyric acid methyl ester (P3HT:PCBM) blends, we investigate here the performance of solar cells based on P3HT:PCBM active layers as a function of the polymer/fullerene ratio and active layer thickness. The polymer infiltration and its relationship to the solar cell performance were examined by scanning electron microscopy, while the solar cell performance was characterized by I-V curve measurements in the dark and under simulated solar illumination.

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4:10pm **H4-10 Photoelectrochemical Properties of Nitrogen-Doped Indium Tin Oxide Thin Films Prepared by Reactive DC Magnetron Sputtering Technique**, ***K.R. Wu*** *(krwu@nkmu.edu.tw),* National Kaohsiung Marine University, Taiwan*, C.Y. Yeh,* Kao Yuan University, Taiwan*, C.H. Hung,* National Kaohsiung First University of Science and Technology, Taiwan*, C.Y. Chung*, National kaohsiung Marine University, Taiwan

Nitrogen-doped indium tin oxide (N-ITO) thin films are deposited on unheated ITO glass substrates as visible-light enabling catalysts by direct current (DC) magnetron sputtering technique. Structural properties of the post annealed N-ITO thin films characterized by X-ray diffraction (XRD) show that some indium nitride (InN) nano-particles are embedded in the ITO matrix. A broad XRD peak around $2\theta=33^\circ$ is assigned to the (101) InN plane. This is also observed in a lattice image of cross-sectional high resolution transmission electron micrograph (HRTEM). The corresponding diffraction pattern confirms the formation of (101) and (112) InN planes. The InN nano-particles enable the light absorption of the N-ITO catalysts to longer wavelengths of about 500 nm, narrowing the band gap from 3.9 eV to approximately 2.5 eV. Under ultra-violet (365 nm) and visible-light (blue LED, $410<\lambda<550$ nm) illumination, the N-ITO catalysts show the photocurrent densities of 80 and 2.5 $\mu A/cm^2$, respectively, with Na_2CO_3 as the sacrificial agent. These are attributed to the heterojunction arrays; the formation of InN phases embedded in the crystalline ITO matrix. The heterojunction arrays can facilitate the photoinduced electrons and holes separation for better photoelectron transfer. Although optimization will be needed to deliver high photocurrents, the present work shows that ITO can be suitably doped with N_2 gas to produce a promising catalyst with improved photoelectrochemical properties for solar water splitting for hydrogen evolution.

Coatings for Fuel Cells
Room: Sunset - Session TS2

Coatings for Fuel Cells

Moderator: J.W. Stevenson, Pacific Northwest National Laboratory, D. Mumm, University of California

1:30pm **TS2-1 Electroplated Coatings on Ferritic Steels for SOFC Interconnect Application**, ***J.H. Zhu*** *(jzhu@tntech.edu)*, Tennessee Technological University **INVITED**

In the planar design of a solid oxide fuel cell (SOFC) stack, the interconnect acts not only as electrical connection between the various cells but also as the mechanical support for the thin electroactive ceramic parts and as gas-proof separation of air and fuel gas. With the reduction of the SOFC operating temperatures to 600-800°C, chromia-forming ferritic steels are widely used as interconnect materials in the planar-type SOFC stacks currently under development. These ferritic steels such as Crofer 22 APU and SS 430 possess an overall combination of properties desirable as SOFC interconnect materials such as low cost, excellent manufacturability, adequate match in coefficient of thermal expansion (CTE) with other cell components, high electronic conductivity and thermal conductivity. Two major concerns with these ferritic interconnect alloys are (1) their long-term oxidation resistance and oxide scale electrical conductivity, and (2) Cr evaporation and associ ated "poisoning" of the cathode under the operating environments of SOFC. To address these issues, protective coatings need to be developed, which should be economically viable, electrically conductive, and chemically compatible with the substrate alloy and other cell components. The present talk provides an overview of the recent research efforts in the development of electroplating as a processing route to fabricate protective coatings on ferritic steels. The progress and the issues with electroplating for synthesis of the protective coatings are summarized. Several promising approaches are highlighted for mitigating the current

problems with the electroplated coatings. Some examples are given where electroplated alloy coatings are utilized for protecting the ferritic steels.

2:10pm **TS2-3 Improved Properties of SOFCs using Pre-Coated Sandvik Sanergy HT 22% Cr Ferritic Interconnect Steel**, ***U. Bexell*** *(ulf.bexell@sandvik.com), M. Schuisky*, AB Sandvik Materials Technology, Sweden

Ferritic stainless steel has attracted a great deal of attention for its use as an interconnector in solid oxide fuel cells (SOFCs). The ferritic Sandvik Sanergy HT chromium steel is specially developed for interconnectors in SOFC with a unique chemical composition, which gives the alloy a good high temperature corrosion resistance as well as good surface conductivity in the formed chromium oxide scale. However, chromium evaporation from metallic interconnectors in SOFC fuel cells tends to poison the cathode of the fuel cell. Furthermore, the evaporation of chromium species from the oxide surface tends to increase the oxidation rate resulting in increased contact resistance. To reduce chromium evaporation from the interconnectors, rather thick coatings have been deposited using various spraying techniques such as plasma spraying. In this study, a 22% Cr ferritic steel, Sandvik Sanergy HT has been coated with thin metallic films. These coated samples are compared to unco ated material. The idea is to promote the formation of a dense cap layer thus reducing chromium evaporation and increasing scale conductivity.

Oxidation studies have been carried out on pre-coated and uncoated samples of Sandvik Sanergy HT in air at different temperatures. The samples were analyzed by XRD, SEM/EDX on polished and/or FIB cut cross sections. Also, the surface morphology and chemistry were studied by SEM/EDS.

2:30pm **TS2-4 Evaluation of LAFAD Multielemental Oxiceramic Nanocomposite Coatings on Ferritic Steel SOFC Interconnects**, *V.I. Gorokhovsky (VIGASE@aol.com),* Arcomac Surface Engineering, LLC, *P.E. Gannon,* Montana State University, ***J.L. Wallace***, Arcomac Surface Engineering, LLC, *M.C. Deibert, R.J. Smith, H. Chen, P. White*, Montana State University

Reduced operating temperatures (600-800°C) of Solid Oxide Fuel Cells (SOFCs) may enable the use of inexpensive ferritic steels as interconnects. Due to the demanding SOFC interconnect operating environment, protective coatings are gaining attention to increase long-term stability by preventing volatilization of chromium and inhibiting TGO growth, while securing high electrical conductivity on the interconnect surface. One unidirectional large area filtered arc deposition (LAFAD) source was able to deposit multielemental nanocomposite oxiceramic, oxinitride and nitride coatings of the MeTiCrAlYO(N) system on ferritic steel (where Me=Co,Mn) with high deposition rates ranging from 4 to 6 μm/hr on substrates installed on a rotating turntable within an industrial batch coating system. The ferritic stainless steel substrates coated with electrically conductive and chromium retentive LAFAD coatings were subjected to various tests in environments which simulated SOFC stack ope ration. Coating compositions and surface morphologies were characterized using AFM, RBS, SEM/EDS and XRD analyses. Coating adhesion was evaluated under thermal cycling conditions. The performance of the full scale metallic IC plates with LAFAD protective coatings was tested in a SOFC stack. It was found that LAFAD multielemental nanocomposite coatings can effectively eliminate chromium volatility and substantially reduce the TGO growth. The technical and economical assessment of LAFAD coating technology for SOFC applications will be discussed.

2:50pm **TS2-5 Development of Spinel Protection Layers for Steel-Based SOFC Interconnects**, ***J.W. Stevenson*** *(jeff.stevenson@pnl.gov), Z.G. Yang, G.B. Xia, X.S. Li, Z.M. Nie, C.M. Wang*, Pacific Northwest National Laboratory

Due to their low cost, high temperature oxidation resistance, and appropriate thermal expansion match to anode-supported cells, ferritic stainless steels are among the most promising candidate materials for interconnect applications in intermediate-temperature planar SOFC stacks. For long term operation, however, several issues remain, including long-term surface stability, electrical resistance due to scale growth, and chromia scale volatility that can lead to cell poisoning and performance degradation. To improve their performance, ferritic stainless steels can be surface-modified via application of a conductive oxide coating. In particular, $(Mn,Co)_3O_4$ based protection layers are being developed and optimized at PNNL for several candidate ferritic stainless steels. Recent progress will be summarized in this paper.

3:10pm **TS2-6 Concentration-Dependent Ionic Conductivity of Magnetron-Sputtered Nanocrystalline Scandia-Stabilized Zirconia**, ***M. Sillassen*** *(mbs@phys.au.dk),* University of Aarhus, Denmark, *P. Eklund,* Linkoping University, Sweden, *N. Pryds, N. Bonanos,* University of Denmark, *J. Bøttiger*, University of Aarhus, Denmark

Scandia-stabilized zirconia (SSZ) electrolyte materials exhibit the highest conductivity of all zirconia systems making them likely candidates for solid oxide fuel cells (SOFC). Nanocrystalline scandia-stabilized zirconia electrolytes with scandia contents varying from 5 to 16 mol% have been synthesized by reactive pulsed DC magnetron sputtering from a Zr target with strips of Sc attached. Upon increasing the Sc content, the crystal structure of the SSZ evolved from the monoclinic phase to the cubic and rhombohedral phase. For films deposited at 400ºC and a bias of -70 V, X-ray single-line profile analysis yielded a grain size of ~10 nm and a microstrain of ~1.5%, regardless of scandia content. SSZ films deposited at 400ºC and selected bias voltages in the range from -70 V to -200 V showed a reduced grain size for higher bias voltages yielding a grain size of ~5 nm and a microstrain of ~2.5% at bias voltages of -175 V and -200 V with additional incorporation of argo n. Impedance spectroscopy analysis of the SSZ films showed that the highest in-plane ionic conductivity was obtained for films with a scandia content close to 10 mol%.

3:30pm **TS2-7 Structure and Conductivity of Apatite-Like Lanthanum Silicate Films for SOFcs Electrolytes**, ***J.C. Oliveira*** *(joao.oliveira@dem.uc.pt),* University of Coimbra, Portugal, *M. Vieira,* Polytechnic Institute of Leiria, Portugal, *A.L. Shaula, A. Cavaleiro*, University of Coimbra, Portugal

The development of Intermediate Temperature Solid Oxide Fuel Cells (IT-SOFCs) will require electrolyte materials with ionic conductivity higher than the conventional yttria-stabilised zirconia (YSZ) at moderate temperatures. Recently, lanthanum silicates materials ($La_{9.33}Si_6O_{26}$) with an apatite-like structure have attracted considerable interest as potential low cost electrolyte materials. Some of these materials show conductivities comparable to, or better than, YSZ at 875 K, and are thus potential electrolytes for economic feasible fuel cells. Their high level of oxide ion mobility is related to the presence of oxygen channels along the c axis which facilitate the diffusion of anionic species. Magnetron sputtering has already been used to synthesize thin film electrolytes for SOFCs owing to its versatility as well as the ability to control composition and morphology. Most of the reported work focuses on the deposition of thin dense yttria-stabilised zirconia (YSZ) gadolinium doped ceria (GDC) and lanthanum gallate electrolyte layers. The main objective of this work is the production of apatite-like lanthanum silicates thin films by magnetron sputtering. Thin films with the appropriate La/Si atomic ratios were deposited by reactive magnetron sputtering from LaSi and Si targets and subsequently annealed in controlled atmosphere to obtain the targeted lanthanum silicate oxide. The chemical composition of the coatings was determined by electron probe microanalysis (EPMA). The structure of the coatings was studied by X-ray diffraction (XRD) using a Phillips diffractometer operated in Bragg-Brentano configuration with Co(Kα) radiation. The cross section and surface topography of the La-Si films were examined on a JEOL scanning electron microscope (SEM) equipped with an EDAX energy dispersive spectrometer (EDS). The electrical properties of the films were measured by AC impedance spectroscopy (HP4284A precision LCR meter, 20 H z – 1 MHz).

3:50pm **TS2-8 The Corrosion Properties and Interfacial Contact Resistance of TiN, TiAlN and CrN PVD Coatings in Simulated PEM Fuel Cell Environments**, ***L. Wang***, *D.O. Northwood,* University of Windsor, Canada, *J. Housden, E. Spain,* Tecvac Ltd., *X. Nie (xnie@uwindsor.ca)*, University of Windsor, Canada

Metallic bipolar plates, especially stainless steels are widely accepted as promising candidates to replace graphite in PEM fuel cell as electrodes. The major concerns on their corrosion susceptibility and contact resistance increments after formation of surface passivation films have induced the increasing interest in finding promising protective coatings on metallic bipolar plates to prevent the degradation of plates. In this study, the interfacial contact resistance (ICR) and electrochemical properties of TiN, CrN and TiAlN PVD coatings and their stainless steel 316 substrate were investigated in a simulated PEM fuel cell environment. The potentiodynamic polarization corrosion tests were conducted with purged O_2 or H_2, and the potentiostatic corrosion tests were performed under both simulated cathodic (+0.6V vs. SCE purged with O_2) and anodic conditions (-0.1V vs. SCE purged with H_2) for a long period (4 hrs). SEM was used to observe the surface m orphologies of the samples after corrosion tests. The test results showed that the TiAlN and CrN coatings had a low ICR but the TiN coating exhibited a slightly high ICR, compared with the uncoated SS 316. TiN and CrN-coated metallic plates could potentially be used as anode plates in PEM fuel cell environment based on their anti-corrosion performance. However, since the main corrosion initiated at the pinholes on

the PVD coatings, more efforts are needed to be explored to eliminate the pinholes resulted from the PVD deposition process.

Coatings for Use at High Temperature
Room: Royal Palm 1-3 - Session A2

Coatings for Use in Harsh Environments
Moderator: J.R. Nicholls, Cranfield University, M. Schütze, DECHEMA e.V.

8:00am **A2-1 Application of PVD Coatings in Food Processing Tools**, ***M. Rostagno*** *(maddalena.rostagno@diadsrl.com), S. Durante,* DIAD, Italy*, M. Perucca,* Clean NT Lab - Environment Park S.p.A., Italy*, E.A. Boot, F. Cartasegna*, Environmental Park, Italy

PVD and CVD coatings have been widely used in automotive, aeronautical and mechanical applications for their outstanding wear resistance and hardness. Sectors such as food processing have not yet benefited of these technologies and at present the material used are not standardised in specific way. The corrosion/erosion of the machinery used for food processing it's not only an economical cost (maintenance, substitution of corroded parts, plant stop), but above all an health and environmental cost. The corroded/eroded materials is transferred into the food during the different processing and, as consequence, it is systematically ingested by the final consumers, with concrete possibilities of accumulation and toxicology effects. A typical example it is the nickel release from inox steels used in bakery sector, that it's suspected to be responsible for allergies and other pathologies, always more frequent even if the allowed nickel threshold has been continuously lower ed. In the food and biomedical sector a growing caution is dedicated to the use of aluminum: since the end of the last century, the aluminum toxicological and neuro-toxicological effects are under investigation: the iper-alluminemy in the humans has been detected as a possible cause of pathologies such as dialysis deficiency, pulmonary diseases, microcistic anemy (no dependent from iron) and others. At present, the scientific community has strong suspects that aluminum ingestion can be a possible cause of Parkinson and Alzheimer. Application of new materials such as coatings can bring a strong innovation, but up to now these are strictly under the responsibilities of the end users who are called to carry out all the test and characterisations necessary to guarantee the safeness and atoxicity of the material applied. The paper presents the results obtained in the application of PVD coatings in steel components used for processing of food in the fodder production sectors. Demon stration test results are shown together with the coatings characterisation. A significant improvement in wear resistance of the coated components has been achieved with a favourable cost/benefit ratio proved by a detailed economic calculation.

8:20am **A2-2 Corrosion Behavior of Titanium Oxide Films in Different Solutions Formed by Ac Pulse Peo**, *Y. Totik, E. Arslan,* ***Y. Vangolu*** *(yvangolu@atauni.edu.tr), A. Alsaran, A. Çelik*, Ataturk University, Turkey

Titanium and its alloys are widely used for various applications in medicine, aerospace, automotive, chemical plant, power generation, oil and gas extraction, sport, and other major industries that involves various solutions. The corrosion behavior of Cp-Ti oxidised by plasma electrolytic oxidation (PEO) was investigated by electrochemical measurements in 0.5 NaCl and 0.5 HCl solutions. The Cp-Ti oxidised was characterized using X-ray diffraction, scanning electron microscopy and potentiodynamic polarization techniques. It was observed that the corrosion media affected on Cp-Ti oxidized by PEO. Keyword: Commercially pure titanium, Plasma electrolytic oxidation, Corrosion.

8:40am **A2-3 Electrochemical Impedance Spectroscopy (EIS) Study on Corrosion Performance of CrAlSiN Coated Steels in 3.5 wt.% NaCl Solution**, ***C.H. Lin****, J.G. Duh (jgd@mx.nthu.edu.tw)*, National Tsing-Hua University, Taiwan

CrAlSiN coatings with Si content from 0 at.% to 11.5 at.% were deposited on mild steels, 304 stainless steels, and 420 stainless steels by magnetron sputtering. According to the broadened peaks in XRD patterns, effect of grain refinement due to dopped Si was verified in as-deposited coatings. Investigations of electrochemical impedance spectrum (EIS) were carried out in 3.5 wt.% NaCl solution to evaluate corrosion resistance of various coating configurations. Two different corrosion mechanisms were proposed for the CrAlSiN/MS and CrAlSiN/SS configurations. From Nyquist plots, CrAlSiN coatings revealed significant improvements on charge-transferred resistance (Rct) with increasing Si content for those deposited on 304 and 420 stainless steels. These improvements were attributed to the grain refinement effect in CrAlSiN coatings. On the other hand, CrAlSiN coatings deposited on mild steel substrates exhibited no specific trend on Rct in EIS tests. From observation of the pitting holes, inferior Rct were attributed to serious delamination of the CrAlSiN coatings. In this case, adhesion between coatings and mild steels was responsible for the measured Rct rather than the microstructure.

9:00am **A2-4 A Dry Drilling Process Contribution with Solid Carbide TiAlN + AlCrN Coated Drill**, ***W. Mattes*** *(wilmar@senai-sc.b),* SENAI-SC, ALVES, Brazil*, S. Martins*, SOCIESC, Brazil

For both economic and environmental reasons, strong demand for dry drilling exists in the global market. However, it has been especially difficult to obtain a long tool life and to ensure the high quality of drilled holes when dry drilling Austempered Ductile Iron (ADI). Dry drilling tests were carried out without the use of cutting fluid, ADI 293HB and using a coated carbide drill (diameter=10mm) with Vc=120m/min, f=0.2mm/rev, and drilling depth 30mm. Newly developed very smooth TiAlN + AlCrN coating films were deposited on carbide drills. The cutting force during drilling, chip formation characteristics, and wear resistance of the drill edges were investigated. The specially designed drill flute shape, which created small-size chips, effectively reduced the cutting force. Newly developed very smooth nitride films effectively reduced not only the cutting force but also the amount of the wear at the cutting edge. The qualities of the drilled holes were also investigated. Based on these results, very deep dry drilling of Austempered Ductile Iron was achieved together with a practical tool life. This report describes an investigation of the appropriate drill shape and the influence of the coated films on the dry drilling of Austempered Ductile Iron.

9:20am **A2-5 High Temperature Corrosion Behaviour of Materials in Heavy-Duty Gas Turbines with Fuel Flexibility**, ***B. Bordenet*** *(bettina.bordenet@power.alstom.com)*, Alstom (Switzerland) Ltd., Switzerland **INVITED**

In stationary gas turbines, hot gas path components are in contact with combustion product gases containing impurities. The materials and coatings will degrade by gaseous attack and by hot corrosion, if corrosive salts can condense. The kind and the extent of corrosion attack are strongly dependent on the type and quantity of corrosive species. These contaminants have their origin in the fuel, the intake air and the injected water, where the fuel is the main source. The contaminants are varying with the different fuel types. From a corrosion perspective two main type of fuels can be differentiated: fuels with a high amount of impurities and cleaner fuels with a low amount of impurities. The ash-bearing fuels such as crude and heavy oil belong to the first group, whereas natural gas and diesel belong to the second group. Other liquid fuels or syngas from coal / biomass gasification can belong to both groups depending on their impurity content, which is determined by the level of fuel cleaning.

The encountered corrosion mechanism will be described in dependence of the chemical composition and the quantity of the contaminants. The different types of corrosion attack are illustrated by micrographs from ex-service components and from laboratory samples.

To minimise the hot corrosion damages, process- and material-related countermeasures are proposed for the different fuels. The process-related solutions are described shortly and their impact on the gas turbine operation will be evaluated. Their main goal is the change of the corrosive environment through fuel treatment or use of additives. The other option is the use of higher corrosion-resistant materials and coatings in the hot gas path. An overview of the typical gas turbine materials will be given with respect to their corrosion performance. The focus will be put on the behaviour of metallic overlay coatings and thermal barrier coatings in corrosive environment. Consequences will be discussed for the use of corrosion-resistant systems in new power plants with low CO_2 emissions.

10:00am **A2-7 Diffusion Coatings for Oxidizing High-Chlorine Environments at Elevated Temperatures**, ***B. Rammer*** *(rammer@dechema.de), T. Weber, M. Schütze*, DECHEMA e.V., Germany

The presented work aims to find material solutions for a recently developed process that allows the recovery of Phosphorus, an essential plant nutrient and therefore integral component of many fertilizers, from sewage sludge ashes. The process offers the possibility to overcome the previsible shortage of Phosphorus as a natural resource. It is distinguished by the use of highly chlorine-containing atmospheres at temperatures up to 1000°C. Unfortunately, there are currently no materials commercially available that can withstand such conditions over longer periods of time.

For the development of a sufficiently resistant material system, nickel base alloys were chosen as a base material. They feature outstanding high temperature corrosion resistance and strength. To further advance these qualities, protective diffusion coatings, applied by pack cementation, were developed. The thermodynamic assessment of different coating elements

revealed that aluminum and silicon have the best prerequisites to form and maintain slow-growing, stable oxide layers with the highest potential for being protective against corrosive attacks.

The performance of the coated materials was examined in long-term tests under simulated field conditions at high temperatures and under atmospheres of up to 10% Cl_2 in air. In the paper the theoretical considerations with regards to coating design and results of these experiments will be presented and discussed.

10:20am **A2-10 Production, Characterization and Evaluation of Protective Cr Oxide Coatings Against Metal Dusting**, ***D. Melo***, IPN, Mexico, *D. Singüenza, O. Salas,* ITESM-CEM, Mexico, *R. Reichelt,* Wilhelms-Universitaet, Germany, *J. Oseguera (joseguer@itesm.mx),* ITESM-CEM, Mexico, . *López*, IPN, Mexico

Cr oxide/Cr films have been deposited on HK40 steel substrates in order to evaluate their behavior under metal dusting conditions. The films were produced by reactive magnetron sputtering under various conditions of oxygen flow, work pressure, power and bias voltage to find the optimum deposition conditions to form an adequate dense structure with good adhesion to the substrate. The coatings were extensively characterized by scanning electron microscopy, x-ray diffraction and atomic force microscopy. The most promising structures were subjected to metal dusting conditions in a thermobalance to evaluate their response to this type of corrosion.

10:40am **A2-9 Factors Affecting the Performance of a Porous, High Temperature Abradable Coating**, ***D. Allen*** *(david.b.allen@siemens.com)*, Siemens Energy

In today's advanced industrial gas turbines, clearance control is of great importance for reducing fuel consumption and decreasing emissions. Clearance control methods such as abradable coatings are commonly employed on the compressor and turbine sections of these engines. Much of the development work to date on these coatings has been based upon empirical data (rub tests) but it would be beneficial to have some a priori basis for predicting the wear performance of these coatings. To this end we have run a directed series of tests, using a design of experiments (DOE) approach, to elucidate the impact of various coating characteristics on abradability.

11:00am **A2-12 An Overview of Sulzer Metco Abradable Coatings and Some New Developments in Blade Tipping**, ***S. Wilson*** *(Scott.Wilson@sulzer.com),* Sulzer Metco, Switzerland, *D. Sporer,* Sulzer Metco Europe, *M. Dorfman*, Sulzer Metco, USA

Thermally sprayed abradable seals are employed in turbomachinery to reduce leakage gaps between stationary and rotating parts to improve efficiency and stall margin. These seals are commonly composite type coatings which derive their abradability from the use of low shear strength materials or from a porous, friable coating structure. The development of mostly zirconia based thermal shock resistant ceramics for use at high temperatures, allowed for abradable seals for the high pressure turbine stages to be developed. On stages with reduced temperatures, both ceramic and metallic coatings of the MCrAlY type can be used.

This paper reviews the state of the art in compressor and turbine clearance control materials and systems, with a focus on novel thermally sprayed ceramic turbine seals with encouraging property combinations which are achieved by the introduction of alternative stabilizers. Normally ceramic seal surfaces require hard tipping of the rotor blades to allow them to cut properly and recent silicon carbide hard tipping technology will be presented.

11:20am **A2-8 Microstructure and Cutting Mechanics in Abradable Seal Coatings**, *S. Goergen,* Cranfield University, United Kingdom, *C. Sellars,* Rolls Royce Plc, United Kingdom, *I. Walton, D.J. Stephenson,* ***J.R. Nicholls*** *(j.r.nicholls@cranfield.ac.uk)*, Cranfield University, United Kingdom

It is widely recognised that a good abradable coating can significantly improve the efficiency of a gas turbine engine, with correct design allowing blade wear to be minimised. Thus my understanding the abradable process, its link to abradable coating microstructure and the underlying cutting mechanics, it is possible to optimise coating properties.

In this study, metallographic characterisation of abraded coatings has been carried out for three different blade tip/coating systems. Based on this understanding, a micro-cutting model has been constructed to predict abradable behaviour.

It is demonstrated that for a given abradable system coating performance is highly dependent on parameters that define the abradable process, particularly blade tip velocity and incursion rate, and their interaction with the abradable coating microstructure/properties. Based on this modelling work guidelines are given to aid the optimisation of abradable coatings.

11:40am **A2-11 Degradation of Hot-Dip Aluminized Coating on the Cyclic Oxidation of the Ferritic 430 Stainless Steel**, ***M. Badaruddin*** *(D9603801@mail.ntust.edu.tw), C.J. Wang*, National Taiwan University of Science and Technology (NTUST), Taiwan

The 430 stainless steel was coated by hot-dip in a molten pure aluminum bath. The high temperature oxidation of aluminized stainless steel was studied at 800 °C in static air. After oxidation for 20 h, the intermetallic layer consists of three major phases ($FeAl_2$, FeAl and $(Fe_2Cr)Al$), while the Fe_2Al_5 phase forms a thin layer in the outer. The intermetallic layer between the Fe_2Al_5 and $FeAl_2$ contains with many Cr_5Al_8 precipitates. At the exposure time 120 h, in the outer layer the remain phase of $FeAl_2$ containing many voids and cavities severely suffers the degradation by causing spallation and cracking an oxide layers. However, the beneath phase of FeAl provides a good performance in protecting the metal during oxidation attacking. A protective oxide formed on the surface is affected by the properties of intermetalic layer.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B1-3

Sputtering Coatings and Technologies
Moderator: C. Rebholz, University of Cyprus, M.S. Wong, National Dong Hwa University

8:00am **B1-3-1 Study of the Effect of Plasma Current Density on Nitrides and Oxynitrides Titanium Thin Films Prepared by Reactive DC Magnetron Sputtering**, *P.K. Barhai (pkb@bitmesra.ac.in), N. Kumari, I. Banerjee,* Birla Institute of Technology, India, *S.K. Pabi,* Indian Institute of Technology Kharagpur, ***S.K. Mahapatra***, Birla Institute of Technology, India

Nitrides and oxynitrides titanium films were deposited by varying the plasma current density from 10mA/cm2 to 40mA/cm^2, using DC magnetron sputtering. Different colours of the films like golden, blue, pink and green were obtained at different current densities. At lower current density (~10mA/cm^2), the film showed stoichiometric TiN, whereas, at higher current densities (~20, 30 and 40mA/cm^2) the films showed non stoichiometric TixONy. Crystallinity and atomic concentration of the films have been characterized by GAXRD and XPS techniques. The average thickness of the films increased with plasma current from 775A0 to 996A0. The hardness, roughness and Young's modulus of the films were analyzed by nano-indentation. Hardness of the films was found to increase from 7.054 Gpa to 17.49 Gpa and Young's modulus from 209.774 Gpa to 257.586 Gpa with increasing plasma current density. AFM image of the films showed uniform nanostructured grains. The durability and the quality o f the film colour have been characterized by colorimetric analysis. I-V characteristics of the films were also studied for their use in microelectronic applications.

8:20am **B1-3-2 Chromium Containing Amorphous Hydrogenated Carbon Thin Films (a-c:H/Cr) as Selective Solar Absorber Coatings**, ***H.Y. Cheng***, *W.Y. Wu, J.M. Ting (jting@mail.ncku.edu.tw)*, National Cheng Kung University, Taiwan

Metal-containing amorphous hydrogenated carbon (a-c:H/Me) thin films are used as spectral solar selective absorber coatings. In this study, the obtained a-c:H/Cr thin films were deposited on silicon wafers or copper foils using a reactive magnetron sputter deposition method. The gases used were mixtures of methane and argon. The methane concentration was varied continuously from 0 to 40 % during the deposition. The obtained films were characterized using scanning electron microscopy, transmission electron microscopy, energy dispersive spectroscopy, X-ray absorption near edge spectroscopy, and extended X-ray absorption fine structure spectroscopy. The optical performance was examined using UV-Vis-NIR spectrophotometry and Fourier transform infrared spectrometry. The films were found to exhibit high absorptance greater than 95 % from 0.3 to 2.5 μm and low emittance less than 1 % from 2.5 to 10 μm. The dependence of the optical properties on the film microstruc ture, and the size distribution, concentration, and chemical state of the chromium will be presented and discussed.

8:40am **B1-3-3 Investigation of the O^- Ion Emission During Reactive Magnetron Sputtering**, ***S. Mahieu*** *(Stijn.Mahieu@ugent.be), W.P. Leroy, D. Depla*, Ghent University, Belgium **INVITED**

One of the most used techniques to deposit thin films is magnetron sputtering. To deposit an oxide thin film, reactive magnetron sputtering, i.e. sputtering a metallic target in an Ar/O_2 mixture, can be used. Since the target is at highly negative potential, negative oxygen ions formed at the target surface or within the cathode sheath will be accelerated away from the target, eventually towards the substrate. Hitting the growing film, the high energy negative ions will influence the film growth and eventually have a detrimental effect on the resulting film properties. Hence, characterizing the amount and energy of these negative ions is an important and challenging task. During planar magnetron sputtering, the energy distribution of negative O^- ions has been measured by means of energy resolved mass spectrometry for 13 different target materials. For the same series of target materials the ion-induced secondary electron emission coefficient was determined in earlier published research. A correlation between this ion-induced secondary electron emission coefficient and the emission of the high energetic negative O^- ions was observed. The influence of the cathode shape, i.e. a planar magnetron versus a rotating cylindrical magnetron, on the outward flux of negative ions has been investigated. Therefore, the amount, energy, and direction of the high energy negative ions have been measured, again by means of energy resolved mass spectrometry.

9:20am **B1-3-5 A Satisfactory Explanation for the Discharge Voltage Behaviour During Reactive Magnetron Sputtering**, ***D. Depla*** *(Diederik.Depla@ugent.be), S. Mahieu, R. De Gryse*, Ghent University, Belgium

The discharge voltage is perhaps the most accessible parameter of the magnetron sputter deposition process. As its value can be easily monitored, research reports generally contain its value. Nevertheless, the interpretation of the discharge voltage and/or its behavior, especially during reactive magnetron sputtering, is less straightforward. To understand its behavior, it is necessary to look into the details of the magnetron discharge, the processes occurring at the cathode or target and the influence of the important discharge parameters such as discharge current, magnet configuration, and discharge gas pressure. The influence of these parameters can be partially understood from a general formula. This formula, based on the original work of Thornton, shows that the discharge voltage behavior during reactive magnetron sputtering finds its origin in the formation of a compound layer on the target. The discharge voltage behavior depends strongly on the material properti es of the compound layer which is formed. Further research is of course still needed, but with this paper we hope to take away some frustration in the reactive sputter deposition community, as summarized by Westwood in his book on sputter deposition when discussing the material dependence of the discharge voltage on target material "A satisfactory explanation for these differences has not yet appeared".

9:40am **B1-3-6 Ion Energy Distributions in AZO Magnetron Sputtering from Planar and Rotatable Magnetrons**, ***F. Richter*** *(f.richter@physik.tu-chemnitz.de), T. Welzel,* TU Chemnitz, Germany, *R. Kleinhempel,* Southwall Europe GmbH, Germany, *T. Dunger,* TU Chemnitz, Germany, *T. Knoth, M. Dimer, F. Milde*, von Ardenne Anlagentechnik GmbH, Germany

Ion energy distribution functions (IEDFs) have been measured at the substrate position in magnetron sputtering of an aluminium doped zinc oxide (AZO) target in inert and reactive atmosphere. The IEDFs have been obtained against ground potential with an in-line energy dispersive ion mass spectrometer (plasma monitor) which was facing the target surface. Two different magnetron configurations have been investigated: a circular planar magnetron on laboratory scale and an industrial scale rotatable magnetron. The magnetrons were operated with asymmetric-bipolar pulsed d.c., the planar magnetron also in d.c. mode. Positive ions of the working gas as well as Al^+, Zn^+, and AlO^+ were observed, which show a low energy peak in their IEDF from the 'on' phase and a high energy peak of several 10 eV from the 'off' phase of the pulsed d.c. Negative ions, being mainly O^- from the target, exhibit a strong peak in their IEDF at several 100 eV correspo nding to the negative target voltage. With strongly varying target voltage in the 'on' phase the IEDF may extend to very high energies. Maximum and average energy of both positive and negative ions depend on the discharge power as the voltage in the 'on' and 'off' phase governing the high energy ions changes with power. The changes in the negative ions have consequences even on floating substrates whereas the changes in positive ions may only affect grounded substrates. The differently sized planar and rotatable magnetron exhibit very similar IEDFs for positive as well as negative ions proving that basic physical processes are essentially the same. Significant differences were observed for both sources when the connection between the anode of the power supply and the magnetron source was altered.

10:00am **B1-3-7 Investigation of the Role of Hydrogen in Silicon Deposition Using an Energy-Resolve Mass Spectrometer in an Ar/H2 Radio Frequency Magnetron Discharge**, ***S.L. Mensah*** *(smensah@uark.edu),* University of Arkansas, *H.H. Abu-Safe,* Lebanese American University, Lebanon, *H.A Naseem, M.H. Gordon*, University of Arkansas

Ion energy distributions of sputtered Si particles have been measured by an energy-resolved mass spectrometer, and we correlate the results with measured thin film properties. The plasmas have been generated in a simple magnetron chamber powered with 30-180W at 13.56MHz at pressures ranging from 5-30mTorr. Various Hx+, SiHy+, fragments (with x,y = 1,2,3) together with Ar+ and ArH+ species were detected in the discharge. The most important species for the film deposition appear to be SiHy+ with y =1,2, and the H+ fragments seem to play the most important role to the hydrogen content in the material. In a pure argon discharge, the Ar+ flux increases with power and pressure, and decreases as hydrogen is introduced into the discharge. The flux of Ar+ decreases in this case as that of ArH+ increases with increase in power and pressure. Plasma parameters, such as plasma potential and electron density and energy, measured with the Langmuir probe and are in good agreement with literature. The ion energy of SiHy+ radicals becomes bimodal with increasing pressure.

10:20am **B1-3-8 Simulation of Layer Sequence in Multilayer Coatings Prepared by Sputtering**, ***M. Panjan*** *(matjaz.panjan@ijs.si),* Jozef Stefan Institute, Slovenia, *T. Peterman,* University of Ljubljana, Slovenia, *P. Panjan, M. Cekada*, Jozef Stefan Institute, Slovenia

Multilayer coatings are commonly prepared by PVD processes. Multilayer structure with specific layer sequence is obtained when substrates rotate around targets of different materials. In order to deposit coating on all parts of the substrate with complicated geometry (e.g. a tool), substrates need to rotate around two, three or even four axes (e.g. planetary rotation). Trajectories of substrates are therefore rather complex and layer sequence depends on the number of rotations and initial positions of the substrate. In some applications, sequence and thickness of layers significantly influences physical properties of the coating. For example, optical properties or hardness of superlattices are both strongly affected by the thickness and sequence of layers. Therefore, when depositing multilayer coatings it is important to have control over these parameters. The most convenient approach to predict the multilayer structure is to use a computer simulation. We developed such simulation for sputtering in industrial deposition system with four magnetron sources arranged in the corners of rectangle where substrates can rotate around three axes. In the simulation we can calculate growth rate in dependence of rotation and determine the sequence and the thickness of layers. Simulation results were compared to layer sequence of TiAlN/CrN and TiAlN/VN multilayer coatings prepared in industrial magnetron sputtering system CC800/9 (CemeCon). Samples were investigated by transmission electron microscope. TEM micrographs and simulated multilayer structure agree well for all types of rotation and any initial position of substrate. Our simulation therefore offers precise information of multilayer structure for all coatings prepared in the single batch.

10:40am **B1-3-9 Improving the Oxidation Resistance of AlCrn Coatings by Tailoring Chromium Out-Diffusion**, ***R. Escobar Galindo*** *(ramon.escobar@uam.es),* Universidad Autonoma de Madrid, Spain, *J. Endrino,* Instituto de Ciencia de Materiales de Madrid, Spain, *G. Fox-Rabinovich,* McMaster University, Canada, *J.M. Albella*, Instituto de Ciencia de Materiales de Madrid, Spain

In this work, we have studied the influence on the oxidation resistance of AlCrN-based coatings of the deposition of an additional subsurface titanium nitride barrier layer. Since oxidation is interrelated with the inward diffusion of oxygen into the surface of AlxCr(1-x)N (x=0.70) coatings and the outward diffusion of Cr to the surface, it is believed that the oxidation behaviour of the aluminium-rich AlCrN coatings can be tuned by the design and composition of the coating. The buried depth of the barrier layer and the oxidation time were varied and changes in the AlCrN/TiN depth composition profiles and surface oxidation states were analyzed by means of Glow Discharge Optical Emission Spectroscopy (GDOES) and Cross Sectional SEM (X-SEM) maps. It was observed that when the TiN barrier was deposited near the top surface (500 nm) the formation of beneficial alumina surface layers was promoted. This is explained in terms of a limited surplus of chromium from the coating t o the surface and corroborated after performing experiments using CrN as barrier layers. The oxidation kinetics of the multilayer CrAlN/TiN was followed using GDOES depth profiles of samples by fixing the barrier buried depth and varying the oxidation time.

11:00am **B1-3-10 Phase Transformation, Thermal Stability, Morphological and Mechanical Characteristics of the Ni- Al and Ni-P-Al Alloy Coating Systems**, ***J.C. Wu***, *F.B. Wu (fbwu@nuu.edu.tw)*, National United University, Taiwan

In this study, binary Ni-Al and ternary Ni-P-Al alloy films were fabricated by magnetron sputtering technique for comparison. Through X-ray diffraction technique, the as-deposited Ni-Al films revealed noticeable crystallization peaks. On the other hand, a broadened peak with relatively low intensity, implying an amorphous microstructure, was found for the Ni-P-Al coating. It was believed that the co-deposition of P element could induce the amorphous feature of the Ni-Al-based coatings. After annealing from 350 to 600°C, the Ni-Al films showed a well-crystallized microstructure similar to that observed for the as-deposited films. The Ni-P-Al coatings maintained amorphous feature below a high annealing temperature of 550°C. As the annealing temperature was increased to 600°C, the Ni-P-Al coating transformed to Ni crystalline matrix with precipitations of Ni_xP_y and Ni_pAl_q intermetallic compounds. The surface morphology evaluatio n was carried out by atomic force microscopy. It was found the Ni-Al coatings exhibited a surface roughness around 2.7 nm in both as-deposited and heat-treated states. On the contrary, the surface roughness of the Ni-P-Al coatings increased with phase transformation phenomenon due to post annealing. Nevertheless, the Ra was kept at a low value of approximately 3.2 nm for the Ni-P-Al alloy coating under 600°C annealing. In addition, the evolution of phase transformation related mechanical properties of these alloy coatings were also discussed.

11:20am **B1-3-11 Oxidation Study of Mo-Ru Hard Coatings**, ***Y.-I. Chen*** *(yichen@mail.ntou.edu.tw)*, National Taiwan Ocean University, Taiwan

Mo-Ru coating, a high melting point metal alloy coating, has been used as a protective coating on the top surface of the glass molding die to prolong the lifetime of the die material. In a realistic molding environment, the atmosphere was consisted of steadily purged nitrogen and residual oxygen. The oxidation resistance of the coating under the cyclic thermal history in mass production is a critical issue. In this study, the diffusion of oxygen into the $Mo_{38.1}Ru_{61.9}$ deposits annealed at 600°C in the molding atmosphere were investigated by EPMA and Auger depth profile, which revealed an apparent oxygen content in the near surface region. An atomic percentage of 5 % was observed in a depth of 0.04 and 0.27 μm of the $Mo_{38.1}Ru_{61.9}$ deposits annealed for 4 and 16 hours, respectively. The microstructure evolution of Mo-Ru coating was studied by XRD, SEM and TEM, which revealed a columnar structure with a major axis perpendicular to the su bstrate either before or after annealed in the molding environment. The oxygen diffusion did not transform the Mo_5Ru_3 phase but strengthen the surface hardness of the $Mo_{38.1}Ru_{61.9}$ deposits from 13GPa to 19GPa after 4 hours annealing and sustain a level of 18GPa till 16 hours. ESCA analysis was performed to confirm the phase stability at the near surface region of the Mo-Ru coatings.

Hard Coatings and Vapor Deposition Technology
Room: Sunrise - Session B6-1

Hard and Multifunctional Nano-Structured Coatings

Moderator: M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Laboratories, U.S. Army ARDEC, R. Sanjines, EPFL

8:00am **B6-1-1 Processes, Properties and Application Potential of Ti-Al-X-N and Cr-Al-X-N Thin Films**, ***P.H. Mayrhofer*** *(paul.mayrhofer@unileoben.ac.at)*, Montanuniversität Leoben, Austria **INVITED**

Thin films based on transition metal aluminum nitrides, like Ti-Al-N and Cr-Al-N, are increasingly important for industrial applications due to their outstanding chemical and physical properties including high hardness, toughness and thermal stability. Prepared by low-temperature (substrate temperatures below 500°C) plasma-assisted vapor deposition techniques, such materials possess metastable phases as the atomic assembly kinetics are limited. These metastable phases in addition to the coating's structure are often the key-components for the high-performance during testing and application. The structure and the phases formed strongly depend on the characteristics of the deposition process like substrate temperature, gas pressure, ionicity of the film forming species and ion bombardment. Consequently, this review deals with the influence of process parameters on the structure and metastable phases of Ti-Al-N and Cr-Al-N thin films and their properties. This is obtained by a variety of analyzing techniques including Langmuir measurements, x-ray diffraction, nanoindentation, high-resolution transmission electron microscopy, differential scanning calorimetry, tribological investigations and machining tests.

A further modification in properties of these ternary nitrides can be obtained by controlled alloying with specific elements to develop tailor-made coatings combining unique properties. In this review, the influence of the alloying elements (X) like B, V, Y, Nb, Hf, and Ta on the phase formation, structure, mechanical and thermal properties (including age-hardenability) of Ti-Al-N and Cr-Al-N is investigated in detail combining experimental and computational studies. Understanding the synthesis-structure-property relations in such 'model-systems' is indispensable to support the massive and collective move in coating industry towards the use of quarternary and multinary nitrides to be followed by carbides, borides, oxides, and oxy-nitrides.

8:40am **B6-1-3 Thermal Decomposition of Arc Evaporated ZrAlN Thin Films**, ***L. Rogström*** *(linro@ifm.liu.se), L.J.S. Johnson,* Linköping University, Sweden*, M. Johansson,* SECO Tools AB, Sweden*, T. Myrtveit,* Sandvik Tooling AB, Sweden*, L. Hultman, M. Odén*, Linköping University, Sweden

Age hardening in thin films has previously been seen for the TiAlN system where the solid solution fcc-TiAlN decomposes into fcc-TiN and hcp-AlN at elevated temperatures. The ZrAlN system is less studied but since ZrN and AlN are immiscible a phase separation can be expected. We have in this study analyzed the decomposition of ZrAlN thin films. $Zr_{1-x}Al_xN$ thin films were deposited onto WC-Co substrates using an industrial arc evaporation system. Samples with an Al content of $0<x<0.84$ as determined by Elastic Recoil Detection Analysis (ERDA) were obtained by the use of cathodes with different Zr:Al ratio. Fractured cross sectional scanning electron micrographs show that the films have a dense and columnar structure. Further analyzes by transmission electron micrographs show that the as deposited films have a nanocrystalline structure with grain sizes in order of 5 nm. In the corresponding SAED pattern an hcp structure with no amorphous phase for the high Al containing films is observed. From X-ray diffractograms a change from fcc to hcp structure occur at Al contents higher than $x>0.35$ in the as deposited films. After deposition the samples were annealed at temperatures between 700 and 1100 °C in two hours respectively in an Ar atmosphere. The hardness of the as deposited and post annealed films was measured using nanoindentation. The hardness for the as deposited films changes with the Al content. A small amount of Al in the films lowers the hardness while a high Al content increases the hardness compared to ZrN. For the post annealed films the hardness is seen to increase with annealing temperature for high Al contents. X-ray diffractograms of the post annealed high Al content films show phase separation into fcc-ZrN and hcp-AlN.

9:00am **B6-1-4 Growth, Annealing Behaviour and Cutting Performance of (Ti,Si)(C,N) Coatings**, ***L.J.S. Johnson*** *(larsj@ifm.liu.se), L. Rogström,* Linköping University, Sweden*, M. Johansson,* SECO Tools AB, Sweden*, M. Collin,* Sandvik Tooling AB, Sweden*, J. Sjölén,* SECO Tools AB, Sweden*, M. Odén, L. Hultman*, Linköping University, Sweden

$(Ti_{1-x}Si_x)(C_yN_{1-y})$ thin films have been deposited by reactive cathodic arc evaporation to investigate the influence of the addition of carbon to the widely studied Ti-Si-N system. Structure characterization by x-ray diffraction (XRD) revealed a single NaCl phase with a lattice parameter close to that of TiCN and composition analysis by elastic recoil detection analysis (ERDA) and energy dispersive x-ray spectroscopy (EDS) showed that Si ratios from $x = 0$ to $x = 0.13$ and C ratios from $y = 0$ to $y = 0.27$ were obtained. Microstructural characterization by transmission electron microscopy (TEM) revealed two distinct microstructures; one dense columnar with large grains (width of around 100-500 nm) for low Si contents, the other a fine "feathered" columnar structure for Si rich films. The hardness, as measured by nanoindentation, is influenced by both the silicon and the carbon content. After annealing at 700, 800, 900, 1000 and 1100°C for two hours each the resulting structure, composition, stress state and microstructure were investigated. No phase transformation was detected by XRD, but a significant reduction in silicon content was detected in the films at temperatures at 1000°C and above. This effect was exacerbated by the addition of carbon. Age hardening of the films was observed which increased in magnitude with carbon content, the nature of which was studied by analytical TEM. A series of cutting tests were performed with as-deposited coatings by longitudinal turning, in which the coatings with a silicon fraction $x = 0.07$ performed best. The worn coatings have also been characterized by analytical TEM.

9:20am **B6-1-5 Microstructure, Mechanical and Tribological Properties of Cr-C-N Coatings Deposited by Pulsed Closed Field Unbalanced Magnetron Sputtering**, ***Z.L. Wu***, Colorado School of Mines, and Dalian University of Technology, China*, J. Lin, J.J. Moore (jjmoore@mines.edu),* Colorado School of Mines*, M.K. Lei*, Dalian University of Technology, China

Nanocrystalline Cr-C-N coatings were deposited by pulsed closed field unbalanced magnetron sputtering (P-CFUBMS). The microstructure,

composition and elemental chemical state of the coatings were investigated using transmission electron microscopy (TEM), glancing incident angle x-ray diffraction (GIXRD) and x-ray photoelectron spectroscopy (XPS). Mechanical and tribological properties of the coatings were measured by nanoindentation, Rockwell C, and ball-on-disk wear tests. The Cr-C-N coatings consist of nanocrystalline Cr_3C_3, Cr_2N compounds embedded in an amorphous carbon and CN matrix. An increase in the hardness and a decrease in the elastic modulus of Cr-C-N coatings were identified when the N content was increased, thereby achieving a high H/E ratio of 0.098 at a N content of 24.9 at.%. The steady state dry coefficient of friction values for Cr-C-N coatings sliding against a WC-Co ball were found in the range of 0.38-0.56. The wear rates of the co atings are in the low range of 1.28-3.44×10^{-6} $mm^3N^{-1}m^{-1}$. The paper will discuss the correlation between pulsing regime, microstructure and properties of the coatings.

9:40am **B6-1-6 Comparative Investigation of TiAlC(N), TiCrAlC(N), and CrAlC(N) Coatings Deposited by Sputtering of MAX-Phase $Ti_{2-x}Cr_xAlC$ Targets**, ***D.V. Shtansky*** *(shtansky@shs.misis.ru), Ph.V. Kiryukhantsev-Korneev, A.N. Sheveyko, D.I. Sorokin,* State Technological University "Moscow Institute of Steel and Alloys", Russia*, B.N. Mavrin,* Institute of Spectroscopy of RAS, Russia*, C. Rojas, A. Fernandez,* Instituto de Ciencia de Materiales de Sevilla, Spain*, E.A. Levashov*, State Technological University "Moscow Institute of Steel and Alloys", Russia

Previous work has demonstrated that various multicomponent nanostructured films with enhanced chemical, mechanical, and tribological properties can be deposited by sputtering of composite targets produced by self-propagating high-temperature synthesis (SHS). SHS allows fabricating ceramic targets with required chemical composition, toughness, and resistance to thermal-cycling, i.e. various properties needed for PVD targets. It is well known that MAX phases $M_{n+1}AX_n$ (n=1-3) are easily machinable, resistant to thermal shock, high temperature oxidation, and thermally and electrically conductive. This combination of properties makes them perspective materials as targets for deposition of hard tribological TiAlC(N), TiCrAlC(N), and CrAlC(N) coatings resistant to corrosion and high-temperature oxidation. The objective of the present study is a comparative investigation of the structure and properties of TiAlC(N), TiCrAlC(N), and CrAlC(N) coatings deposited in an Ar atmosphere or in a gaseous mixture of Ar+N_2 by sputtering of SHS targets based on the MAX-phases in the system $Ti_{2-x}Cr_xAlC$ (where x=0, 0.5, 1.5, and 2). The coatings were characterized in terms of their structure, elemental and phase composition, adhesion, hardness, elastic modulus, elastic recovery, thermal stability, friction, wear-, corrosion and high-temperature oxidation resistance. The structure of the coatings was studied by means of X-ray diffraction, scanning and transmission electron microscopy, X-ray photoelectron spectroscopy, glow discharge optical emission spectroscopy, and Raman spectroscopy. To evaluate the thermal stability and oxidation resistance, the coatings were annealed either in vacuum or in air in the temperature range 600-1000°C. The results obtained show that the Cr-doped coatings obtained under thoroughly controlled deposition parameters possess high hardness up to 35 GPa, improved oxidation resistance up to 1000°C, thermal stability up to 800°C, and good corrosion resistance. With increased chromium content the (Ti,Cr)-Al-C-N coatings demonstrated improved adhesion strength and oxidation resistance, whereas their friction coefficient against WC+Co counterpart increased from 0.3 to 0.5 and the hardness decreased down to 25 GPa.

10:00am **B6-1-7 The Effect of Magnetron Pulsing on the Structure and Properties of Nanostructured Multifunctional Tribological Coatings**, ***J.J. Moore*** *(jjmoore@mines.edu), J. Lin, B. Mishra,* Colorado School of Mines*, W.D. Sproul,* Reactive Sputtering, Inc.*, J.A. Rees*, Hiden Analytical, LTd., United Kingdom **INVITED**

The paper will discuss the effect of pulsing unbalanced magnetrons used in three systems: (i) single magnetrons, (ii) in a closed field configuration, and (iii) in a high energy pulsing (Modulated Pulsed Power) system on the structure and properties of tribological coatings. In particular, the effect of pulsing regime (e.g, frequency, duty cycle, etc) on the plasma species and ion energy distributions (IED) will be discussed and correlated with the microstructure and tribological properties of the thin films and coatings for Cr, Al, Ti, graphite and composite TiC-TiB2 targets.

The main objective of this research is to determine the pulsing regimes that produce optimized coatings for specific applications, such as aluminum pressure die casting, and glass molding operations. In this respect the optimization of graded, multi-layer Cr-Al-N, and nanocomposite Ti-C-B-N and Ti-C coatings will be used as the main examples.

The application of Modulated Pulsed Power (MPP) deposition (an alternative to HIPIMS/HPPMS) will be discussed using some recent results.

10:40am **B6-1-9 Nano-Structured CrN/AlN Superlattice Coatings Synthesized by Pulsed Closed Field Unbalanced Magnetron Sputtering**, *J. Lin (jlin@mines.edu),* ***B. Mishra,*** Colorado School of Mines*, M. Pinkas,* Nuclear Research Center, Israel*, J.J. Moore,* Colorado School of Mines*, W.D. Sproul*, Reactive Sputtering, Inc.

Chromium nitride/aluminum nitride (CrN/AlN) superlattice coatings were prepared using a pulsed closed field unbalanced magnetron sputtering system from pure Cr and Al targets. The bilayer periods of the coatings were obtained between 2.0 to 15 nm by controlling the target powers and the substrate rotation speed. The effects of the bilayer period (especially in the 2-7 nm range) on the structure and properties of the CrN/AlN coatings were characterized by means of low angle and high angle X-ray diffractions, scanning electron microscopy, transmission electron microscopy, nanoindentation, Rockwell C indentation, and ball-on-disk wear tests. All CrN/AlN superlattice coatings synthesized in the current study exhibit a single phase face-centered cubic structure. Compared with the homogeneous $Cr_{0.4}Al_{0.6}N$ coatings, significant improvements on the hardness and wear resistance were achieved in the CrN/AlN superlatice coatings. The CrN/AlN superlattice coatings exhib it super hardness above 40 GPa in a wide range of bilayer period of 2.7~4 nm, where the highest hardness of 45 GPa was achieved when the bilayer period is at 2.7 nm. When the bilayer period is between 2.7 to 5.5 nm, the nanolayered coatings also showed low coefficient of friction in the range of 0.3 to 0.35 and low wear rate in the 10^{-7} $mm^3N^{-1}m^{-1}$ range. The CrN/AlN superlattice coatings also exhibit lower residual stress, improved adhesion and toughness as compared to those of the homogeneous CrAlN coatings.

11:00am **B6-1-10 Thermally Enhanced Mechanically Properties of Arc Evaporated TiN/TiAlN Multialyer Thin Films**, ***A. Knutsson*** *(knutsson@ifm.liu.se),* Linköping University, Sweden*, M. Johansson,* SECO Tools AB, Sweden*, M. Odén*, Linköping University, Sweden

The cubic phase $Ti_{1-x}Al_xN$ has been used to coat cutting tools since the late 1980's. It has been shown that the excellent tool performance is closely related to age hardening of c-$Ti_{1-x}Al_xN$ where it decompose to c-TiN and c-AlN at elevated temperature. In this work the possibility to control the decomposition temperature of $Ti_{1-x}Al_xN$ with help of multilayers is investigated. Cubic metastable $Ti_{0.34}Al_{0.66}N$ / TiN 3µm thick multilayers were grown by reactive arc evaporation using $Ti_{0.33}$-$Al_{0.67}$ and Ti cathodes in a N_2 atmosphere.

The difference in decomposition between multilayers with different layer thickness (TiN/TiAlN: 50/25, 25/12 and 10/5 nm) and single layer $Ti_{0.34}Al_{0.66}N$ was investigated using differential scanning calorimetry (DSC) up to 1400°C. The results revealed that the phase transformations in the multilayers are initiated at higher temperatures with decre ased $Ti_{0.34}Al_{0.66}N$ thickness, suggesting a higher thermal stability compared to single layers.

The hardness increased with decreased layer period which is to be expected from Koehler and similar multilayer effects. Despite the 75 vol% TiN in the as deposited 15/5 nm multilayer a slightly higher hardness was observed compared to single layer $Ti_{0.34}Al_{0.66}N$. In addition, the multilayers exhibit a more significant age hardening effect than the single layer when annealed to 900°C, with a hardness increase of ~45 %. The hardening phenomena are discussed in terms of particle constraints from neighboring TiN-layers.

11:20am **B6-1-11 Characterization of the Adhesion and Tribology of Ti/TiAlN Multilayer PVD Coatings Deposited on Pre-Nitrided Tool Steels**, *W. Tillmann (wolfgang.tillmann@udo.edu), E. Vogli,* ***S. Momeni***, Dortmund University of Technology, Germany

Employing multilayer PVD coating including ceramic and metallic inter-layers can enhance wear resistance and fracture toughness of coating systems. It is also known that the performance of the PVD coating can be enhanced by increasing hardness of the substrate. In this research work, different alloy steels were hardened using plasma nitriding process in the Arc-PVD device. The nitrided zones on steels were studied by means of XRD analysis. Afterwards, on both unnitrided and plasma nitrided substrates, different Ti/TiAlN multilayer coatings were deposited by a magnetron sputtering device. Scratch tests were performed on the duplex systems in order to characterize their adhesive properties. The failure modes of individual coating systems under various normal loads were described using light microscope, scanning electron microscope and EDX analysis. Furthermore, to study the effect of plasma nitriding on the friction coefficient and wear rate of the systems, pin-on-disc tests were carried out.

11:40am **B6-1-12 Characteristics of Cr_2N/Cu Multilayered Thin Films with Different Bilayer Thickness**, ***C.-L. Li***, *J.-W. Lee (cwlee@mail.tnu.edu.tw),* Tungnan University, Taiwan*, L.-C. Chang*, Mingchi University of Technology, Taiwan

Nanostructured Cr_2N/Cu multilayer coatings were deposited periodically by a bipolar asymmetric pulsed DC reactive magnetron sputtering technique. The structures of multilayer coatings were characterized by an X-ray

diffractometer. The surface and cross sectional morphologies of thin films were examined by a scanning electron microscopy (SEM) and transmission electron microscopy (TEM), respectively. The surface roughness of thin films was explored by an atomic force microscopy (AFM). The nanohardness and elastic modulus of multilayer coatings were investigated by means of a nanoindenter. The scratch and wear testers were used to evaluate the tribological properties of thin films. The electrochemical tests in 3.5 wt. % NaCl aqueous solution were performed to evaluate the corrosion resistance of multilayered coatings with different bilayer thickness. It is observed that coatings with bilayer thickness ranges from 5 nm to 40 nm were produced in this work. The surface roughness of the multilayered coating decreased with increasing bilayer thickness. An optimal hardness and corrosion resistance were found on the coating with a critical bilayer thickness of 10 nm.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
Room: Royal Palm 4-6 - Session D3-2

Carbon and Nitrogen-Containing Nanostructured Composite and Nanolaminated Films
Moderator: U. Jansson, Uppsala University, Y. Pauleau, Grenoble Polytechnic Institute, J.M. Ting, National Cheng Kung University

8:00am **D3-2-1 The MAX Phases and Kinking Non-Linear Elastic Solids; A Newly Identified Class of Solids**, ***M.W. Barsoum*** *(barsoumw@drexel.edu)*, Drexel University **INVITED**

The layered, hexagonal carbides and nitrides with the general formula: $M_{n+1}AX_n$, (MAX) where n = 1 to 3, M is an early transition metal, A is an A-group (mostly IIIA and IVA) element and X is either C and/or N combine some of the best attributes of metals and ceramics. Like metals, they are electrically and thermally conductive, most readily machinable (manual hack saw will suffice) not susceptible to thermal shock, plastic at high temperatures, and exceptionally damage tolerant. Like ceramics, they are elastically rigid, lightweight, and maintain their strengths to high temperatures. The ternaries Ti_3SiC_2 and Ti_2AlC are creep, fatigue and oxidation resistant. More recently we have also shown that the MAX phases are but a subset of solids that we termed kinking nonlinear elastic, KNE, because one of their important – and in many cases only - deformation mode is the formation of fully reversible, dislocation-based incipient kink bands, IKBs. We further claim, and present compelling evidence, that most if not all solids with c/a ratios > 1.5 - which per force are plastically anisotropic – will deform by kinking. KNE solids include most layered solids, such as mica, $LiNbO_3$, Mg, Ti, Zn, Co, and other hexagonal metals, h-BN, GaN, MoS_2, as well as sapphire, among many others. Given the diversity and ubiquity of KNE solids it is clear that incipient IKBs play a much more important role in our daily life than has hitherto been appreciated. Based on the totality of our work it is now clear that IKBs are one of the last, but crucial, missing pieces in the deformation-of-solids puzzle. The implications of these conclusions to researchers working in the area of thin films will be touched upon.

8:40am **D3-2-3 Electronic Structure Investigation of MAX-Phases by Soft X-ray Emission Spectroscopy**, ***M. Magnuson*** *(Martin.Magnuson@ifm.liu.se),* Linköping University, Sweden, *U. Jansson,* Uppsala University, Sweden, *L. Hultman*, Linköping University, Sweden

The electronic structures of epitaxially grown films of MAX-phases were investigated by soft X-ray emission spectroscopy. Specifically, the symmetry characteristics in the interior of these nanolaminate carbide and nitride compounds have been revealed[1]. The bulk-sensitive soft X-ray emission technique is shown to be particularly useful for obtaining detailed electronic structure information about internal monolayers and interfaces[2]. A weak covalent Ti-Al bond is manifested by a pronounced shoulder in the Ti L-emission of Ti3AlC2, Ti2AlC and Ti2AlN. When Al is replaced by Si or Ge, the shoulder disappears. Furthermore, the spectral shapes of Al, Si and Ge in the MAX-phases are strongly modified in comparison to the corresponding pure elements[3]. Measurements on V2GeC via V L2,3, C K, Ge M1 and Ge M2,3 spectra, including polarization variation in the excitation process, permits detailed decomposition of the valence electronic structure and chemical bonding in the interior of the materials. The macroscopic properties of the V2GeC nanolaminate result from the chemical bonds with the anisotropic pattern as shown in this work. The measured X-ray emission spectra are compared and interpreted with ab initio density-functional theory including core-to-valence dipole matrix elements. The calculated results are found to yield consistent spectral functions to the experimental data. By varying the constituting elements, a change of the electron population is achieved causing a change of covalent bonding between the laminated layers, which enables control of the macroscopic properties of the material.

[1]Anisotropy in the electronic structure of V2GeC investigated by soft x-ray emission spectroscopy and first-principles theory, M. Magnuson, O. Wilhelmsson, M. Mattesini, S. Li, R. Ahuja, O. Eriksson, H. Högberg, L. Hultman and U. Jansson; Phys. Rev. B 78, 035117 (2008).

[2]Bonding mechanism in the nitrides Ti2AlN and TiN: An experimental and theoretical investigation, M. Magnuson, M. Mattesini, S. Li, C. Höglund, M. Beckers, L. Hultman and O. Eriksson; Phys. Rev. B., 76, 195127 (2007).

[3]Electronic structure investigation of Ti3AlC2, Ti3SiC2, and Ti3GeC2 by soft-X-ray emission spectroscopy, M. Magnuson, J. -P. Palmquist, M. Mattesini, S. Li, R. Ahuja, O. Eriksson, J. Emmerlich, O. Wilhelmsson, P. Eklund, H. Högberg, L. Hultman and U. Jansson; Phys. Rev. B 72, 245101 (2005).

9:00am **D3-2-4 A Computational Study of the Effects of Impurities on MAX Phase Elastic Properties**, ***M.F. Cover*** *(myles@physics.usyd.edu.au), M.M.M. Bilek, D.R. McKenzie*, University of Sydney, Australia

The MAX phases are a class of nanolaminate materials with a unique combination of ceramic and metallic properties. MAX phases mimic ceramics in that they are stiff, resistant to oxidation, and remain strong at temperatures exceeding 1400 °C. The metal like properties of MAX phases manifest themselves in their machinability, resistance to thermal shock, high damage tolerance, and electrical and thermal conductivity. This unique combination of properties suggests them as structural materials for demanding operating environments.

$M_{n+1}AX_n$ phases are composed of three elements: an early transition metal (M), a main group element (A), and either carbon or nitrogen (X). They are characterised by a nanolaminate structure in which slabs of the binary carbide/nitride (MX) are separated by single atomic layers of the main group element.

Experimentally prepared MAX phases however contain as impurities elements not in the MAX phase formula, with oxygen and hydrogen being the most common. In this work we use first principles density functional theory calculations to investigate the effects of these impurities on the MAX phases elastic properties. Elastic properties are of interest for MAX phases as they underpin macroscopic properties such as lubrication, friction, and machinability, which are important for structural applications. We calculate the elastic constants of MAX phases with an increasing impurity content, taking into account the effect of the location of the impurities in the MAX phase lattice. We also investigate the mobility of these impurities within the MAX phase structure.

9:20am **D3-2-5 Mechanical Deformation Properties of Ti_2AlC MAX Phase Thin Films with High Oxygen Content**, ***A. Mockute*** *(aurmo@ifm.liu.se), P. Persson, F. Giuliani, L. Hultman,* Linköping University, Sweden, *M.M.M. Bilek,* University of Sydney, Australia, *J. Rosén*, Linköping University, Sweden

High current pulsed cathodic arc has been used for synthesis of Ti_2AlC MAX phase thin films. The films were deposited at 700, 800, and 900°C, and were characterised with respect to composition, structure, and mechanical properties, by means of ERDA, XRD, FIB, TEM, and nanoindentation. Deformation mechanisms in the material were also investigated. A high oxygen incorporation of 7-12 at.% was detected in all the films, likely originating from residual gas and the Al_2O_3 substrate. The highest growth temperature resulted in 0001-oriented single-crystal material. Resulting elastic modulus and hardness of 259 GPa and 16 GPa, respectively, was determined by nanoindentation using a Berkovich tip. Analysis of loading-unloading curves and scanning probe microscopy images revealed no relation between pop-in events and presence of pile-ups around the residual imprints. This implies that Ti_2AlC MAX phase deformed without kinking and delamination, as opposed to observations in related oxygen-free single-crystal Ti_3SiC_2 (0001) thin films. This is also corroborated by cross-sectional TEM investigation of an indent. Reasons for the different deformation mechanisms observed are discussed, together with the effect of impurity incorporation on the MAX phase material properties.

9:40am **D3-2-6 The Effects of Surface Structure, Incident Ion Energy, and Impurity Incorporation on MAX Phase Nucleation and Growth**, ***M.D. Tucker*** *(tucker@physics.usyd.edu.au), M.C. Guenette, P.O.Å. Persson, J. Rosén, M.M.M. Bilek, D.R. McKenzie*, University of Sydney, Australia

The MAX phase materials are a class of nanolaminated three-element compounds that have been found to possess an unusual combination of ceramic and metallic material properties. Currently, the mechanisms

involved in the growth of these materials as thin films are not well understood. An interlayer between the substrate and the MAX phase film is commonly used to promote oriented growth. However, several reports have been published of successful MAX phase growth without the use of an interlayer. The orientation of the substrate has also been found to be of critical importance, both for nucleating MAX phase growth, and in affecting its orientation.

We present a systematic investigation of the substrate influence on the growth of MAX phases in our pulsed cathodic arc deposition system. X-ray diffraction has been used to determine phases present and their orientation, and cross-sectional transmission electron microscopy has been used to give further information on the film growth modes and the resulting film microstructure. Deposition by pulsed cathodic vacuum arc has the advantage that the arc plasma is entirely ionized, allowing the energy of the deposited species at the substrate to be controlled through the manipulation of the substrate bias. We have used this capability to investigate the relationship between the energy of the incident ions and the minimum temperature at which the MAX structure nucleates.

Our previous work has shown that the Ti_2AlC MAX phase structure is capable of accommodating substantial oxygen impurities. We have used X-ray absorption near edge spectrosopy (XANES) to examine oxygen bonding in order to positively identify the impurity locations within the MAX structure, and here we present results from this investigation.

10:00am **D3-2-7 Microstructural Modifications of TiX(X=C or N)/TiAl Multilayers During Thermal Annealing and Ion-Irradiation**, ***T. Cabioch*** *(thierry.cabioch@univ-poitiers.fr), M. Bugnet, M. Jaouen*, University of Poitiers, France

TiX(X=C or N)/TiAl multilayers of various modulation wavelengths were deposited at room temperature onto Si(100) substrates or Al_2O_3(001) either by Ion Beam Assisted Deposition or Magnetron sputtering. The microstructural modifications induced by thermal annealing under vacuum (600-900°C) as well as those induced by ion-irradiation were studied by the use of several characterization techniques (X-Ray diffraction (XRD), High Resolution Transmission Electron Microscopy (HRTEM), Energy Filtered Transmission Electron Microscopy imaging and X-Ray Photoelectron Spectroscopy (XPS) experiments).

In the case of TiN/TiAl multilayers the formation of a MAX Phase was achieved during the thermal annealing so that a (Ti,Al)N/Ti_2AlN multilayer structure was obtained (Whereas only (Ti,Al)C was obtained for the TiC/TiAl system). XRD and HRTEM characterization of the irradiated samples revealed the very strong resistance under ion-irradiation of TiX (X=C or N) but also of the MAX Phase Ti_2AlN.

10:20am **D3-2-8 Ion Beam-Assisted Pulsed Laser Deposition from a Ti_3SiC_2 MAX-Phase Target**, *C. Lange (clange1@uni-goettingen.de),* Georg-August-Universität Göttingen, Germany*, J. Schawohl, M. Wilke,* TU Ilmenau, Germany*, M.W. Barsoum,* Drexel University*,* ***P. Schaaf***, TU Ilmenau, Germany

Pulsed laser deposition with a Nd:YAG laser was used to grow thin films from a pre-synthesized Ti_3SiC_2 MAX-phase formulated ablation target on oxidized Si(1 0 0), MgO(1 0 0) and stainless steel substrates with and without an 200 V Ar ion beam directed at the substrate surface during deposition. The depositions were carried out in a substrate temperature range from room temperature to 650 °C at a chamber pressure of 10^{-5} Pa without ion beam and at 10^{-2} Pa Ar background pressure when the ion beam was applied. The properties of the films have been investigated by glow discharge optical emission spectroscopy (GDOES) for film thickness and stoichiometric composition, X-ray diffraction for the crystallinity of the films and X-ray photoelectron spectroscopy (XPS) at the Ti 2p core level. X-ray diffraction measurements for all samples show no signs of the MAX phase Ti_3SiC_2, but only reflections of crystalline TiC. Also a TiC doubl et is seen in the Ti 2p XPS measurements. GDOES measurements show that the films do not represent the desired stoichiometric Ti_3SiC_2 composition. Instead the silicon content is decreased to about 11 at.-% and below and a quite high oxygen content of about 7 at.-% and more is observed. Obviously, silicon is oxidized by the residual oxygen during deposition and partially lost due to the formation of volatile SiO.

10:40am **D3-2-9 Elastic and Electrical Properties of Thin Film MAX Phase Alloys Deposited by Pulsed Cathodic Arc**, ***M.C. Guenette*** *(guenette@physics.usyd.edu.au), M.D. Tucker,* University of Sydney, Australia*, M.F. Cover,* University of Sydney, Australis*, M.M.M. Bilek, D.R. McKenzie*, University of Sydney, Australia

MAX phases have attracted significant attention in recent years due to their unique combination of ceramic and metallic like properties. Deposition of the correct composition is critical for successful growth of MAX phase thin films. Their hexagonal, nanolaminate structure leads to a strong anisotropy in their elastic properties. The elastic properties of MAX phases underpin important macroscopic properties such as stiffness, lubrication, friction and machinability, with C_{44} strongly influencing friction and machinability properties while C_{11} and C_{33} influence the stiffness for example. Elastic properties such as the average bulk modulus have been reported in the literature for several MAX phases, however there have been no reports of determining the directional dependence of the elastic constants experimentally. The techniques we employ for the deposition of M_2AlC thin films (for various M) by pulsed cathodic arc will be described. Control/quant ification of the film composition using secondary neutral mass spectrometry (SNMS) and crystal structure analysis using X-ray diffraction (XRD) will be outlined. The relationship between electrical conductivity measurements and film composition will be presented. Thermal diffuse scattering of x rays is a technique suitable for studying elastic waves in small samples of crystalline materials. The technique and its application to the measurement of key elastic constants of MAX phases will be discussed.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E4/G4

Coatings for Machining Advanced Materials and Advanced Manufacturing Methods

Moderator: W. Kalss, Oerlikon Balzers Coatings, H.-G. Fuss, CemeCon A.G.

8:00am **E4/G4-2 Mechanical Properties and Cutting Performance of Nanocomposite Cr-Si-N Coated Micro-Tool for Green Machining of Flexible Fine Die**, ***M.W. Kim***, Pusan National University, Korea*, K.T. Ryu,* One Plus Two Co. Ltd, Korea*, M.C. Kang (kangmc@pusan.ac.kr)*, Pusan National University

Green machining means the use of the so-called dry machining technology and also semi-dry machining with minimal quantity of lubricant (MQL). In this paper, comparative studies on mechanical properties and cutting performance between CrN and Cr-Si-N coatings for green machining of flexible fine die were conducted. Ternary Cr-Si-N coatings, in which Si was incorporated into CrN, were synthesized onto WC-Co substrates using a hybrid system of arc ion plating and sputtering techniques. The high hardness of Cr- Si-N coatings was related to the composite microstructure consisting of the fine CrN crystallites and amorphous Si3N4. The average friction coefficient of Cr-Si-N coatings gradually decreased with increase of Si content in CrN coatings. The values of tool wear for coated tools were evaluated under a high-speed cutting condition using vertical high-speed machining center (Makino, V-55). It is found that cutting under flood coolant condition results in the shortest tool life due to severe thermal cracks while the use of MQL leads to the best performance. MQL is beneficial to tool life both in the CrN and Cr-Si-N coating tools. This conclusion proves the feasibility in the ultra-precision machining of high–hardened materials for flexible fine die.

8:20am **E4/G4-3 Adaptive PVD Coating for Machining of Hard to Cut Materials**, ***G. Fox-Rabinovich*** *(gfox@mcmaster.ca),* McMaster University, Canada*, K. Yamamoto,* Kobelco, Japan*, S. Veldhuis*, McMaster University, Canada **INVITED**

New generation of adaptive PVD coating for high performance machining of hard to cut materials is presented. Adaptation during cutting is a complex process that is related to generation of the surface tribo-films with unique protective/lubricious ability in synergy with beneficial structure and properties transformations within the layer of the nano-structured coatings as well. If all these parameters work together then the coating is capable to sustain strongly varying and intensifying external impacts with unattainable tool life. A number of novel adaptive hard coatings is developed best suited for specific applications associated with machining of hardened tool steels as well as aerospace materials such as nickel-based superalloys and Ti-based alloy. Results of long-term research of adaptive TiAlCrN-based coatings (nano-crystalline and nano-multilayered) is presented. Comprehensive investigation of the structural characteristics of the coating is made using XRD, TEM, SEM/EDX. Micro-mechanical properties of the coating such as hardness, micro-hardness dissipation parameter, impact fatigue fracture resistance are studied at RT and elevated temperatures using Micro Materials Test System. Tribological characteristics of the coatings are investigated vs. temperature in contact with corresponding materials. Characteristics of the tribo-films are studied in detail using XPS, EELFAS and EELS methods. Tool life and wear behavior are investigated for specific operating conditions. Wear patterns are indentified. Based on the

data obtained some principles of adaptive hard coating development are outlined for specific applications.

9:00am **E4/G4-5 Crystalline γ-Alumina Deposited in an Industrial Coating Unit for Demanding Turning Operations**, *K. Bobzin, N. Bagcivan, P. Immich,* ***M. Ewering*** *(ewering@iot.rwth-aachen.de)*, RWTH Aachen University, Germany

Crystalline PVD γ-Al_2O_3-coatings offer great potential for their use in high-speed cutting operations. They promise high hot hardness and high oxidation resistance at elevated temperatures. This is important for coatings which are used for machining of materials with low thermal conductivity like stainless steel or Inconel 718 because heat, generated during cutting, can hardly be dissipated by the chip. Because of the prevailing bonding forces of alumina adhesion-related sticking can be reduced, even for dry cutting. Furthermore, the high formation enthalpy of alumina prevents chemical reactions with frictional partners. The present work gives an overview of the deposition of γ-Al_2O_3-thin films on WC/Co-cutting inserts by using pulsed MSIP (Magnetron Sputter Ion Plating) PVD technology. To improve adhesion a (Ti,Al)N bond coat was employed. The samples were analyzed using common thin film test equipment such as scratch test, calo-test , X-ray diffraction, Scanning Electron Microscopy (SEM) and nanoindentation for the mechanical properties. It could be proved, that the developed coatings show excellent premises for their use in cutting operations. In order to proof the coating's performance, cutting tests were carried out. For drilling operations the difficult-to-machine austenitic steel 1.4301 (X5CrNi18-10) was used. In comparison to a state-of-the-art (Ti,Al)N coating the (Ti,Al)N/γ-Al_2O_3 showed a longer tool life.

9:20am **E4/G4-6 Investigating the Performance of TiN and AlTiN Coatings on Milling Cutter Used for Machining Bimetal Steel**, *M. Sarwar,* ***J. Haider*** *(julfikar.haider@northumbria.ac.uk),* Northumbria University, United Kingdom*, M. Persson, H. Hellbergh*, SNA Europe, Sweden

Surface engineering of cutting tools (single point or multipoint) through advanced coatings (e.g., TiN) has contributed towards the improvement of tool life, productivity and machining quality@super 1@ by modifying the substrate. New coating species (e.g., AlTiN) are also being developed to further improve the performance of cutting tools@super 2@. In this study, machining tests were carried out with a Powder Metallurgy High Speed Steel (PM HSS) milling cutter when cutting bimetal (M42 + D6A) steel strips to produce bandsaws. The milling cutter was modified with TiN and AlTiN coatings in order to evaluate their performances. Physical Vapour Deposition (Arc evaporation) technique was used to deposit the coatings after carefully preparing the cutting edges. Failure modes and mechanisms of the milling cutter have been identified by examining the worn cutting edges. Flank wear measurement in the milling cutter teeth was used as the criterion for assessing the performance of coatings. The properties of the coatings were evaluated to correlate with the performance. The product quality of the bandsaw teeth formed by the milling cutter was also studied. The information should be useful for material suppliers, tool designers and tool users.

@super 1@M. Sarwar, D. Gillibrand, S.R. Bradbury, Forces, surface finish and friction characteristics in surface engineered single- and multi-point cutting edges, Surf. Coat. Technol., Vol. 41 (1991) 443-450. @paragraph2@super 2@W. Kalss, A. Reiter, V. Derflinger, C. Gey and J.L. Endrino, Modern coatings in high performance cutting applications, Int. J. Refract. Met. Hard Mater., Vol. 24 (2006) 399-404.

9:40am **E4/G4-7 Process Variability in Honing of Cylinder Liner with Vitrified Bonded Diamond Tools**, ***L. Sabri*** *(leila.sabri@renault.com),* Renault, France*, S. Mezghani, M. El Mansori*, Arts et Métiers Paris Tech, France

A fundamental investigation is reported on the variability in honing behaviour due to stone inconsistency and nature of its properties. The conventional abrasives used in honing are indeed bodies consisting of Silicon Carbide grits and Vitrified Bond bridge (VBSC). Theses stones are soft and have an openness structure for a permanent "resharpening" of grains and high chip accommodation. Even if VBSC stones produce high surface quality, they are not suitable for high production usage. The use however of superabrasives stones such as Metallic Bonded Diamond (MBD) sticks fulfil the tool life requirement but generate some background texture harmful for the reciprocating mechanical components of the engine.

This study discusses the honing process variability with Vitrified Bonded Diamond (VBD) stones compared to the VBSC one. An experiment test rig is developed consisting of industrial honing machine instrumented with sensors to measure spindle power, expansion pressu re and honing head displacement. Microchips and honed surfaces are also investigated with Scanning Electron Microscopy (SEM). Moreover, a surface multi-scale characterization approach is introduced to assess deeply the effects of stone's properties on the quality of honed surfaces. The results show a large influence of the expansion velocity in correlation to the depth of indentation per grit. Considerable variability in honing behaviour observed is related to the mechanics of the process.

10:00am **E4/G4-8 Influence of Nanostructured CVD Diamond Coatings During Dry Turning of a SiC Particle-Reinforced Metal Matrix Composite**, ***A. Kremer*** *(arnaud.kremer@chalons.ensam.fr), M. El Mansori*, Arts et Métiers Paristech, France

This paper reports on a experimental test program to study the effect of the coating structure associated with the machining of a particle-reinforced metal matrix composite (PRMMC). The composite materials investigated were respectively a 5%, 15% and 25% by volume SiC particle-reinforced A2009 aluminium alloy. The machining of these materials is indeed very difficult due to the abrasive effect of the ceramic reinforcement upon the cutting tool. In this work, the ability of coated tungsten-carbide tools with nanostructured CVD diamond coatings to cut PRMMC components is discussed during dry machining. The performances of three different structures of CVD diamond coating are then considered using classical parameters (cutting forces, tool life, residual stress) and innovative criterion such as dust emission. The results show that in spite of their similar thickness, the coatings have various performances. The tool life was varied in a ratio of 1 to 6 irrespectively to the cumulative wear evolution which was monitored in situ with a power measurement and quantified ex situ using a white light interferometer.

10:20am **E4/G4-9 Hot Filament Diamond CVD – Technology and Applications**, ***W. Reichert*** *(walter.reichert@cemecon.de), R. Cremer, O. Lemmer*, CemeCon AG, Germany

Hot filament CVD is a well-established technology for coating of tools and other three-dimensional components with microcrystalline and nanocrystalline diamond. CemeCons state-of-the-art CC800/9 Dia coating unit produces advanced quality diamond films. Due to its large 3-chamber recipient and the new and easy to use operating software this coating machine uniquely combines high quality, high flexibility and economical production capability. CVD diamond coated tools are used in a variety of applications. To obtain best results the carbide material, tool geometry, coating, and application need to be matched. High precision tools are needed for accurate machining of graphite electrodes. In this case the diameter of the coated tool is of greatest importance. When drilling CFRP parts (carbon fiber reinforced plastics) for aerospace applications sharp cutting edges and abrasion resistant coatings are significant for the overall cutting performance. Similar challenging require ments need to be met when routing printed circuit boards (PCBs) or processing aluminum, MMC and other composite materials. The presentation will show new developments in hot filament technology and cutting test results in different materials.

10:40am **E4/G4-10 Coating Thickness Effects on Diamond Coated Cutting Tools**, ***F. Qin****, Y. Chou (kchou@eng.ua.edu),* The University of Alabama*, D. Nolen, R. Thompson*, Vista Engineering

CVD-grown diamond films are finding applications as a coating for cutting tools. Even though use of conventional diamond coatings seems to be established in the cutting tool industry, selections of proper coating thickness for different machining operations have not been known. Coating thickness affects the characteristics of diamond coated cutting tools in different perspectives that critically and mutually, in a complex way, impact the tool performance in machining. In this study, coating thickness effects on the deposition residual stresses, particularly around a cutting edge, and on coating failure modes were numerically investigated. On the other hand, coating thickness effects on tool surface smoothness and cutting edge radii were experimentally compared. In addition, machining Al matrix composites using diamond coated tools with varied coating thicknesses was conducted to evaluate the effects on cutting forces, part surface finish and tool wear. The results are s ummarized as follows. (1) Increasing coating thickness will increase the residual stresses at the coating-substrate interface. (2) On the other hand, increasing coating thickness will generally increase the resistance to surface cracking and delamination. (3) Thicker coatings have a higher surface roughness and naturally enlarged edge radii, which tend to increase the machining load on the tool surface. (4) Preliminary results show a non-monotonic relation between the coating thickness and diamond coated tool performance.

11:00am **E4/G4-11 Evaluation of Microstructures and Mechanical Properties of HFCVD Diamond-Coated WC-Co Substrates with Hard Chromized Interlayers**, ***C.-C. Chou*** *(cchou@mail.ntou.edu.tw),* National Taiwan Ocean University, Taiwan, *J.-W. Lee,* Tungnan University, Taiwan, *H.-H. Lin, Y.-I. Chen*, National Taiwan Ocean University, Taiwan

Chromized interlayers produced by pack chromization on WC-Co substrates were tested and identified to be a potential microstructure for improving tribological performance of diamond surface coatings in the previous work[1]. However, interlayer's thickness and temperatures of the chromization and hot filament chemical vapor deposition (HFCVD) processes were found critical to the formation and adhesion of the diamond films on WC-Co substrates. The cobalt binder of the WC-Co substrate penetrates across the microcracks on a thick chromized interlayer or diffuse through a thin but crack-free one. The temperature of chromization furnace and HFCVD substrate's surface under higher values accelerate cobalt's diffusion rate and interfere the nucleation and growth of diamond film. All the critical process parameters were systematically studied basing on substrates with two cobalt contents (6wt.% and 12 wt.%). Scratch tests were first implemented to verify the strength between chromized interlayers and substrates. Daimler-Benz Rockwell-C indentation tests were conducted to evaluate the adhesion properties of chromized interlayer themselves and the later diamond-coated structures on the substrates. Compositions of diamond films were verified by X-ray diffractometer (XRD) and Raman spectroscopy. In conclusion, the optimal process parameters and the accompanied microstructure of the diamond coatings were addressed.

[1] Chou, C. C., Lee, J. W., and Chen, Y. I., 2008, "Tribological and Mechanical Properties of HFCVD Diamond-Coated WC-Co substrates with Different Cr Interlayers," Surface & Coatings Technology, in press (available online), http://dx.doi.org/10.1016/j.surfcoat.2008.08.055.

New Horizons in Coatings and Thin Films
Room: Sunset - Session H2-1

High Power Impulse Magnetron Sputtering
Moderator: A.P. Ehiasarian, Sheffield Hallam University, K. Marchev, P&G Company

8:00am **H2-1-1 Target-Plasma-Film Interactions in High Power Pulsed Magnetron Sputtering**, ***K. Sarakinos*** *(sarakinos@mch.rwth-aachen.de)*, RWTH Aachen University, Germany **INVITED**

High power pulsed magnetron sputtering (HPPMS) provides high fluxes of ionized species to the growing film allowing for superior film properties in comparison to those obtained by conventional techniques, such as direct current magnetron sputtering (dcMS). In addition, in HPPMS lower film deposition rates (Rd) than the dcMS ones are commonly obtained. In order to elucidate the mechanisms responsible for these features, the investigation and the understanding of the target-plasma-film interactions in HPPMS are of key importance. Based on experimental studies of an Ar-Cr HPPMS discharge, the lower rates are primarily attributed to the re-direction of ionized sputtered species to the target (self-sputtering). It is shown that the self-sputtering occurs when high degree of ionization of the sputtered species and depletion of Ar species in front of the target (rarefaction) are simultaneously present. This notion is further supported by a semi-quantitative analysis that enables the estimation of the composition of the ion target current during HPPMS of C, Cr and Cu. In the case of reactive HPPMS it is shown that the deposition rate is mainly affected by the target coverage. Using a Zr target in an Ar-O2 ambient, it is demonstrated that HPPMS exhibits a stable transition sputtering zone, as opposed to dcMS. This facilitates growth of films with Rd values up to 2 times higher than in dcMS. Finally, HPPMS is employed for the growth of nitride (CrN) and oxide (TiO_2 and Al_2O_3) films allowing for effective tailoring of films' morphology and phase composition. These findings are discussed in the light of the energetic bombardment provided at the various conditions.

8:40am **H2-1-3 Time Resolved Optical Imaging of HIPIMS Discharges: Selecting Pulse Parameters Based on Target Mass**, ***J.G. Jones***, *C. Muratore,* Air Force Research Laboratory, *A.N. Reed (Amber.Reed.ctr@wpafb.af.mil),* Air Force Research Laboratory/University of Dayton Research Institute, *A.R. Waite,* Air Force Research Laboratory/UTC, Inc./University of Dayton, *C.A. Cerbus,* Air Force Research Laboratory/University of Dayton Research Institute, *S.F. Noss, A.A. Voevodin*, Air Force Research Laboratory

The highly ionized flux associated with high power impulse magnetron sputtering (HIPIMS) can yield unique and useful physical characteristics in thin film materials. Unfortunately, the deposition rate in HIPIMS tends to be lower than in standard magnetron sputtering when processes conducted with the same time-averaged power are compared. Dependence of deposition rate on target atom ionization energy and self-sputter yield have been reported for HIPIMS processes. Another property of the sputtered material that affects the deposition rate is the mass of the sputtered atoms, which dictates their transit time over characteristic lengths within the plasma chamber. The role of mass on the deposition rate of elemental metals with equivalent ionization energies but very different masses was investigated, and compared to the effects of ionization energy and self-sputter yield. Additionally, the effect of pulse parameters for targets of different masses on deposition rate was also examined. Two-dimensional optical images and plasma spectra were taken simultaneously during HIPIMS sputtering of several elemental targets, including hafnium and titanium with masses of 178 amu and 48 amu, respectively, and equivalent ionization energies of 6.8 eV. The characteristics of a density wave traveling perpendicular to the target surface at 5-8 km/s was observed in the images. Dependence on the mass of the target atoms was also observed. Spectroscopy techniques were used to observe temporal and spatial plasma chemistry. Time-resolved temperature measurements at the substrate surface were correlated to events occurring within the plasma, and compared to measurements of the time-averaged thermal load. The importance of flux composition and near surface temperature on growth phenomena are discussed in light of the results.

9:00am **H2-1-4 Characterization of HIPIMS Discharge for Next Generation Semiconductor Fabrication**, ***A.N. Cloud*** *(cloud1@illinois.edu), R.E. Flauta, M.J. Neumann, S.L. Rohde, D.N. Ruzic*, University of Illinois at Urbana-Champaign

Thin films produced using High Power Impulse Magnetron Sputtering (HIPIMS) have attracted considerable attention in the coating industry due to their excellent adhesion, superior density, and other favorable tribological properties. The extreme high power pulse densities provide a high concentration of metal ions and produce high-quality, homogeneous coatings. The high ionization fraction allows for fine control of the sputtered species during deposition, a feature well suited to the needs of future semiconductor fabrication techniques. HIPIMS may provide a highly scalable means of depositing diffusion barrier coatings and metallic features in the high aspect ratio interconnect trenches required for future chip designs. HIPIMS discharges from a planar magnetron were characterized and evaluated for potential as a chip processing tool. A gridded energy analyzer and quartz crystal microbalance were used to measure a higher ionization fraction in the HIPIMS discharge than with conventional magnetron sputtering under a variety of deposition conditions. The energy spectrum and flux of these ions at the substrate location were also measured. Plasma electron temperature and density as a function of pressure and power were mapped over a 3D region between the sputter target and substrate level by Langmuir probe analysis. A triple probe was used to study plasma conditions during the pulse. Deposition rates and film quality were evaluated. Characterization of the resultant films' structure, quality, and uniformity over the width of a 200 mm wafer and across surface features were performed.

9:20am **H2-1-5 Distance Dependent Plasma Composition and Ion Energy Distribution Functions in High Power Impulse Magnetron Sputtering of Ti and Cr**, ***A.P. Ehiasarian*** *(a.ehiasarian@shu.ac.uk),* Sheffield Hallam University, United Kingdom, *J. Andersson,* Ångstrom Laboratory, Sweden, *A. Anders*, Lawrence Berkeley National Laboratory

Research has shown that the target material in HIPIMS operation influences the total plasma density at the substrate, the metal ion–to–gas ion ratio, the speed of evolution of the discharge current and establishment of a self-sustained steady state. At the same time the ionization degree of metal vapor is constant with distance. However, the transport of particles from target to substrate is not well understood. We compare the diffusion of metal and gas ions to the substrate for Ti and Cr target materials with significant difference in sputter yield but similar mass. Ti and Cr HIPIMS plasmas operating at current density of ~0.5 Acm^{-2} have been characterised with a HIDEN plasma-sampling energy-resolved mass spectrometer at distances from 50 to 300 mm from the sputtering target. Measurements of the argon and metal ion content as well as the ion energy distribution functions showed that: (1) single and doubly charged ions were present for argon and target metal , (2) the majority of ions were singly charged argon for both metals and all distances investigated, (3) the Cr ion density was maintained to further distances from the target than Ti. Gas rarefaction was identified as the main mechanism promoting transport of the metal ion vapor with stronger effect achieved for materials with higher sputter yield. In the Cr case, metal ions were found to displace significant proportion of the gas ions, whereas this was less evident in the Ti case. Electron temperature cooling through metal ionizing collisions was responsible to eliminate production of highly charged Ar^{2+} ions.

9:40am **H2-1-6 Mass/Energy Analysis of the Plasma During MPP and Conventional DC Sputter Deposition of Cr and CrN Films**, ***W.D. Sproul*** *(bsproul@cox.net),* Reactive Sputtering, Inc., *J. Lin, J.J. Moore,* Colorado School of Mines, *Z.L. Wu,* Colorado School of Mines, and Dalian University of Technology, China, *X. Zhang,* Colorado School of Mines, *R. Chistyakov,* Zond, Inc., *B. Abraham,* Zpulser, LLC, *J.A. Rees*, Hiden Analytical, Ltd.

An energy/mass analyzer was used to characterize the plasmas during modulated pulse power (MPP) and conventional DC power sputter deposition of Cr and reactive sputter deposition of CrN films in a two-cathode closed field unbalanced magnetron sputtering system. The inlet to the energy/mass analyzer was located midway between the two cathodes at the position where the substrates normally are located, which is about 14 cm from the target surfaces. Experiments were run with the different types of power applied to either just one of the cathodes or to both of the cathodes when the cathodes were set up in the closed field configuration or when one of the cathodes was removed. The mass analysis detected Cr plus one, Ar plus one, and Cr plus two ions. The intensity of the Cr plus one ions when the MPP power is used is significantly higher compared to when DC power is used. As the peak power and the average power of the MPP pulse was increased, the intensity of these ions also increased in the closed field condition as it did when DC power was used. When one of the cathodes was removed and the cathode magnetic field was not closed, the intensity of the ions decreased significantly. The energy analysis revealed that the average energy for the Cr and Ar plus one ions is about 2 eV and that the energy distribution is very small. There is a slight high energy tail to the ion energy distribution, and there is almost a mono-energetic source of ions in the MPP process.

10:00am **H2-1-7 Stress in TiN Coatings Grown by HIPIMS**, ***R. Machunze*** *(r.machunze@tudelft.nl),* Delft University of Technology,, Netherlands, *A.P. Ehiasarian,* Sheffield Hallam University, United Kingdom, *G.C.A.M. Janssen*, Delft University of Technology, Netherlands

Titanium nitride coatings (TiN) are used amongst other applications as wear-protective coatings or as diffusion barriers in IC technology. In these applications the biaxial stress is a key factor determining the performance of the coating.

High power impulse magnetron sputtering (HIPIMS) leads to a high degree of ionization of 20-30% of the metal flux towards the growing film, which allows film growth at high ad-atom mobility and yet, a low incident ion energy of < 6 eV average. The ad-atom mobility is an important parameter influencing the film growth. The microstructure of HIPIMS grown TiN coatings is significantly denser compared to films grown by unbalanced magnetron sputtering (UBM). The crystallographic texture of HIPIMS coatings tends towards (001) in contrast to (111) for UBM coatings [J.Paulitsch et al., TSF (2008), doi:10.1016/j.tsf.2008.06.080]. In previous work we related the film stress and the stress gradient in UBM grown TiN coatings to their microstructure and texture. We proposed a model for increased compressive stress generation in crystals with a (001) orientation with respect to crystals with (111) orientation [R.Machunze and G.C.A.M.Janssen, submitted for publication to JAP].

In the present contribution we will compare film stress, microstructure and texture in UBM and HIPIMS grown TiN coatings. The influence of increased ad-atom mobility on the development of crystallographic texture and stress with film thickness will be discussed.

10:20am **H2-1-8 Cutting Performance Improvement of PVD TiAlN-Based Coatings, Produced by HPPMS Technology**, *K.-D. Bouzakis (bouzakis@eng.auth.gr), G. Skordaris, S. Gerardis, G. Katirtzoglou, S. Makrimallakis, M. Pappa,* Aristoteles University of Thessaloniki, Greece, ***R. Cremer****, H.-G. Fuss, W. Koelker, J. Dukwen*, CemeCon A.G., Germany

Recently High Power Pulsed Magnetron Sputtering technology (HPPMS) has been identified as a most powerful tool in the production of hard coatings. The enormous increase in metal atom ionization in HPPMS-generated plasmas gives to the PVD films' development of new possibilities. HPPMS technology affects significantly many physical properties of hard coatings like crystallographic, topographic and mechanical ones. In turn the properties result in higher cutting tools performance. In the investigations described in the paper, TiAlN and TiAlCrN PVD coatings have been produced using an industrial CemeCon CC800/9 HPPMS coating unit. Nanoindentations were conducted in all examined films and the corresponding results were evaluated with the aid of a FEM based algorithm, to determine the films' stress strain characteristics. Additionally, perpendicular as well as inclined impact tests were performed. By FEM-based simulations of these procedures, the fatigue properties and th e film adhesion were quantified. Moreover the wear behavior of coated cemented carbide inserts was investigated in milling of hardened steel. Appropriate FEM calculations of the developed temperature and stress fields in the cutting wedge region were conducted, to enable insights in the cutting process during milling. The results revealed the effect of the applied HPPMS technology during PVD films' deposition on the fatigue strength and film's adhesion as well as on the wear behaviour of coated tools.

Coatings for Use at High Temperature
Room: Royal Palm 1-3 - Session A3-1

Thermal Barrier Coatings
Moderator: A. Bolcavage, Rolls-Royce Corporation, R. Mevrel, ONERA, K. Murphy, Howmet Castings

1:30pm **A3-1-1 Toughening Mechanisms in 7YSZ Thermal Barrier Coatings**, *N.R. Philips,* ***E.M. Donohue*** *(erin_donohue@engineering.ucsb.edu),* University of California, Santa Barbara*, D. Kohl,* Universität Stuttgart, Germany / summer internship at University of California, Santa Barbara*, C.G. Levi, A.G. Evans*, University of California, Santa Barbara

The cyclic durability of thermal barrier coatings (TBCs) based on yttria-stabilized zirconia (YSZ) is greatest for compositions in the tetragonal (t') phase field, with the maximum occurring at 7 mole % (7YSZ). This trend in durability has been ascribed to a relationship between toughness and tetragonality, enabled by ferroelastic domain switching. While the existence of this mechanism has been demonstrated on dense, polycrystalline 7YSZ (toughness, Γ=45Jm^{-2}), it has yet to be demonstrated on coatings. This investigation probes the mode I crack propagation resistance of air plasma sprayed (APS) 7YSZ coatings incorporating dense, vertical-cracks (DVCs). High fidelity has been enabled by using a wedge-loaded double cantilever beam method that extends the cracks in a stable manner. The toughness Γ=250Jm^{-2} is substantially higher than that for the dense material, indicating unexpectedly large effects of the APS microstructure and of the DVCs. Various x-ray and electron probes, as well as nanoindentation, have been used to assess the associated mechanisms.

1:50pm **A3-1-2 Nondestructive Microstructural Characterization of Thermal Barrier Coatings by Laser Flash Technique**, ***F. Cernuschi*** *(federico.cernuschi@cesiricerca.it),* Cesi Ricerca, Italy*, P. Bison,* CNR-ITC, Italy*, A. Moscatelli*, Politecnico di Milano, Italy

In the case of materials with open porosity such as thermal barrier coatings, the environment the thermal diffusivity measurement is carried out (i.e. vacuum or gases) significantly affects the thermal diffusivity value. In fact, most of the more common gases have higher thermal diffusivity than that of YPSZ even if gas thermal diffusivity within the pores is lower than in large volumes ("bulk" value), when the pore dimension is comparable to the mean free path of gas molecules (Knudsen effect). The contribution of the measurement atmosphere to the thermal diffusivity strictly depends on the specific microstructural features of the porous sample under investigation such as porosity content, orientation and morphology. Thus, if thermal diffusivity measurements would be performed by varying gases a non destructive estimation of microstructural parameters would be possible. This approach can be used also to characterise sintering phenomena typically consisting in the healing of very fine crack-like pores which are difficult to be properly detected by conventional image analysis techniques. In this work an inversion procedure has been developed to obtain microstructural parameters describing the porosity morphology of porous thermal barrier coatings (TBC) from the thermal diffusivity measured in different environments by a Laser Flash technique. A simplified approach in the inversion procedure has been proposed, and the reliability has been checked by simulating different microstructures within the TBC. The inversion procedure has been also applied to experimental thermal diffusivity values of a TBC which were obtained by filling pores with He, N2, Ar and in vacuo.

This work has been financed by the Research Fund for the Italian Electrical System under the Contract Agreement between CESI RICERCA and the Ministry of Economic Development - General Directorate for Energy and Mining Resources stipulated on June 21, 2007 in compliance with the Decree n.73 of June 18, 2007.

2:10pm **A3-1-3 Recent Results on Advanced Thermal Barrier Coatings**, *D. Stöver,* ***R. Vaßen*** *(vassen@fz-juelich.de), O. Jarligo, H. Kassner, Y. Zhang, D. Mack, G. Mauer*, Institute of Energy Research (IEF-1), Germany **INVITED**

The performance of thermal barrier coating (TBC) systems is largely influenced by the microstructure of the ceramic topcoat. This has been observed for standard TBC systems made of yttria partially stabilized zirconia (YSZ), and also for those made of new TBC materials. One of the major processing routes for the manufacture of TBCs is the atmospheric plasma spraying (APS). The microstructure of atmospheric plasma-sprayed coatings with conventionally micro-cracked, highly porous, segmented and other advanced microstructures and their processing conditions will be outlined. This overview will also include suspension plasma spraying in which a liquid feedstock instead of particulates is used. A comparison of the properties of these different kinds of coatings will be given.

Although the standard TBC material YSZ shows unique properties with respect to a TBC application it has a limited temperature capability of about 1200°C in long-term applications. Therefore potential new TBC materials are being evaluated as pyrochlores, perovskites, and aluminates. The typical relevant physical properties of the bulk materials will be shown. In addition, specific processing issues for the different materials will be discussed. Finally, the performance of these materials as TBC systems will be presented.

2:50pm **A3-1-5 The Relation Between Morphology, Phase and Thermal Conductivity Changes in the Thermally Loaded EB-PVD TBCs**, ***R. Ochrombel*** *(rene.ochrombel@dlr.de),* German Aerospace Center, Germany*, V. Ryukhtin,* Technical University of Berlin, Germany*, B. Saruhan*, German Aerospace Center, Germany

Increase in combustion temperature for improved turbine efficiency causes greater thermal loading at the thermal barrier coatings (TBCs) of the stationary and aircraft turbine blades. At these elevated temperatures (> 1300°C), the state of the art TBC-material, partially yttria stabilized zirconia (PYSZ), suffers phase instability and heavy sintering-induced morphological changes resulting in an increase at their thermal conductivity. Previously, it was demonstrated that fully stabilized zirconia (FYSZ) containing 14 wt. % Ytrria yields intrinsically lower thermal conductivity. It is also possible to optimize the TBC properties by tailoring its microstructure, since an important relationship between thermal conductivity and processing conditions of EB-PVD TBCs exists. To optimize the performence of TBCs, a parameter study is necessary. In this work, the results obtained on EB-PVD deposited PYSZ and FYSZ samples varying the process parameters and applying X-Ray powder d iffraction (XRD), Scanning Electron Microscopy (SEM), Laser Flash Analysis (LFA) and in-situ high temperature Small Angle Neutron Scattering (SANS) will be presented and compared with each other. The influence of the resulting texture, the resulting phase changes/phase mixture, morphology, especially thermal induced changes in pore shape and size will be correlated with the thermal conductivity and discussed in terms of process parameter and phase constituency influences.

3:10pm **A3-1-6 The Behavior of High-Purity, Low-Density Air Plasma Sprayed Thermal Barrier Coatings**, *G.H. Meier (ghmeier@pitt.edu),* ***M.A. Helminiak****, N.M. Yanar, F.S. Pettit,* University of Pittsburgh*, T.A. Taylor*, Praxair Surface Technologies

This paper describes research on the behavior of high-purity, low-density (85%) air plasma sprayed (APS) thermal barrier coatings (TBC) with NiCoCrAlY bond coats deposited by argon-shrouded plasma spraying. The microstructure of the APS topcoats was controlled to maximize the coating thicknesses that can be applied without spallation and to minimize the thermal conductivity of the TBC. The specimens are being evaluated using cyclic oxidation and thermal shock tests, and important properties of the TBCs, such as resistance to sintering and phase transformation, thermal conductivity, and fracture toughness are being determined. The high purity resulted in top coats which are highly resistant to sintering and transformation from the metastable tetragonal phase to the equilibrium mixture of monoclinic and cubic phases. The porous topcoat microstructure also resulted in significant durability during thermal cycling. A 750 μm thick APS coating was found to have a cyclic life that was at least as long as that of a standard 100 μm thick electron beam physical vapor deposition (EBPVD) coating, as measured in a furnace cycle test. The actual failure mechanisms of the APS coatings were found to depend on topcoat thickness and the nature of the thermal exposure.

3:30pm **A3-1-7 Thermo-Mechanical Properties and Gradient Testing of Thermal Barrier Coatings Subject to Spallation Due to CMAS (Calcium-Magnesium-Alumino-Silicate) Penetration**, ***S. Faulhaber*** *(safaha@engr.ucsb.edu), A.G. Evans*, University of California Santa Barbara

Spallation in Thermal Barrier Systems can occur as a consequence of changes in the mechanical and thermal properties of the coating due to infiltration with molten deposits. Investigation of delaminations and microstructural changes by microscopy and related techniques has allowed insights into the thermal conditions experienced by the material leading to a basic understanding of the delamination mechanism. To refine delamination maps that help predict conditions under which spallation occurs the mechanical properties, Young's modulus and fracture toughness, and the thermal properties, diffusivity and thermal expansion coefficient, have been

measured. Thermal gradient experiments with well-defined thermal conditions were conducted to confirm the thermomechanics of spallation.

3:50pm **A3-1-8 Effect of Low Level CMAS Attack on EB PVD TBCs**, ***R.G. Wellman*** *(R.Wellman@cranfield.ac.uk), G. Whitman, J.R. Nicholls*, Cranfield University, United Kingdom

When debris deposits onto thermal barrier coatings (TBCs) in gas turbine engines at high temperatures, it can melt to form Calcium-Magnesium-Aluminosilicates (CMAS). This molten CMAS attacks the TBC by infiltrating the columnar structure, which has a detrimental effect on the TBCs morphology and microstructure. This paper discusses the early effects of CMAS attack at low concentrations to understand the evolution of the degradation. By depositing small amounts of CMAS onto TBCs, a minimum safe level was established under which CMAS degradation does not occur. Both pre-reacted CMAS and CMAS as constituent powder were used in this study, since it is unknown as to whether the CMAS forms before or after deposition onto the turbine blade. It was found that at least 4.8mg/cm^2 of CMAS powder was required to form a uniform level of attack after 4hrs at 1300^{o}C. This minimum level reduced significantly by increasing the exposure time to 12 hours, but variations in temperature was found to have much less of an effect. Increasing the temperature did not reduce the level required to cause the transformation of the TBC. Preliminary studies into mechanical properties of CMAS degraded samples showed that 4.8mg/cm^2 for 4 hours at 1300^{o}C was sufficient to cause an increase in room temperature erosion rate by factor of five. This evidence suggests that even very low levels of CMAS infiltration is sufficient to cause severe degradation of the TBC, leading to significantly reduced service lives of TBC coated components. This paper looks at the differences between using pre-reacted CMAS as opposed to the constituent powders on the degradation of EB PVD TBCs at very low levels as well as investigating how the low levels of CMAS infiltration affect the erosion performance and erosion mechanism of the EB PVD TBCs.

4:10pm **A3-1-9 The Role of Crystallization in Arresting CMAS Infiltration into TBCs**, ***E.M. Vogel*** *(evogel@engineering.ucsb.edu), S Krämer, C.G. Levi*, University of California, Santa Barbara

Thermal barrier coatings (TBCs) in engines subject to siliceous debris ingestion with the intake air experience infiltration by calcium magnesium alumino-silicate (CMAS) melts with detrimental effects on the durability of the coating. Field samples reveal that CMAS penetration in 7YSZ TBCs is arrested by crystallization at temperatures within the coating well below the melting isotherm. When CMAS interacts with zirconate TBCs, penetration is arrested by crystallization much closer to the surface, even in the absence of a thermal gradient. The implication is that the dynamic crystallization behavior is influenced not only by the thermal history, but also by the chemical interaction with the coating. The present investigation aims at understanding these phenomena and underlying mechanisms by a combination of differential scanning calorimetry (DSC) and direct visualization of the infiltration behavior in a tube furnace set up, followed by extensive microstructural analysis. DSC of a model crystalline CMAS with average composition 35CaO-10MgO-7Al_2O_3-48SiO_2 revealed a sharp melting endotherm at T_m ~1240°C, with subsequent crystallization on cooling marked by a broad exotherm peaking at ~1150°C. Amorphous CMAS of the same composition showed also crystallization over a broad exotherm with a maximum at ~1100°C. Visualization experiments reveal remarkably different behaviors of CMAS on the TBC depending on whether the "deposit" is crystalline or amorphous, the temperature ramp up rate, the hold temperature and the nature of the TBC. These issues and the implications to the potential mitigation of CMAS penetration are discussed in the context of recent research.

4:30pm **A3-1-10 Electrophoretically Deposited Alumina as Protective Overlay for Thermal Barrier Coatings Against CMAS Degradation**, ***P. Mohan*** *(pmohan@mail.ucf.edu), T. Patterson, Y.H. Sohn*, University of Central Florida

Thermal barrier coatings (TBCs) can be highly susceptible to degradation due to air ingested CMAS (calcium-magnesium alumino silicate) sand deposits during operation in a dust-laden environment. At high temperature, CMAS deposits melt and degrade the TBC system via repeated freeze-thaw action and to a certain extent, direct chemical reaction with TBC constituents (e.g., destabilization of yttria stabilized zirconia, YSZ coatings). In order to protect TBCs from CMAS attack, a dense crack-free overlay of alumina was fabricated by electrophoretic deposition (EPD) on YSZ coatings. For EPD, a colloidal suspension of α-Al_2O_3 powders in acetone/ethanol mixture was used. At an applied DC voltage of 25V, positively charged alumina powders in the colloidal suspension were drifted and deposited on YSZ for up to 10 minutes. The deposited powder compact by EPD was carefully dried and densified by sintering at various temperatures up to 1400°C for 5 h. EPD process can deposit dense , crack-free alumina overlay of uniform thickness up to 20 μm. Effect of alumina as protective overlay against CMAS melt ingression was examined by exposing the modified YSZ coatings to a laboratory-synthesized CMAS deposit at temperature up to 1350°C. The tested specimens were characterized by x-ray diffraction (XRD) and scanning electron microscopy (SEM). Preliminary results show that CMAS melt ingression into the YSZ was completely suppressed with dense EPD alumina overlay. Attributed mechanisms include complete crystallization of CMAS glass due to a shift in glass composition with an increase in Al content resulting in compounds such as anorthite ($CaAl_2Si_2O_8$), which can further act as an overlay constituent to suppress CMAS ingression. Improved performance of modified YSZ coatings against CMAS attack will be presented in detail along with processing strategies adoptable for coatings industry.

4:50pm **A3-1-11 Dependence of Microstructure and Mechanical Properties with Starting Powder Morphology in Zirconia-Based Thermal Barrier Coatings**, ***Y.G. Jung*** *(jungyg@changwon.ac.kr), S.I. Jung, J.Y. Kwon, Y.S. Sim, U. Paik,* Changwon National University, Korea, *K.S. Lee*, Kookmin University, Korea

The effects of powder morphology in air plasma-sprayed coatings on microstructure and mechanical properties of thermal barrier coatings (TBCs) were investigated under thermal exposure. Two kinds of powders were prepared with different processing parameters, especially solvent, in spray drying process, showing a deformed hollow type and a filled spherical type, and then heat-treated at 1,250^{o}C for 1 h. The coating thickness prepared by the hollow type is thicker than that by the spherical type. All peaks in phase analysis are tetragonal and cubic phases without any monoclinic phase in the developed powders. The relatively porous microstructure could be obtained in the hollow type, while the thickness of thermally grown oxide (TGO) layer in the hollow type is thicker than the spherical type with a saturated point at 100 h thermal exposure. The hardness values on sectional plane of TBCs are slightly higher than those on surface plane, whereas the spherical type show s higher values on both planes in all temperatures tested. However, the toughness values on surface plane are definitely higher than those on sectional plane, without an effect of power morphology. The hardness values obtained by nanoindentation in each component—substrate, bond coat, and top coat are not greatly affected by thermal exposure with a large scatter in the bond and top coats, whereas the values of elastic modulus in the bond coat are dominantly affected by thermal exposure.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B6-2

Hard and Multifunctional Nano-Structured Coatings

Moderator: M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Laboratories, U.S. Army ARDEC, R. Sanjines, EPFL

1:30pm **B6-2-1 Metal Carbide/Amorphous C-Based Nanocomposite Coatings for Tribological Applications**, ***J.C. Sanchez-Lopez*** *(jcslopez@icmse.csic.es), D. Martinez-Martinez, M.D. Abad, A. Fernandez*, Instituto de Ciencia de Materiales de Sevilla, Spain **INVITED**

This paper tries to assess the factors governing the tribological behaviour of different nanostructured or nanocomposites films composed by metallic carbides (MeC) mixed with amorphous carbon (a-C). Different series of MeC/a-C coatings (with Me: Ti(B) and W) were prepared by magnetron sputtering technique varying the power applied to the graphite target in order to tailor the carbon content into the films. A deep investigation of the chemical and structural features at the nano-scale is carried out for each family of coatings by many different spectroscopic, microscopic and diffraction techniques in order to establish correlations with the tribological properties measured by a pin-on-disk tribometer in ambient air. The analysis of the counterfaces by Raman confocal microscopy after the friction tests is used to follow the chemical phenomena occurring at the contact area responsible of the observed friction behaviour. The importance of determining the nanocrystalline/amorphous ratio is highlighted as a key-parameter to control the tribological properties. A comparative analysis of the mechanical and tribological performance of the three systems (TiC/a-C, TiBC/a-C, WC/a-C,) is revised and conclusions are obtained concerning the friction and wear mechanism involved.

2:10pm **B6-2-3 XPS Analysis of Binary and Ternary TiC-Based Alloy and Nanocomposite Coatings**, ***E. Lewin*** *(erik.lewin@mkem.uu.se),* Uppsala University, Sweden*, M. Gorgoi,* BESSY GmbH, Germany*, U. Jansson*, Uppsala University, Sweden

Through thin film techniques a continuously growing amount of meta-stable phases and microstrucures are synthesised and proposed for use in a diverse field of applications. A commonly used technique to analyse these coatings with regards to chemical composition and bonding is X-ray photoelectron spectroscopy (XPS) coupled with sputter etching using an ion gun to attain information below the outermost surface. For carbide based systems XPS is presently often used to determine relative amount of carbide and free carbon phases, well as the sp^2/sp^3 ratio of the free carbon phase. This analysis is often performed after sputter-etching. Although it is well known that sputter etching can cause damage to the analysed sample and interfere with the results, it is often not thoroughly investigated as one moves from well known systems such as TiC to alloyed or nanocomposite systems such as (Ti,Me)C and nc-TiC/a-C. We here present an analysis of binary and ternary TiC-based alloy an d nanocomposite coatings in the Ti-Ni-C and Ti-Cu-C systems. We have used angle-resolved XPS with different sputter energies (acceleration voltages) and compare these results with results attained by use of high kinetic energy (HIKE) XPS, which has a larger sampling depth, of undamaged samples. We observe substantial sputter damage (strongly dependent on sputter energies), specially for the case of alloyed carbides such as (Ti,Ni)C. This clearly limits the use of XPS together with sputter-etching as a tool to understand the structure of meta-stable coatings. In the light of these results we discuss when and how one can use XPS as a reliable analytical tool.

2:30pm **B6-2-4 Fabrication and Tribological Properties of Composite Coatings Produced by Lithographic and Microbeading Methods**, ***J.E. Krzanowski*** *(jamesk@cisunix.unh.edu)*, University of New Hampshire **INVITED**

Nanoscale multiphase and composite coatings have been the subject of intensive research and are being proposed as the next generation of tribological coatings. This research has been driven by the need for coatings to function in extreme, as well as multiple, environments. However, fabrication of coatings with distinct phases and appropriate microstructure remains a challenge. One approach is to pattern a coating by external intervention. These methods include masking, laser drilling of holes, and lithographic methods. Our recent work has focused on lithographic methods, as well as a new masking approach which we refer to as microbeading. In the latter method, microscopic ceramic or glass beads are used to decorate the surface before deposition, and act as placeholders. After deposition, these beads are removed leaving behind a random arrangement of holes. The surface is then coated with a solid lubricant, and during wear the holes act as microreservoirs for lubricant s torage. Evaluation of microreservoir-containing coatings fabricated by the microbeading method was carried out by depositing a TiN film, and then using graphite as the solid lubricant. Pin-on-disk tests using an alumina counterface showed that substantial reductions in friction coefficients were obtained for the larger bead sizes (5-10 microns). Examination of the wear tracks using optical microscopy showed that the graphite lubricant was successfully trapped by the microreservoirs during the pin-on-disk test running. The microbeading coating method was also implemented on cutting tools for machining where indium was used as the solid lubricant. Turning tests were conducted by high-speed machining of hardened 4340 steel. TiN-In coated inserts were tested by measuring flank wear in lubricated (cutting fluids used) machining, and showed up to 4 times longer wear life than a TiN coating without indium. TiN, TiN with an In topcoat, and TiN with In and 5 micron microreservoirs we r e also tested in dry machining, and the coating with the 5 micron microreservoirs exhibited the least wear for most of the test period. XPS analysis confirmed the presence of the indium near the tool tip, where the chip flows on the rake face. In addition, samples with microreservoirs showed greatly reduced crater wear on the rake surface. These results demonstrate that the use of indium with microreservoir-patterned coatings can be beneficial in dry machining of steel.

3:10pm **B6-2-6 Microstructures and Corrosion Resistance of Pulsed DC Reactive Magnetron Sputtered nanocomposite Zr-Si-N Thin Films**, ***Y.-B. Lin****, J.-W. Lee (cwlee@mail.tnu.edu.tw),* Tungnan University, Taiwan*, L.-C. Chang*, Mingchi University of Technology, Taiwan

The nanocomposite Zr-Si-N thin films with various Si contents have been deposited by a bipolar asymmetric pulsed DC reactive magnetron sputtering system. The Zr-Si- N thin films with Si contents ranging from 1.5 at.% to 8 at.% were achieved. The structures of Zr-Si-N films were characterized by XRD. The surface and cross sectional morphologies of thin films were examined by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The surface roughness of thin films was explored by atomic force microscopy (AFM). The nanoindentation, nanoscratch and nanowear tests were adopted to evaluate the nanomechanical properties of Zr-Si-N coatings. The electrochemical test in 3.5 wt.% NaCl aqueous solution was conducted to evaluate the corrosion performance of each coating. The amorphous structure was observed when the silicon content reached 7 at.% in Zr-Si-N thin films. In general, the corrosion resistance of the coating increased with increasing Si content. A high hardness and good corrosion resistance were found in the Zr-Si-N thin film with around 3 at.% Si in this work.

3:30pm **B6-2-7 Hard and Decorative Coatings Based on Al-N + Au Nanocomposite Structures**, ***A. Cavaleiro*** *(albano.cavaleiro@dem.uc.pt), C. Louro, N. Figueiredo*, University of Coimbra, Portugal

One of the most interesting aspects of the metals clusters when placed in a dielectric matrix is that their optical properties depend strongly upon the particle size and shape. Metals with free electrons (essentially Au, Ag and Cu) possess plasmon resonances in the visible spectrum, which give rise to intense colour in this situation. The main objective of this research is to produce new decorative coatings by dispersing nanocrystals of gold in a dielectric and transparent matrix (e.g. Al-O). Simultaneously, it is expected that the coatings have suitable mechanical properties for being wear resistant. Al-N+Au coatings were deposited by sputtering from an aluminium target embedded with thin gold plates, in a reactive environment containing nitrogen. The N2 flow was high enough for working in compound mode producing transparent dielectric films. The Au content was varied from 0 up to 15 at.%. Subsequently, the films were treated to promote the segregation of metallic nano crystals of Au in the dielectric matrix. The precipitation process was followed by high-temperature X-ray diffraction. For selected temperatures, samples were annealed in a furnace in order to produce samples to be optically and mechanically characterized. In this work, only the results concerned the optical properties, evaluated by colorimetry and UV-Vis spectroscopy, and the mechanical properties, hardness and Young´s modulus, will be presented as a function of the Au content, before and after annealing at increasing temperatures up to 1000ºC.

3:50pm **B6-2-8 Microstructure – Property Relationships in Nitride-Based Coatings on Steel Substrates Prepared by Pulsed Laser Deposition**, ***A. Jahja*** *(z8642664@student.unsw.edu.au), P. Munroe*, University of New South Wales, Australia

A range of sub-micron thick TiN coatings were deposited on a H13 hot worked tool steel substrate via pulsed laser deposition as a function of processing condition. The coatings were subject to detailed microstructural characterization, including cross-sectional TEM studies. Coatings prepared at high substrate temperatures (450°C) and reactive gas conditions exhibited nanoscale grain sizes, whilst slightly coarser structures were prepared in inert environments. Mechanical behaviour was assessed through nanoindentation using a spherical indenter. The coatings exhibited high hardness values and significant resistance to cracking, even at high loads. Examination of the indented layers revealed intercolumnar cracks within the TiN coatings, together with shear steps at the coating-substrate interface, whilst inclined cracks were observed at the periphery of the indentations.

4:10pm **B6-2-9 Chromium-Aluminum Oxide Coatings Deposited by Reactive Magnetron Sputtering**, ***P. Eklund*** *(perek@ifm.liu.se),* Linköping University, Sweden*, K. Pedersen,* University of Aarhus, Denmark*, K.P. Almtoft, L.P. Nielsen,* Danish Technological Institute, Denmark*, M. Sridharan, M. Sillassen, J. Bottiger*, University of Aarhus, Denmark

Chromium oxide and aluminum oxide thin films were deposited by reactive inductively coupled plasma magnetron sputtering at substrate temperatures from room temperature to 700°C. X-ray diffraction and electron microscopy showed that the as-deposited chromium oxide coatings crystallized in a corundum structure, while aluminum oxide thin films deposited at low ion flux during growth were amorphous in the substrate-temperature range 200 to 450°C. A higher ion flux and/or temperature resulted in predominantly crystalline γ-alumina[1]. For Cr_2O_3, the hardness was 29 GPa, while the hardness values of the as-deposited alumina films varied from 8–20 GPa, with the highest values corresponding to larger crystalline volume fractions. The growth of α-alumina at 450°C could be promoted by depositing onto chromia templates, a result consistent with previous findings[2]. In contrast, we observed a strong texture effect with extended growth of α-Al_2O_3 at a substrate temperature of 450°C using a predominantly <104>-textured Cr_2O_3 template layer, while only limited α-Al_2O_3 nucleation was seen on a <001>-textured Cr_2O_3 template[3]. The texture of the chromia templates could be controlled from a <001> texture due to competitive growth to an ion-bombardment-induced <104> texture, to a mixed <110>-dominated texture at high ion bombardment. Codeposition of chromia and alumina onto <104>-textured templates yielded corundum-structured films and a slight hardness increase as compared to pure chromia, possibly due to phase separation of Cr_2O_3 and Al_2O_3. Further, alumina films were deposited in a large-scale industrial coating facility employing pulsed dc sputtering and a range of pulsed dc bias. The morphology and density of the alumina

coatings could be controlled by bias modulation and ramping, with columnar growth obtained for negative bias of 30 to 65 V. A dense, featureless, insulating alumina was obtained for bias above 70 V.

[1] M. Sridharan et al Surf. Coat. Technol. 202 (2007) 920.

[2] e. g. J. M. Andersson et al J. Vac. Sci. Technol A 22 (2004) 117, and references therein.

[3] P. Eklund et al Thin Solid Films 516 (2008) 7447.

Optical Thin Films
Room: Royal Palm 4-6 - Session C1

Recent Advances in Optical Thin Films
Moderator: J. Bellum, Sandia National Laboratory, M. Trubetskov, Moscow State University

1:30pm **C1-1 Unusual Optical Thin Film Solutions Based on Advances in Materials and Deposition Processes**, ***J.A. Dobrowolski*** *(dobrowolski@magma.ca)*, NRC, Canada **INVITED**

Optical thin film systems must meet a specified optical performance, as well as satisfy a number of auxiliary specifications that are equally important. These may include stability of the optical performance over time and at high and low temperatures, mechanical robustness, chemical inertness, resistance to damage by radiation and by high power lasers. These auxiliary requirements are often much harder to meet than the specified optical performance. The spectral and angular characteristics of thin film systems depend on the number of layers and their thicknesses, on the overall thickness of the layer system and on the optical constants of the available coating and substrate materials. Examples will be given in this paper showing how the optical performance of designs has been improved in the past through the use of unusual properties of coating materials, or through the use of deposition techniques that resulted in films with effective optical constants that lie outside those that nature has provided. However, a layer system with the calculated optimum optical performance may not meet the auxiliary requirements. Also shown will be examples in which both the thin film designs and the deposition process had to be adjusted in order to meet both the auxiliary requirements and the required optical performance. The layer systems that will be used for illustration purposes will include antireflection coatings, reflectors, cut-off filters, narrow band pass filters and polarizing beam splitters and they will include examples taken from the extreme ultraviolet to the far infrared spectral regions.

2:10pm **C1-3 Thermal Stability of SiO_xN_y Thin Films with Tailored Refraction Index**, *V. Godinho (godinho@icmse.csic.es),* Instituto de Ciencia de Materiales de Sevilla-CSIC/US, Spain, *C. Fernandez-Ramos,* Institute for Prospective and Technological Studies-JRC European Commission; Instituto de Ciencia de Materiales de Sevilla-CSIC/US, *M.C. Jimenez de Haro,* Instituto de Ciencia de Materiales de Sevilla-CSIC/US, *M.P. Delplancke-Ogletree,* Université Libre de Bruxelles, Belgium, ***A. Fernandez***, Instituto de Ciencia de Materiales de Sevilla, Spain

Silicon oxynitride is transparent in the visible range and the refraction index can be varied from that of pure silicon dioxide (1.47) to the one of pure silicon nitride (2.3). A continuous change in its composition allows to obtain films with different optical, electrical and also mechanical properties[1]. Navid et al[2] studied theoretically the possibility of tuning the optical properties of thin films of similar composition introducing nanopores with different shape, size and spatial distribution. The introduction of pores decreases the refraction index of the coatings. In a previous work[3] we showed that the formation of close porosity on SiO_xN_y thin films, in the form of nano-voids (3-15 nm in size), allowed tuning the dielectric constant of silicon oxynitride films in a wide range of values without significant change in their composition. In this work we show the tuning of refraction index and focus on the thermal stability (chemic al and microstructural) of these coatings under different atmospheres considering their possible applications. Electron microscopy (SEM-FEG), elipsometry, X-ray photoelectron spectroscopy (XPS), electron energy loss spectroscopy (EELS), X-ray diffraction (XRD), as well as nanoindentation results will be discussed demonstrating the control of the optical properties together with a good thermal stability of the amorphous thin films for optical applications up to 900°C.

[1]M. I. Alayo, D. Criado, L. C. D. Gonçalves, I. Pereyra, Journal of Non-Crystalline Solids 338-340 (2004) 76-80.

[2]A. Navid, L. Pilon, Thin Solid Films 516 (2008) 4159-4167.

[3]V. Godinho, V.N. Denisov, B.N. Mavrin, N.N. Novikova, E.A. Vinogradov, V.A. Yakovlev, C. Fernández-Ramos, M.C. Jiménez de Haro, A. Fernández (submitted).

2:30pm **C1-4 Improved Contrast and Reflectivity of Near Normal Incidence Reflective Mjltilayer Optics for FLASH and Next Generation Soft X-ray Lithography**, ***T. Tsarfati*** *(t.tsarfati@rijnhuizen.nl), E. Zoethout, R.W.E. van de Kruijs, F. Bijkerk*, FOM Institute for Plasma Physics Rijnhuizen, Netherlands

For applications in e.g. FLASH, next generation lithography, soft x-ray spectroscopy, fluorescence analysis and imaging, we performed an experimental feasibility study of multilayer reflective optics for use in the 6.7 nm wavelength range. A B_4C/La combination offers a reflection maximum of around 40% and FWHM of ~0.06 nm. It is observed to yield chemical reactivity and LaB_6 interlayer formation at the interfaces, severely limiting optical contrast and reflection. We have now successfully passivated the individual interfaces. Simultaneously, we managed to increase optical contrast [1,2]. Extrapolations of experimental results with 50 period multilayers suggest more than 1/3 higher reflection maximum and FWHM than currently reported record values for 200 period multilayers. Calculations show that even further improvement of FWHM can be achieved by replacing B_4C with B and La with Th or U[3].

[1] T. Tsarfati, E. Zoethout, R. W. E. van de Kruijs, F. Bijkerk, submitted to PRL

[2] T. Tsarfati, E. Zoethout, E. Louis, F. Bijkerk, patent

[3] T. Tsarfati, E. Zoethout, R. W. E. van de Kruijs, F. Bijkerk, submitted to APL

2:50pm **C1-5 Multi-Layer Dielectric Coatings and Processing for Production of Optical Gratings on Large Substrates**, ***D.J. Smith*** *(dsmith@plymouthgrating.com)*, Plymouth Grating Laboratory, Inc. **INVITED**

Multi-Layer Dielectric Coatings and Processing for Production of Optical Gratings on Large Substrates Douglas J. Smith Plymouth Grating Laboratory, Inc. Plymouth, MA, 02360 Abstract Laser Pulse Compression methods first use a diffraction grating to stretch a short pulse, then amplify the lengthened pulse in a conventional amplifier, and finally re-compress the pulse using a staged set of large diffraction gratings. Because the gratings are often used at high incidence angles, the required gratings are large rectangles that may be tiled together. Typical grating sizes range to 91cm x 42cm. The gratings may use a gold overcoat to obtain very high fluences or a multi-layer dielectric (MLD) mirror will be coated underneath the grating structure. Since the electric field of the final compressed laser pulse will interact strongly with air at atmospheric pressure, the compression gratings must operate in a high vacuum environment. The gratings are often made on fused silica substrates to provide better thermal stability. The MLD gratings are subjected to an aggressive acid cleaning procedure that removes traces of organic contamination to increase the laser damage threshold.

Special ion-assisted deposition coating methods were developed to meet the requirements of coating uniformity, laser damage, stress deformation, and coating failure due to tensile fracture or adhesive failure during fabrication and use. The coatings use hafnia and silica in a multilayer optimized for the final grating wavelength of 1054 nm. Additionally, the coating design must not allow excessive standing waves to develop in the photoresist at the exposure wavelength of 351 nm. Other aspects of exposure, such as scattered light or reflected light from rear surfaces, and efficiency of coupling to the photoresist layer must be considered as well. An organic absorbing layer is used with the MLD and photoresist to meet these requirements to provide the best resolution for the grating structure. An RF ion source is used to make the coatings and can be adjusted to meet the stringent mechanical requirements of the coatings. An RF source is also used to etch the grating structure into the top fused silica layer. Results from some of these large gratings, and some of the pitfalls in manufacturing will be presented.

3:30pm **C1-7 Visible Light Photocatalysis of N-Doped TiO_2 Films Prepared by Reactive Sputtering Using Air/Ar Mixtures**, ***M.-H. Chan***, National Chung Hsing University, Taiwan, *F.-H. Lu (fhlu@dragon.nchu.edu.tw)*, National Cheng Kung University, Taiwan

Nitrogen-doped titanium dioxide thin films were prepared by reactive sputtering using air/Ar mixtures. The modification of TiO_2 for photocatalysis is desirable to effectively utilize the visible light region of the solar spectrum. Using air as a reactive gas allows the process to perform at a high base pressure (low vacuum) and could then drastically reduce the processing time. X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), and UV-visible spectroscopy were used to characterize obtained thin films. XRD patterns show that the films possessed mixed anatase and rutile phases. The constituent of the films was determined by XPS. N-doped TiO_2 films exhibited absorption in the visible light region. The optical band gap of the films calculated from Tauc plots was 3.0 eV, which is lower than that of pure TiO_2. A red shift was clearly observed by introducing the N-dopant.

3:50pm **C1-8 Low Temperature HIPIMS Deposition of AZO Coatings on Polymeric Web**, ***P.J. Kelly*** *(peter.kelly@mmu.ac.uk), P. Barker, G. West,* Manchester Metropolitan University, United Kingdom*, J.W. Bradley*, University of Liverpool, Untied Kingdom

Functional films, particularly transparent conductive oxides (TCOs), are widely used in devices such as flat panel displays and solar cells. The coatings must have specific optical and electronic properties whilst maintaining good crystalinity, structure and low defect density. These properties are often achieved through post deposition annealing. It would be significant to industry if TCO coatings, such as aluminium-doped zinc oxide (AZO) could be readily deposited onto flexible polymeric web, rather than onto rigid glass substrates. This would provide reductions in weight and cost of the finished products, whilst also increasing throughput and efficiency by utilising roll to roll web coating technology. The thermally sensitive nature of the substrates, though, currently limits the choice of deposition process. However, HiPIMS (high power impulse magnetron sputtering) may provide a solution to this problem. Despite the very high peak powers (up to MWs) achievable in th is mode, the thermal energy flux to the substrate has been shown to be significantly reduced, compared to other magnetron sputtering processes. Furthermore, the process also produces high levels of ionisation of the target material, which offers the potential to produce high quality TCO coatings on polymeric web without the need for post deposition annealing processing. This paper, therefore, discusses the deposition of AZO coatings onto PET web through the use of HiPIMS. The coatings have been characterised in terms of their structural, optical and electrical properties, and these characteristics have been related to specific features in the HiPIMS process.

4:10pm **C1-9 Fabrication of ZnO Thin Films by Atomic Layer Deposition Using Interrupted Flow-Rate Method**, *C.-S. Ku,* National Synchrotron Radiation Research Center, Taiwan*, J.-M. Huang,* National Hsinchu University of Education, Taiwan*,* ***C.-M. Lin****,* National Hsinchu University of Education,, Taiwan*, H.-Y. Lee (hylee@nsrrc.org.tw)*, National Synchrotron Radiation Research Center, Taiwan

High quality of crystalline ZnO films has been successfully grown on c-plane sapphire substrate by atomic layer deposition (ALD) technique at extremely low temperature. In this work, we obtained the ZnO thin films by ALD technique using interrupted flow-rate method at extremely low temperature with diethylzinc and D. I. water as precursors. The ALD growth window was found to be 50-90ºC by using interrupted flow-rate method (IFM). It's effectual to decrease the growth temperature about 100ºC for ALD with general continuous flow rate method. X-ray reflectivity and high-resolution x-ray diffraction measurements were employed to characterize the microstructure of these films. The results show the low growth temperature result higher crystalline quality and to agree with thermodynamically blocked self-compensation processes.

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E3-1

Tribology of Nanostructured and Amorphous Films
Moderator: J. Fontaine, Ecole Centrale de Lyon, T.W. Scharf, The University of North Texas

1:30pm **E3-1-1 Friction Behavior of Nanocrystalline Diamond Coatings at Variable Sliding Speeds**, ***N. Theodore*** *(nimel.theodore@nrl.navy.mil),* Naval Research Laboratory / North Carolina State University*, K. Wahl*, Naval Research Laboratory

Tribology studies were performed on nanocrystalline diamond coatings to investigate changes in friction with sliding speed. Microstructure, surface morphology, and composition of the coatings were characterized using X-ray diffraction (XRD), atomic force microscopy (AFM), interferometry, and infrared and Raman spectroscopy. The nanocrystalline diamond coatings possessed crystallite sizes ranging from to 8 to 60 nm. These nanocrystalline diamond coatings produced similar visible wavelength Raman absorption bands with a peak at 1332 cm^{-1} typical of crystalline diamond bonding, strong peaks at 1340 cm^{-1} and 1580 cm^{-1} characteristic of the D and G peaks in sp^2 hybridized carbon, and peaks at 1135 cm^{-1} and 1470 cm^{-1}, which are commonly attributed to polyacetylene bonding. Reciprocating sliding tests, using sapphire and diamond counterfaces were performed in controlled air and nitrogen environments with variable humidity. The lo ad and track length were fixed at 3 N and 5 mm, respectively, and the sliding speed was varied between 100 μm/s to 2 mm/s. Sliding with either diamond or sapphire counterfaces resulted in low friction values, between 0.03 and 0.08. However, in diamond-on-diamond tests, slowing the oscillation speeds caused a subtle but noticeable reduction in the average friction value by about 5%. Fourier transform infrared (FTIR) and Raman spectroscopy were used to examine tribochemical changes on the worn surfaces.

1:50pm **E3-1-2 Tribology of Deuterated Diamond-Like Carbon Films : An Imaging TOF-SIMS Study**, ***O.L. Eryilmaz*** *(eryilmaz@anl.gov), A. Erdemir, G. Kartal*, Argonne National Laboratory

In this study, we investigated friction and wear behavior of deuterated diamondlike carbon (DLC) films in dry nitrogen and hydrogen environments. The coatings were synthesized in a gas discharge plasma by applying 500 V rf. power to the substrates. Combination of deuterated methane, methane, hydrogen and/or deuterium mixtures with different ratios were used during deposition. The highly-hydrogenated or deuterated DLC films provided superlow friction (i.e., less than 0.01) regardless of the test environments, while the films that are hydrogen/deuterium-free or – poor had to be tested in hydrogen- and/or deuterium-containing test environments to attain low friction. When tested in dry nitrogen or vacuum, these hydrogen/deuterium-free films exhibited very high friction and wore out very quickly. All test were performed using pin-on-disk machines under 2-10 N loads and at 0.2 to 0.5 m/s sliding velocities. Near surface chemistry and depth profiles of sliding surfaces were analyzed by using time of flight secondary Ion Mass Spectroscopy (ToF-SIMS) and based on the results of surface analytical and tribological studies a mechanistic explanation was provided for the superlow friction behavior of hydrogen- and deuterium-rich DLC films.

2:10pm **E3-1-3 Super-Low Friction of Carbon-Based Coatings in Nitrogen Gas**, ***K. Adachi*** *(koshi@tribo.mech.tohoku.ac.jp)*, Tohoku University **INVITED**

Carbon-based coatings such as carbon nitride (CN_x)coatings exhibit a wide range of very attractive properties such as low friction and wear, good thermal and chemical stability which make them very suitable for demanding mechanical applications. In sliding of CN_x-coatings in nitrogen (N2) gas stream and/or atmosphere, it provides super low friction. The beneficial effect of N2 gas on reducing friction of carbon-based coatings is much more enhanced by selection of counter material, coating condition, surface roughness of the coatings, atmospheric humidity and running-in condition, which is pre-sliding before introducing N2 gas to the sliding interface. In this presentation, comprehensive overviews of the unique lubricious effects of "Nitrogen" on friction with carbon-based coatings are introduced. Based on those attractive results high potential of carbon-based coatings for future successful tribological usage by controlling atmospheric gases is discussed.

2:50pm **E3-1-5 Tribological Behavior of DLC Coated Spinal Disk Implants**, ***G. Thorwarth*** *(goetz.thorwarth@empa.ch), U. Müller, C.V. Falub, R. Hauert, B. Weisse,* Empa, Switzerland*, C. Voisard,* Synthes GmbH, Switzerland*, M. Tobler*, IonBond AG, Switzerland

Coatings from diamond-like carbon (DLC) have been proven to be an excellent choice for wear reduction in many technical applications. Also, a multitude of attempts for adaption to the MedTech field has been undertaken. However, data gained from realistic in-vitro test setups are in short supply despite being quintessential for application development; the prediction of layer stability and failure mechanisms in the human body environment are important tasks still to be researched.

In our team's efforts to develop DLC implant coatings with predictably stable layer adhesion in the human body, a new type of simulator has been developed for testing of spinal disk implants. Test results gained from this setup using coated and uncoated spinal disk replacements are presented and compared with conventional tribotests. The relevant differences and possible factors leading to implant failure are discussed. Requirements on the DLC and interfacial layers in the bio-tribo environment are suggested.

3:10pm **E3-1-6 Tribological and Mechanical Properties of Nanostructured Hydrogenated Amorphous Carbon and Titanium Diboride Films**, ***B. Zhao****, Y.W. Chung (ywchung@northwestern.edu)*, Northwestern University

Hydrogenated amorphous carbon films are of great interest due to their favorable ultra-low friction and low wear rate properties in dry environments. Our work demonstrated that sulfur doping of hydrogenated carbon films enabled them to achieve ultra-low friction performance in both dry and humid environments. However, these films have a hardness of 7 - 10 GPa and an elastic modulus around 80 GPa, which are too low for some high stress applications. Formation of nanostructured coatings is known to improve hardness. With the aim to produce hard, low-friction coatings, we synthesized nanolayered and nanocomposite films of sulfur-doped and undoped hydrogenated carbon and titanium diboride using dual-target magnetron sputtering. Titanium diboride deposited by this method had a

hardness >30 GPa. This paper will discuss the film structure and how such structure correlates with its tribological and mechanical properties.

3:30pm **E3-1-7 When and Why a-c:H Films are Hydrophobic?**, ***L.V Santos*** *(santoslv@las.inpe.br),* National Institute for Space Research - INPE, Brazil*, V.J. Trava-Airoldi, A.F. Azevedo, R.P.C.C. Statuti, P.A. Radi*, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil

Lots of people have been working with a-c:H films for different kinds applications one of that is protective hydrophobic coatings. The development of a-c:H hydrophobic films have been studding through power supply system deposition range , surface roughness, and from nom metallic chemical elements interactions. In this paper we showed that the hydrofobicity or hydrophilic tendency from a-c:H films changes with the hydrogen content in the bulk of the film. To support our argument we showed the Raman, ERDA and AFM analyses from five different a-c:H content films correlated with contact angle results. All results were correlated with roughness films topography analyzed by atomic force microscopy. The results showed that a-c:H films hydrofobicity increase when hydrogen content decrease. When the content of bulk film has huge hydrogen connections these bonds connections increase the chemical structural disorder of amorphous carbon film them it increase the hydro-affinity. Th e carbon and hydrogen bonds provides aromatic rings groups formation and the aromatic rings are more hydrophobic them some aliphatic bonds. In addition treating the low hydrogenated content carbon films surface with oxygen plasma for two minutes the hydrophobicity increases. These results show that to produced a-c:H with hydrophobic tendency is necessary increased the c-H bonds and decreasing structural disorder of the film.

3:50pm **E3-1-8 Friction and Wear Behavior of Hydrogenated Amorphous Diamond-Like Carbon (a-C:H) by Reactive Magnetron Sputtering in Water Environment**, ***B. Hilker*** *(hilker@iwt-bremen.de), H.-R. Stock, M. Diesselberg*, Stiftung Institut fuer Werkstofftechnik, Germany

Amorphous hydrogenated carbon (a-C:H) films are known to have low friction coefficients and high wear resistance. However, the main problems in tribological applications are emerging heat and abrasion. For ecological and economic reasons there is a need to reduce the use of oil-based lubrications. Water lubrication is a promising candidate as an alternative. For this reason the main focus of this work was to improve a-C:H films which show very good tribological characteristics in humid environment. The films were developed with dc pulsed reactive magnetron sputtering (Cemecon CC 800/9). For depositing the films onto polished steel discs we produced a transition layer to improve adhesion by decreasing the voltage of a chromium target, using nitrogen gas flow and increase the voltage of a graphite target. The top a-C:H layer were deposited by sputtering from graphite targets and reactive deposition from acetylene. We examined bias voltage and acetylene flow as well as heating or plasma etching duration before starting the sputtering process and their impact on hardness and average surface roughness of the resulting coatings. One factor that had a major influence on wear was the thickness of the a-C:H films. The optimised a-C:H films had a total thickness of 2 µm and a plastic hardness of 25 Gpa. The tribological properties were investigated by a pin-on-disc test in water. Under an applied load of 20 N the coated samples were slid against 100Cr6 pins and also a-C:H coated 100Cr6 pins. The a-C:H films showed low friction coefficients against a-C:H coated 100Cr6 pins of μ = 0.06. These tribological tests illustrate the potential of a-C:H films to effectively reduce friction and wear in humid environment.

4:10pm **E3-1-9 Water Adsorption on Phosphorus Carbide Thin Films**, *E. Broitman (broitman@andrew.cmu.edu),* Carnegie Mellon University*, **A Furlan**, G.K. Gueorguiev,* IFM, Linköping University, Sweden*, Zs Czigány,* Research Institute for Technical Physics and Materials Science, Hungary*, A.M. Tarditi,* Universidad Nacional del Litoral, Argentina*, A.J. Gellman,* Carnegie Mellon University*, S. Stafström,* IFM, Linköping University, Sweden*, L. Hultman*, Linköping University, Sweden

Amorphous phosphorus doped carbon (C_xP_y) thin films have been considered as a new tribological coating material with unique electrical properties. However, the material could not find practical applications so far since C_xP_y rapidly oxidizes, hydrolyzes, and delaminates when in contact with air. Recently, we demonstrated that phosphorus carbide (CP_x , $x \leq 0.15$) thin solid films with a short range fullerene-like structure can be deposited by magnetron sputtering. Thus, the introduction of P atoms in the graphene structure induces the formation of bent and interlinked graphene planes[1,2]. In this work we compare the uptake of water of amorphous phosphorus-carbide (a-CP_x) films, with fullerene-like phosphorus-carbide (FL-CP_x) and amorphous carbon (a-C) films. Films with thickness in the range 100-300 nm were deposited onto silicon, quartz, and gallium orthophosphate substrates by reactive DC magnetron sputtering. The film mi crostructure was characterized by X-ray photoelectron spectroscopy, and transmission electron microscopy and diffraction. A piezoelectric crystal microbalance placed in a vacuum chamber was used to measure film water adsorption[3]. Results indicate that the amount of adsorbed water is highest for the pure a-C films and that the FL-CP_x films adsorbed less water than a-CP_x. To provide additional insight into the atomic structure of defects in the FL-CP_x, a-CP_x, and a-C compounds, we performed first-principles calculations within the framework of Density Functional Theory. Emphasis was put on the energy cost for formation of vacancy defects and dangling bonds in relaxed systems[3]. Cohesive energy comparison reveals that the energy cost for formation for dangling bonds in different structural configurations is considerable higher in FL-CP_x than for the amorphous films. The simulations thus confirm the experimental results that dangling b onds are less likely in FL-CP_x than in a-CP_x and a-C films.

[1] A. Furlan, G.K. Gueorguiev, Zs. Czigány, H. Högberg, S. Stafström, and L. Hultman, Phys. Stat. Solidi RRL 2 (2008) 191.

[2] G. K. Gueorguiev, A. Furlan, H. Högberg, S. Stafström, and, L. Hultman, Chem. Phys. Lett. 426 (2006) 374.

[3] E. Broitman, G. K. Gueorguiev, A. Furlan, N. T. Son, A.J. Gellman, S. Stafström, and L. Hultman, Thin Solid Films, in press (2008).

4:30pm **E3-1-10 Measured and Simulated Wear and Friction Maps of DLCH20% and DLCH35% Films**, ***P.A. Radi*** *(polyana@las.inpe.br),* INPE - Instituto Nacional de Pesquisas Espaciais, Brazil*, L.V Santos,* INPE - Instituto Nacional de Pesquisas Espaciais and Instituto Technologico da Aeronautica - ITA, Brazil*, M.C.M Farias,* Laboratório de Fenômenos de Superfície (LFS) Escola Politécnica/USP, Brazil*, L.F. Bonetti,* INPE - National Institute for Space Research, Brazil*, V.J. Trava-Airoldi*, INPE - Instituto Nacional de Pesquisas Espaciais, Brazil

DLC (Diamond-Like Carbon) films exhibit unique mechanical and tribological properties and have applications on terrestrial and spatial environments. Depending on the hydrogen concentration on the DLC films and on the environment they can present high or low friction coefficient and wear rates. In this paper experimental results were extended to construct simulated wear and friction maps as a function of load and sliding speed in air and in vacuum to show the films behavior in different conditions as well as its potential for industrial and space application. Friction coefficient and wear rate of DLC films with 20% and 35% hydrogen content (DLCH20% and DLCH35%) produced under strictly controlled growth conditions on titanium alloy (Ti6Al4V) substrate were used to construct such maps. Additionally, the experimental results were used to develop a mathematical modeling of friction and wear of these films in order to identify the parameters that control friction and wear and to obtain the equation that describes these parameters. The techniques required to ensure high adhesion and film reproducibility are in the methodology. Hardness was measured and was considered as an additional reference in terms of DLC films quality. All tests were carried out at different loads and speeds. The results show the relationship between the DLC composition and the environment on the friction coefficient and wear of these films.

4:50pm **E3-1-11 Synthesis and Characterization of Nanocomposite and Nanolaminate Multilayers by High-Power Ion Beam Ablation***, *T.J. Renk (tjrenk@sandia.gov),* ***T.E. Buchheit****, S. Prasad*, Sandia National Laboratories

The Repetitive High Energy Pulse Power (RHEEP) facility at Sandia National Laboratories was used to generate ion-beams of 10 J/cm^2 energy. Ablating targets with the high energy ion-beams makes this facility a unique tool for low-stress multilayer thin film deposition. In the first part of this study, Mo-based films were grown on Si wafers, while keeping the substrate at 300°C during the deposition process with the intent of investigating MoS_2 films with improved tribological characteristics. Friction and wear measurements were performed using a Si_3N_4 ball in dry nitrogen and air with 50% relative humidity. Characterization of the films was performed on cross-sections of wear scars suitable for TEM analyses prepared using focused ion beam (FIB) microscopy. Results showed that dual target ablation of MoS_2 and Ti or V onto heated substrates permitted self-assembly of Mo species into 50 nm size boulders in the film structure. Consequen tly, unlike pure MoS_2 which oxidizes in humid air, the nanocomposites exhibited extremely low wear even when tested in air with 50%RH. Thus, we demonstrated that the nanostructure comprising of relatively hard particles of Mo metal in a matrix of softer MoS_2 provided a low friction and highly wear-resistant film that is also resistant to moisture.

By ablating different multiple metal targets, the RHEEP facility also demonstrated the capability for synthesizing a wide range of metal nanolaminate films, providing opportunities to understand fundamental mechanisms driving strength, stiffness and wear characteristics of this category of films. The metal laminate systems we investigated include Mo-Ir, Mo-Ti, and Mo-W. In these cases, nanolaminate films consisted of at least 100 alternating material layers, each layer less than 10 nm thick. Films were deposited on Silicon and Al_2O_3 substrates. The mechanical response of these nanolaminates was in vestigated using instrumented indentation. The

role of layer thickness and shear modulus difference on the strength and wear characteristics of the nanolaminate films will be discussed.

[1] Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000.

5:10pm **E3-1-12 Wear-Resistant and Low-Friction Diamond-Like-Carbon (DLC)-Layers for the Wood Machining Industry**, *W. Tillmann (wolfgang.tillmann@udo.edu),* Technische Univeristaet Dortmund, Germany, *E. Vogli,* ***F. Hoffmann***, Technische Universitaet Dortmund, Germany

Forestry, timber-, and paper-industry in Europe have a production value of approximately 400 billion Euros per year. For this reason, the supplying industry is very conscious to develop highly efficient tools. Especially wear and high friction of cutting tools are limiting factors in the processing of wood and wood-based products. Excellent wear and friction properties increase the tool life just as affecting the power consumption, the surface finish of the workpiece and the production rate in a positive way. To facilitate higher productivity at lower operating cost, it is of particular importance to use tools with enhanced wear and friction attributes. Novel amorphous diamond-like-carbon (DLC) coated tools provide these properties in dry and even in humid environment. This work is focused on the development of a DLC-system with high wear and friction resistance also under humid conditions which especially exist during the processing of wood and wood-based products. Usi ng the Physical Vapor Deposition (PVD)-process different DLC-coating systems have been deposited, in which the layer properties have been designed related to the humidity conditions. Wooden counterparts were used during wear and friction tests to analyse the tribological behaviour of the coatings. Furthermore Raman spectroscopy was applied to characterize the layers microstructure. The layer properties and coating parameters have been systematically analyzed with special emphasis on tribological attributes. Correlations between layer structure and corresponding wear and friction properties have been scrutinized.

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G2-1

Coatings for Automotive and Aerospace Applications
Moderator: R. Mertens, Oerlikon Balzers AG, G. Dadheech, General Motors

1:30pm **G2-1-1 Design of Novel Metallic Corrosion Protective Coatings on Steel Sheet by Means of Thin Film Technologies**, ***R. Weinhold*** *(richard.weinhold@thyssenkrupp.com), B. Schuhmacher,* Dortmunder OberflächenCentrum, Germany, *M. Rohwerder,* Max-Planck-Institut für Eisenforschung, Germany, *C.-P. Klages*, Institut für Oberflächentechnik, TU Braunschweig, Germany

For steel strip used in automotive applications an effective corrosion protection has to be provided, which is often realized by conventional zinc or zinc alloy coatings with a thickness of 7-10 µm. New vacuum processes allow the deposition of thin multi-layer coating systems having an improved corrosion resistance and – due to a significantly reduced thickness - a better workability. Within this work, a multi-layer coating system consisting of three metal layers (zinc, magnesium and aluminium) with a total thickness of less than 3 µm was deposited on steel sheet using a PVD- or a combined electro galvanizing/PVD-process. A thermal treatment was applied in order to adjust the structure of the coating system.

The coatings were characterized by means of scanning electron microscopy (SEM), glow discharge spectroscopy (GDOS) and X-ray photoelectron spectroscopy (XPS). The corrosion behaviour was examined in a cyclic corrosion test according to standard 621-415 of the German car makers (VDA) using specific flange samples. A significant increase in flange corrosion resistance of thin film coatings compared to commonly used 7.5 µm electro-galvanized zinc (EG) coatings can be attested. Furthermore, thin film coatings showed an excellent laser-weldability in overlap mode without gap, which is relevant for an efficient use in automotive applications.

The excellent corrosion resistance and good workability achieved indicates that PVD processes for metallic layers are promising to be enhanced as an advanced industrial technology for coated steel strip products.

1:50pm **G2-1-2 Arc Ion Plating System for Coating Piston Ring**, ***H. Fujii*** *(fujii.hirofumi@kobelco.com),* Kobe Steel, Ltd., Japan, *M. Osako, K. Kusaka*, Shinko Techno Engineering Co., Ltd., Japan

Cathodic arc evaporation (AIP : Arc Ion Plating) process, which provides a good performance thin film with a superior adhesion and a good wear resistance, is widely adopted in many industrial fields such as cutting tools and mechanical/automobile parts. CrN-coated piston ring, one of those to which AIP process were successfully adopted, has been used for about 15 years and has been contributing to a remarkable improvement of diesel/gasoline engine performance and fuel consumption rate. Generally, the film thickness for piston ring is 20-50 µm, which is so much thicker than that for normal cutting tools or mechanical parts. So not only high productivity but also high reliability as much as withstanding a very long coating operation is strongly required for coating equipment used for piston rings. The coating cost must, needless to say, be reduced as using for parts of mass-produced automobiles. In order to meet these requests, the new AIP system has been developed, which is featured by newly developed rod type evaporation source, water-cooled rotary table and twin door system with slant seal face. The arc spot position on a rod target can precisely be controlled even with over 1000A arc current, and the substrate temperature during coating process gets greatly lowered.

2:10pm **G2-1-3 Atmospheric Plasma - Cleaning, Activation and Coating of Materials Surfaces by Openair® Plasma**, ***C. Buske*** *(christian.buske@plasmatreat.de)*, Plasmatreat GmbH, Germany **INVITED**

Since its inception in 1995, Plasmatreat, which today is a globally operating company, has focused on the development of atmospheric-pressure plasma processes. Openair®- Plasma technology is protected internationally by patents and is em-ployed in almost all fields of industrial production.

The technique based on a jet principle operates at atmospheric pressure. The jets are operated only with air, possibly also with another desired process gas, and at high voltage. A particular characteristic of the emerging beam of plasma is that it is electrically neutral which greatly extends and simplifies its range of uses. Its intensity is so high that machining speeds of several 100 m/min can be attained. The Openair® Plasma technique is characterised by a threefold action: it activates a surface by selective oxidation processes, eliminates static charge and brings about microfine cleaning. The jet systems employed require neither a complex vacuum chamber nor do they interrupt the production process. The system is capable without restriction of implementation in-line and is compatible with robots, offering extraordinary cost-effective solutions.

If a special precursor material is added to the Openair® process, selective nanocoating of surfaces can be accomplished. Whether the aim is to provide protection against corrosion or to facilitate cleaning of a surface, the new PlasmaPlus® technology, developed by Plasmatreat in collaboration with the internationally renown institute Fraunhofer IFAM, is a unique possibility to offer an abundance of differently functionalised layers for selective coating. The properties of these layers can be varied from hydrophilic to hydrophobic by using different precursors and the carbon content of the individual layers can be varied from organic to very inorganic. These properties enable the technology to be customized for different material surfaces like PC,PP,PET and aluminum. The anticorrosive action is particularly effective for aluminium alloys.

Due to the small quantities and non-toxicity of the chemicals used in coating the process is highly environmentally friendly. The removal of coatings prior to a recycling process is not required, the coatings can be passed on for recycling together with the substrate material. Since vacuum chambers are not required, the process is especially useful for huge parts or high automated mass production.

In December 2007 America's leading finishing journal Finishing Today Magazine (FTM) honored Plasmatreat with the FTM Innovation Award 2007 in the processing technology category. Plasmatreat received the prize for a revolutionary new environmentally friendly process in the surface pretreatment of aluminium.

The Plasmatreat Group has an international presence through technology centers in Germany, the United States and in Japan as well as sales offices around the globe.

The presentation will give an overview of this plasma technology as well as the latest results from Fraunhofer Institute for thin film coating.

2:50pm **G2-1-5 Low Temperature Reactive Magnetron Sputtered nc-Ti(N,C)/a-C:H Coatings for Automotive Applications**, ***C. Tsotsos*** *(tsotsos@ucy.ac.cy), K. Polychronopoulou,* University of Cyrpus*, N. Demas,* University of Illinois at Urbana-Champaign*, M. Baker, Y. Chen,* University of Surrey, United Kingdom*, K. Kanakis, A. Leyland, A. Matthews,* University of Sheffield, United Kingdom*, C. Rebholz,* University of Cyprus*, A.A. Polycarpou*, University of Illinois at Urbana-Champaign

Tribological coatings targeted for automotive applications are required to be functionally graded using a different approach to that of coatings developed for cutting tool applications. Depending on the operating environment such coatings do not necessarily require a high hardness but instead an optimal H/E ratio, along with relatively low friction, high corrosion resistance and thermal stability at moderate operating temperatures. 2.5µm thick nc-Ti(N,C)/a-C:H coatings containing ≥ 50% amorphous C:H phase were deposited, using low temperature (~200°C) DC reactive magnetron sputtering, on 100Cr6 steel substrates. The a-C:H phase content was varied with acetylene partial pressure. XPS analysis indicates that the main coating structure is that of TiN with C substituting for N for the crystalline phase and sp^2 dominant a-C:H phase. TEM analysis is used to characterise the coating structure and ascertain the volume fraction of the a-C:H phase. The me chanical properties are affected by the nc-Ti(N,C)/a-C:H phases volume fraction ratio and the nanoindentation hardness (H) values were measured and found to range from 9 to15GPa and the reduced Young's modulus values (E) from 80 to 150 GPa. The synthesized coatings were tested using pin-on-disc sliding configuration and the measured friction coefficient values were around 0.2 and survived up to 100 m sliding distance. The performance of the coatings in corrosion resistance tests is greater for predominantly a-C:H coatings. nc-Ti(N,C)/a-C:H coatings are promising candidates for automotive applications offering a lower mismatch in mechanical properties between a relatively low modulus steel substrate and a low modulus low friction topcoat layers.

3:10pm **G2-1-6 Gas Flow Sputtering - An Approach to Coat Complex Geometries and Non-Line-of-Sight Areas**, ***S. Tang*** *(stanley.tang@dlr.de), U. Schulz*, German Aerospace Center, Germany

Most PVD techniques are limited in coating complex geometries like turbine blades without additional substrate manipulation. Their application is subject to the Line-of-Sight condition that means they can only coat areas of the substrate which are directly in view to the sputtering or evaporation source and shaded areas can not be coated. If a coating on the whole substrate surface is required, the component has to rotate and process parameters have to be adapted to the requirements. A novel approach to overcome these limits is the innovative Gas Flow Sputtering. This technique combines a hollow cathode glow discharge with an argon gas flow, which supports transportation of sputtered material to the substrate. The mixture of gas and coating material performs a circulation around the contour of the geometry and reaches Non-Line-of-Sight areas. Distribution of coating thickness depends on gas flow kinetics and dynamics. In these investigation different complex geometries such as u-shaped profiles, pipes and turbine blades were coated with pure titanium. The influence of the angle between gas flow direction and substrate surface (angle of impact) on coating thickness distribution, microstructure and crystal orientation was examined by SEM and XRD. To investigate the correlation between gas flow formation and coating thickness distribution some coating procedures were simulated by Computational Fluid Dynamics (CFD). Results show that a stripe or a pipe placed parallel to the gas flow direction is coated over the whole length. Pipes are coated outside as well as inside. The outer diameter of a pipe positioned perpendicular to the gas flow direction is completely covered with material. Interestingly, a coated turbine blade that was not rotated shows a maximum coating thickness at the leading and trailing edge. The microstructure of the coatings is correlated with gas flow and deposition conditions, while the CFD results helped to understand coating growth on complex geometries.

3:30pm **G2-1-7 Thick Plasma Enhanced PVD Coatings for Weapons Applications**, ***S.L. Lee*** *(SabrinaLinLee@cs.com), M. Todaro,* US Army ARDEC-Benét Labs*, R. Wei, E. Langa*, Southwest Research Institute

Plasma-enhanced PVD (physical vapor deposition) process was investigated to deposit thick coating for potential weapon system applications. In the enhanced process, externally generated plasma and substrate biasing were used to deposit coatings on weapon system parts with complex geometry. Tantalum coatings of 200-500µm thickness were sputter deposited on ASTM A723 steel samples cut from curved 120mm smooth bore, and rifled 155mm gun bore sections. SEM showed dense, adhesive, crack-free coatings with improved morphology. XRD showed (110) textured body-centered-cubic tantalum. Fracture tantalum surface showed excellent microvoid coalescence with ductile mode of fracture, resilient to thermal shock cracking. Adhesion tests were performed; including groove test, cyclic pulsed laser heating test, and vented erosion simulator test, simulating the thermal-mechanical-chemical environment of firing. Interior ballistic model of heat load and heat transfer programs were used to simulate the temperature profile for pulsed laser heating test. The plasma enhanced PVD deposition process, the analytic and adhesion test results for thick tantalum depositions on steel, and future potential applications of the techniques will be discussed.

3:50pm **G2-1-8 Enabling Lightweight, High Load Aero-Bearings**, *J. Avelar-Batista Wilson, S. Banfield,* Tecvac Ltd, United Kingdom*, B. Karadia, N. Vahegla,* Airbus, United Kingdom*, P. Smith,* Cranfield University, United Kingdom*, G. Cassar, A. Leyland, A. Matthews,* The University of Sheffield, United Kingdom*,* ***J. Housden*** *(jonathan.housden@tecvac.com)*, TECVAC Ltd, United Kingdom

Environmental and commercial considerations are strongly driving research into weight saving in aircraft. In this research, innovative manufacturing processes were developed to produce lightweight titanium alloy bearings capable of withstanding high bearing pressures. This will enable the replacement of heavier conventional bearing materials with titanium alloy bearings of the same size thereby saving weight. Plasma processing and PVD coating techniques were refined and combined and a sound scientific understanding of the resulting novel processes developed to assure high performance, reliability and repeatability. FEA modelling fed into developments and trials on test samples and small bearings showed the effects on torque and wear by applying progressively greater pressures with the optimum result coating being applied to a full size bearing for full-load simulated aircraft-lifetime tests. The novel treatment has potential applications for many bearings and bearing surfaces throughout aircraft.

New Horizons in Coatings and Thin Films
Room: Sunset - Session H2-2

High Power Impulse Magnetron Sputtering
Moderator: A.P. Ehiasarian, Sheffield Hallam University, K. Marchev, P&G Company

1:30pm **H2-2-1 Modulated Pulse Power Sputtered Chromium and Chromium Nitride Coatings**, ***J. Lin*** *(jlin@mines.edu), J.J. Moore,* Colorado School of Mines*, W.D. Sproul,* Reactive Sputtering, Inc.*, B. Mishra,* Colorado School of Mines*, Z.L. Wu,* Colorado School of Mines, and Dalian University of Technology, China*, M. Hasheminiasari, S. Myers,* Colorado School of Mines*, R. Chistyakov,* Zond, Inc.*, B. Abraham*, Zpulser, LLC

Modulated pulse power (MPP) sputtering is a variation of high power pulse magnetron sputtering that overcomes the rate loss issue and achieves the enhanced plasma ionization through modulation of the pulse shape, intensity, and duration. In the current study, Cr and CrN coatings were synthesized using MPP in a closed field unbalanced magnetron sputtering system under various pulse durations and the pulse modulations, which in turn strongly affect the target power density, voltage, current, and ion current density. It was found that MPP sputtering exhibits higher deposition rates than in the dc conditions when the average target power is above 10-12 W/cm^2 for the Cr coating depositions. Plasma diagnostics proved that extremely high level of metal ion flux with low ion energies (less than 10 eV) was identified in the MPP plasma compared to the relatively low metal ion flux in the dc and pulsed dc discharged plasma. The structure and properties of Cr and CrN coatin gs were characterized using x-ray diffraction, scanning electron microscopy, transmission electron microscopy, nanoindentation tests, and ball-on-disc wear test, and further compared with those synthesized using dc and pulsed dc magnetron sputtering. A high hardness of 28 GPa has been achieved in CrN coatings deposited in MPP conditions with a floating substrate bias. The MPP CrN coatings also exhibit dense near equi-axial structure and improved tribological properties and oxidation resistance as compared to the dc CrN coatings.

1:50pm **H2-2-3 Physical Properties and Potential Applications of High Power Impulse Magnetron Sputtering Plus (HIPIMS+) Deposited Chromium Nitride and Titanium Nitride Coatings**, *F. Papa (fpapa@hauzer.nl),* ***C. Strondl****, I. Kolev, T. Krug, R. Tietema*, Hauzer Techno Coating BV, Netherlands

Chromium Nitride (CrN) and Titanium Nitride (TiN) coatings are two well studied coatings which have played a significant role in the industrialization of PVD coatings. These coatings are typically deposited via magnetron sputtering or cathodic arc. However, with the current industrialization of High Power Impulse Magnetron Sputtering (HIPIMS) technology, a new process window for such coatings is now open due to the partial ionization of the sputtered material. Due to this ionization, the coating structure and properties can be uniquely tailored. The results of coating analysis will be

presented. These will include physical and mechanical properties such as crystal texture, stress and film structure. Potential industrial applications for such coatings will also be discussed.

2:10pm **H2-2-4 Low Friction CrN/TiN Multilayer Coatings Prepared by a Hybrid HIPIMS/UBMS Process**, ***J. Paulitsch*** *(joerg.paulitsch@mcl.at),* Materials Center Leoben Forschung GmbH, Austria*, P.H. Mayrhofer,* University of Leoben, Austria*, M. Schenkel*, SVS Vacuum Coatings Technologies, Germany

CrN and TiN coatings are known for their high hardness and good wear resistance. Many research activities concentrate on the correlation between plasma conditions, microstructure and resulting properties of these coatings. Generally, the density and the mechanical properties of coatings can be improved by increasing the energy and the density of ions in the plasma. As high power impulse magnetron sputtering (HIPIMS) is known to allow high ion densities in the plasma we study the structure and mechanical properties of CrN/TiN multilayer coatings deposited by a combination of HIPIMS with conventional unbalanced magnetron sputtering (UBMS). Here we show primary results on structure, mechanical and tribological properties of CrN/TiN multilayer coatings deposited by the combined HIPIMS/UBMS deposition technique. Hardness values, obtained by an ultra micro indentation system nanoindenter, are approximately 25 GPa. The friction coefficient during dry sliding ball on disk tribo meter tests is approximately 0.2 at RT, and the wear coefficient is 6•10-16 m^3/Nm, as evaluated from the wear track by optical profilometry. Structural analyses and details on the interface regions of the grown coatings are conducted by transmissions electron microscopy, scanning electron microscopy and X-ray diffraction. Our results clearly demonstrate that low-friction and wear-resistant CrN/TiN multilayer films can be prepared by a hybrid sputtering process combining HIPIMS and UBMS.

2:30pm **H2-2-5 Industrial-Scale Deposition of Highly Adherent CN_x Films on Steel Substrates**, ***E. Broitman*** *(broitman@andrew.cmu.edu),* Carnegie Mellon University*, Zs Czigány,* Research Institute for Technical Physics and Materials Science, Hungary*, R. Cremer,* CemeCon AG, Germany*, X. Zhou,* SKF Engineering and Research Center, Netherlands*, L. Hultman*, Linköping University, Sweden

Due to their superior wear resistance, high hardness, and low friction coefficient, carbon nitride (CN_x) coatings have been proposed as the best candidates to replace diamond-like carbon (DLC) films. The first successful industrial application of this material has been the use of very thin (~2 nm) films for the protection of hard disks since the first applications were obviously for thicker topcoats. However, the scalability of CN_x coatings produced in laboratories to industrial scale has been difficult in applications where thicker films (1-5 μm) on steel substrates were required. The main reason is the development of high compressive intrinsic stresses during deposition which causes mechanical damage, i.e., adhesion failures and delamination of films from the substrate surface. The common practice to increase the adhesion of carbon-based coatings on steel substrates is to make a pretreatment of the substrates and the use a glue layer interposed betwe en the surface substrate and the film. Recently, it has been reported that high power impulse magnetron sputtering (HIPIMS) technology produces coatings with adhesion exceeding that of arc methods. However, there are still some technical problems to solve: during the discharge, the high discharge current produces instabilities in the DC bias power supply. In this paper we study the deposition of highly adherent CN_x films using a novel HIPIMS pretreatment where two HIPIMS power supplies are used: one to establish the discharge and one to produce a pulsed substrate bias. All processing was done in a commercial CEMCON CC800 system. During the pretreatment, SKF3 steel substrates were pulse-biased in the environment of a HIPIMS of Cr plasma in order to sputter clean the surface and to implant Cr metal ions. Subsequently, carbon nitride films were prepared by DC unbalanced magnetron sputtering from a high purity graphite target in a N_2/Ar discharge at 3 mTorr. A series of depositions were obtained with samples at different bias voltages (DC and pulsed). X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), scanning transmission microscopy (STEM) and high resolution transmission electron microscopy (HRTEM) has been use to study the microstructure and the nature of the interface steel/Cr/CN_x. Identification of coating layer failures was done by the Daimler-Benz Rockwell-C adhesion test, scratch test, and Calotest.

2:50pm **H2-2-6 Development of Ready to use Cr_2AlC Max-Phase on Complex Geometries**, ***O. Schroeter*** *(schroeter@tu-cottbus.de), A. Flores Renteria,* Brandenburg University of Technology at Cottbus, Germany*, C. Leyens*, TU-Cottbus, Germany

Due to its thermo-mechanical properties, Cr_2AlC is one of the interesting MAX-phases. It is known that these materials display good mechanical properties due to their nano-laminated structure, combining the characteristics of metals and ceramics. This is interesting with regard to damage tolerant protective coatings working in an oxidative environment. This work is focused on the deposition of Cr_2AlC-coatings in an industrial PVD-coater. One of the critical steps on the application of new technologies is the up-scaling process. The use of an industrial-scale coater allows an ideal optimization of the coatings manufacture, facilitating the transfer of technology from research to industry. The obtained results demonstrate that the crystallization process of the manufactured coatings was enhanced by a post heat-treatment within the coater, applied directly at the end of the coating process. Furthermore, the distribution of the coating´s thickness and ch emical composition on complex geometries was improved by the use of high power impulse magnetron sputtering (HIPIMS) compared to direct current – magnetron sputtering (DC-MS). The enhancement of the coating´s homogeneity was achieved by using the Al-target in an HIPIMS-modus.

3:10pm **H2-2-7 Impact Behavior of (Ti,Al,Si)N Deposited by HPPMS**, *K. Bobzin,* ***N. Bagcivan*** *(bagcivan@iot.rwth-aachen.de), S. Bolz*, RWTH Aachen University, Germany

Over the last decade the interest in High Power Pulse Magnetron Sputtering (HPPMS) and High impulse Magnetron Sputtering (HiPIMS) has undergone a considerable increase. This is mainly due to the fact that several researchers have shown that in these processes a distinct increase of the ionization of deposition species is observable. This enables an active manipulation of the deposition flux eliminating line of sight behavior of sputter processes. So far the understanding of the processes within the plasma has grown steadily. Moreover some researchers have deposited different films using HPPMS or HiPIMS. However there is only little known about the performance of these films with regard to applications. Recently Hovsepian et al.[1] and Bobzin et al.[2] presented cutting results of different films. Both authors show that films deposited using HPPMS or HiPIMS outperform state-of-the-art coatings. Coatings for that kind of applications in harsh conditions like in cutting have to provide outstanding properties. Depending on the cutting process, besides hardness and adhesion also excellent impact behavior is expected. Until now there have been reported no results about the impact behavior of HPPMS or HiPIMS cotings. Therefore this work deals with the impact behavior of (Ti,Al,Si)N which was deposited using HPPMS for the application in interrupted cutting process. The impact behavior of HPPMS coating under normal and tangential loads is compared with a DC magnetron sputtered one of the same composition and coating thickness. During impact tests number of impacts, loads and inclination angle of the samples with regard to the load direction are varied. The results are related to scratch tests, morphological and topological analysis using optical and secondary electron microscopy.

[1]P. Eh. Hovsepian, A.P. Ehiasarian, A. Deeming, C. Schimpf, Vacuum 82 (2008) 1312-1317

[2]K. Bobzin, N. Bagcivan, P. Immich, S. Bolz, T. Leyendecker, R. Cremer, presented at PSE conference 2008, Garmisch-Patenkirchen, Germany.

3:30pm **H2-2-8 Modulated Pulse Power Deposition of Optical Coatings**, ***R. Chistyakov*** *(rchistyakov@zondinc.com),* Zond, Inc.*, B. Abraham,* Zpulser, LLC*, W.D. Sproul,* Reactive Sputtering, Inc.*, J.J. Moore, J. Lin*, Colorado School of Mines

Modulated pulse power (MPP) sputtering is a variation of high power pulse magnetron sputtering that overcomes the rate loss issue through modulation of the pulse shape, intensity, and duration. Usually MPP uses a two-step voltage pulse to create highly ionized magnetron plasma. The first voltage step creates a low power magnetron discharge, and once it is stable the cathode voltage is modulated with an increase of this voltage, resulting in the generation of high power magnetron discharge and strongly ionized plasma. Total pulse time is typically 1-3 milliseconds. The pulse shape and duration and plasma perturbations directly affect the degree of ionization of the sputtered material. In this study, silicon, silver and reactive Al-doped zinc oxide and ITO films were deposited with the modulated pulse power sputtering approach. The applied voltage pulse shape to the magnetron generated a high power pulse discharge and directly affected the degree of ionization of the sput tered material. Nanometer scale layers of silicon, silver and reactive Al-doped zinc oxide and ITO films were alternately deposited, the thickness and structure of each nanolayer was controlled by varying the output voltage pulse shape of the MPP plasma generator. The OES of plasmas, film structure, orientation, and surface roughness were analyzed and measured. The results of the film property measurements and OES will be presented.

3:50pm **H2-2-9 Arbitrary Voltage Pulse Shape Plasma Generator with RF Capabilities for Material Processing**, *R. Chistyakov (rchistyakov@zondinc.com),* ***B. Abraham***, Zond Inc.

A new arbitrary voltage pulse shape plasma generator with capabilities to generate RF discharge was developed for RF superimposed high power pulse magnetron sputtering. Plasma generator consists from two units; arbitrary voltage pulse shape unit and RF unit. A special designed RF filter

prevents RF power to penetrate inside the arbitrary voltage pulse shape unit. An arbitrary voltage pulse shape plasma generator with RF discharge capabilities gives unique opportunity for controlling plasma parameters. The principals of operation of new arbitrary voltage pulse shape plasma generator with RF generation capabilities will be presented. Method of generating multi step voltage pulses in the presence of RF discharge will be discussed.

4:10pm **H2-2-10 An Investigation of Magnetron Magnetic Field Strength Requirements for HIPIMS**, *P.J. Kelly, P. Barker,* Manchester Metropolitan University, United Kingdom, *D. Ochs,* Hüttinger Elektronik GmbH, Germany, ***A.G. Spencer*** *(rick@alacritas.net)*, Alacritas Consultancy Ltd., United Kingdom

High Power Impulse Magnetron Sputtering (HIPIMS) is an exciting technique to produce a highly ionized sputter flux for film densification, surface modification, trench filling, adhesion promotion and other applications. Layers produced with HIPIMS show superior properties in many applications. The most investigated and promising HIPIMS application is for hard coatings in wear and corrosion protection. HIPIMS significantly changes the hysteresis curve in reactive sputtering, offering much higher deposition rates of compound thin films. Also the HIPIMS process has a significantly lower substrate heat load than standard magnetron sputtering enabling high rate coating even on temperature sensitive substrates.

HIPIMS power supplies can be added to existing sputter systems with little or no system modification, making them attractive as a way of extending process capability. However the magnetron magnetic field strength must be significantly higher for HIPIMS than for standard DC sputtering. If the magnetic field is too low then the required voltage in the HIPIMS pulse rises and the desired pulse currents cannot be reached. The impact of magnetic field strength/degree of unbalance on the voltage/Current dependence has been investigated and is reported here.

Coatings for Use at High Temperature
Room: Town & Country - Session AP

Symposium A Poster Session

AP-1 Hard Protective Si-Zr-O Coatings Resistant to Thermal Cycling in Air up to 1400°C, *J. Musil (musil@kfy.zcu.cz),* ***V. Satava****, P. Zeman, R. Cerstvy*, University of West Bohemia, Czech Republic

The article reports on structure, mechanical properties and oxidation resistance of 7000 nm thick Si-Zr-O film reactively sputtered using a closed magnetic field dual magnetron system operated in ac pulse mode. The films were sputtered from a composed target. Main attention is devoted to the investigation of the effect of the structure of film on the thermal stability of its mechanical properties and oxidation resistance. It was found that (1) the $Si_{31}Zr_5O_{64}$ film sputtered at the substrate temperature T_s=500°C is amorphous, (2) the $Si_{31}Zr_5O_{64}$ film is sputtered at high deposition rate a_D=100 nm/min, (3) the structure of $Si_{31}Zr_5O_{64}$ film gradually changes during thermal annealing in air from the amorphous to the polycrystalline with t-ZrO_2 phase, (4) the t-ZrO_2 phase is stable in wide range of T_a up to 1550°C and no conversion of the t-ZrO_2 phase into m-ZrO_2 phas e is observed during subsequent cooling from 1550°C down to room temperature RT, (5) the hardness H and effective Young s modulus E* of $Si_{31}Zr_5O_{64}$ film are thermally stable and resistant to the oxidation in flowing air during thermal cycling from room temperature (RT) up to 1400°C for 4 hours with T_a=1000°C and (6) the $Si_{31}Zr_5O_{64}$ film converts into a surface layer composed of a mixture of t-ZrO_2+m-ZrO_2+$Al_6Si_2O_{13}$+SiO_2 phases at annealing temperatures T_a=1550°C due to its strong interaction with the Al_2O_3 substrate. Main issue of this investigation is the finding that the properties of protective coating do not change during thermal cycling as far the structure of the coating is unchanged during increasing and decreasing of the annealing temperature T_a.

AP-2 Thermal Stability and Mechanical Properties of CrZr-Y-N Coatings Synthesized by Closed Field Unbalanced Magnetron Sputtering, ***S.C. Oh****, G.S. Kim, Y.S. Kim, S.M. Kim,* Korea Aerospace University, Korea*, S.Y. Lee (sylee@kau.ac.kr)*, Korea Aerospace University, South Korea

Ternary Cr-Zr-N films synthesized by a closed field unbalanced magnetron sputtering with vertical magnetron sources showed quite unique characteristics in that, compared with CrN, they have not only much improved mechanical properties, but also a very low surface roughness, and their surface became smoother with increasing Zr content. However, investigations on the high temperature characteristics of the Cr-Zr-N films revealed that their mechanical properties deteriorated severely with increasing Zr content at 500°C due to the surface roughness increment as well as the hardness reduction by the oxidation. In this paper, the CrZr-Y-N films were developed by adding the Y element into the Cr-Zr-N ternary system for high temperature applications. The films were synthesized by closed field unbalanced magnetron sputtering (CFUBMS) with vertical magnetron sources and their chemical composition, crystalline structure, morphology and mechanical properties were characteriz ed by energy dispersive X-ray spectrometer (EDS), X-ray diffraction (XRD), scanning electron microscopy (SEM), atomic force microscopy (AFM), and nanoindentation tests. In addition, the characteristics such as hardness, surface morphology, friction coefficient of the films as a function of the Y content after annealing up to 700°C in air were evaluated and compared with those of Cr-Zr-N coatings. Preliminary results showed that the average friction coefficient of the Cr-Zr-N coatings increased significantly from approximately 0.23 to 0.81 as the wear test temperature increased from room temperature to 500°C, but the average friction coefficient of the CrZr-Y-N coatings did not changed as extensively as the Cr-Zr-N films did. Also, the thermal stability of the CrZr-Y-N coatings was improved significantly than that of Cr-Zr-N coatings. Detailed experimental results on the thermal stability and mechanical properties of the CrZr-Y-N coatings will be presented.

AP-3 Self Organised Nanostructuring for Drag Reduction and Self-Cleaning in Composite Coatings for High-Temperature Applications, ***P. Schaaf*** *(peter.schaaf@tu-ilmenau.de), M. Hopfeld,* TU Ilmenau, Germany*, V. Drescher, J. Wilden*, TU Berlin, Germany

Coated and nanostructured surfaces gain much importance for improving the efficiency of high temperature applications (e.g. in turbines). By embedding ceramic particles with a negative thermal expansion coefficient (NTEC) into a metallic matrix, a reversible thermal activation of a nanostructured surface can be established. At high temperatures a defined drag reducing surface microstructure ("shark-skin") is formed in the the coating surface, while a self-cleaning effect at low temperatures (in idle period) is achieved by the reversal of the deformation. A feedstock powder produced by high energy ball milling and consisting of nanocrystalline yttrium oxide and tungsten oxide particles embedded into a conventional MCrAlY alloy was used for the investigations. By using different thermal spray and cladding techniques the powder is deposited onto different substrates. In a next step the coating is implanted with oxygen, yttrium, or xenon to induce the formation of Y2W3O 12 particles inside the coating in the desired morphology. Y2W3O12 is a ceramic with a strong negative thermal expansion coefficient and is stable also at temperatures above 1373 K. The effects on phase formation and morphology changes are analyzed in detail. Results of phase formation, surface micro-morphology, microstructure and properties of these high-temperature coatings are presented.

Work is supported by the German Research Foundation (DFG) within Priority Programme 1299 (HAUT).

AP-4 Analysis of Hf-Rich Precipitates in NiAl-Hf Bond Coats via Transmission Electron Microscopy and Atom Probe Tomography, ***M.A. Bestor****, M.S. Kirsch, R.L. Martens, M.L. Weaver (Mweaver@eng.ua.edu)*, The University of Alabama

It has been reported that NiAl-based bond overlay coatings with concentrations of reactive elements (e.g. Hf and Zr) in excess of their solubility limits can exhibit oxidation resistance comparable to state-of-the-art platinum aluminides. In this study, a microstructural investigation was conducted using atom probe tomography (ATP) and transmission electron microscopy of precipitation/dispersion strengthened NiAl-Hf bond overlay coatings deposited using DC magnetron sputtering onto a single crystal Ni-based superalloy. Post-deposition annealing of NiAl-1.0 at% Hf coatings at 1000°C for one to four hours produced nanometer-sized Hf-rich precipitates. Longer pre-oxidation annealing times resulted in smaller mass gains during isothermal oxidation with values approaching those of model NiAl-0.05 at.% Hf coating alloys. It is believed that the precipitates improve the oxidation performance of these coatings by inhibiting the inward diffusion of oxygen and the upward diffusion of aluminum. High angle annular dark field (HAADF) images showed that the Hf segregates to the grain boundaries and also forms precipitates after annealing. APT determined that the precipitates were disk-shaped, Hf-rich, and that carbon localizes with the precipitates. Results suggest that suitable oxidation resistance can be obtained in "over-doped" alloys by optimizing the coating chemistry, grain structure and precipitate distribution.

AP-5 Properties of the Thermally Stable Si-B-C-N Coatings Prepared by Reactive dc Magnetron Co-Sputtering, ***P. Calta*** *(pcalta@kfy.zcu.cz), J. Capek, P. Zeman, P. Steidl, R. Cerstvy, J. Vlcek*, University of West Bohemia, Czech Republic

Novel quaternary Si-B-C-N materials are becoming increasingly attractive because of their possible high-temperature and harsh-environment applications. In our previous investigation, the Si-B-C-N coatings were prepared by reactive dc magnetron co-sputtering using a single C-Si-B or B_4C-Si target in nitrogen-argon gas mixtures at an rf induced negative substrate bias voltage and the substrate temperature T_s = 350°C kept by an Ohmic heater. In this work, the Si-B-C-N coatings were deposited using the B_4C-Si target with a fixed 75% Si fraction in the target erosion area on floating Si, SiC, Cu and glass substrates (U_f = -20 to -40 V) without an additional heating (T_s = 180 – 250°C), i. e., under simplified experimental conditions. The total pressure of the N_2-Ar gas mixture of 0.5 Pa and the discharge current on the magnetron target of 1 A were held constant. The effect of the gas mixture composition on mechanical, optical and electrical properties of the coatings, and their high-temperature oxidation resistance and thermal stability in inert gases (He and Ar) were investigated. The coatings, typically 2 μm thick, were found to be amorphous with high hardness (around 21 Gpa) and low compressive stress (approximately 0.5 Gpa). With the increasing Ar fraction in the gas mixture, their optical transparency decreases while their electrical conductivity raises (up to 5 Sm^{-1}). The coatings prepared with a 50% Ar fraction exhibited very high oxidation resistance in air up to 1500°C and extremely high thermal stability in the inert gases up to 1600°C.

AP-6 Observation of Growth Behavior in the Aluminide Mild Steel Using EBSD, ***W.-J. Cheng*** *(d9603505@mail.ntust.edu.tw), C.-J. Wang*, National Taiwan University of Science and Technology, Taiwan

Mild steel was coated by hot-dipping into a molten aluminum bath. The growth behavior in the aluminide layer during diffusion at 750°C in static air was analyzed by Electron Backscatter Diffraction (EBSD). The results showed that the aluminide layer of the as-coated specimen consisted of an outer aluminum topcoat, minor $FeAl_3$, and major Fe_2Al_5, respectively.

Besides, Fe_2Al_5 possessed a tongue-like morphology, which caused the corresponding serration-like morphology of the steel substrate. A portion of the peaks of serration-like substrate were isolated and accompanied by the formation of voids with increasing exposure time at 750°C. The aluminide layer composed of single Fe_2Al_5 phase as the aluminum layer was consumed, leading to the disappearance of $FeAl_3$. After 60 min of exposure, $FeAl_2$ and FeAl phases formed at the interface between Fe_2Al_5 and the steel substrate. With increasing exposure time, the voids condensed and the serration-like morphology disappeared, while $FeAl_2$ and FeAl phases kept growing. After prolonged exposure, the aluminide layer composed of $FeAl_2$ and FeAl.

AP-7 Control of Coating Thickness Ratio for Optimizing Adhesive Strength and Thermal Shock Resistance in Air-Plasma Sprayed Zirconia Based Thermal Barrier Coatings, *Y.G. Jung (jungyg@changwon.ac.kr),* ***S.I. Jung****, Y.S. Sim, J.Y. Kwon, J.H. Lee, U. Paik*, Changwon National University, Korea

The effects of thickness ratio between top and bond coats on adhesive strength and thermal shock resistance have been investigated under thermal fatigue in thermal barrier coating (TBC) systems prepared using an air-plasma spray (APS) process. The thermal fatigue tests were conducted at T = 1,100°C for fully impeded tests into a furnace and at T = 1,210°C for one-side exposed tests, with dwell times of 8 and 24 h. In the as-prepared TBC systems, as the top coat thickness is increased, the adhesive strength is decreased, showing the fracture origin at inside of top coat. Relatively long-term cyclic thermal fatigue of 24 h easily delaminate the top coat rather than that of 8 h, and the tests impeded into the furnace pursue the delamination or fracture of the top coat. The thermal fatigue before delamination in both thermal exposure methods increases the adhesive strength in both cases, while the one-side exposed sample extends the thermal cycling lifetime. E ven though the thickness ratio is same, the thicker top coat shows better thermal shock resistance than thinner one. The optimum thickness ratio between bond and top coats is 1:2 for improving and enhancing the adhesive strength and the thermal shock resistance, respectively. The thermally grown oxide (TGO) layer thickness is not much affected by the top coat thickness, showing a nominal thickness of 8 μm after delamination in both thermal exposure methods. The effects of the thermal fatigue condition on microstructural evolution, mechanical properties, and thermal shock resistance are discussed.

AP-8 Diffusion Aluminide Coatings for TiAl Intermetallic Turbine Blades, ***M. Goral*** *(marek.goral@polsl.pl), L. Swadzba, G.J. Moskal,* The Silesian University of Technology, Poland, *G. Jarczyk*, ALD Vacuum Technologies AG, Germany

Alloys based on the intermetallic phases from Ti-Al system are materials which, on the base of their resistance characteristics, could be widely used in automotive and aerospace applications. The main restriction of the usage of those materials is their still insufficient oxidation resistance above 850°C.

This parameters might be improved using aluminide coatings containing $TiAl_2$and $TiAl_3$phases, which induces the creation of Al_2O_3scale (possessing much better protective attributes) in oxidation process. This type of aluminides can be deposited on the surface of TiAl alloys by different methods such as pack cementation, plasma spraying or magnetron sputtering. This article presents a new method of production of aluminide coatings using out of pack technology. The investigated coating has been produced on turbine blades made from Ti47Al5Nb intermetallic alloy. The surface morphology, structure and phase and chemical composition has been in vestigated using XRD phase analysis, SEM and EPMA. The phase analysis showed that $TiAl_2$is main component of deposited coating. The oxidation resistance of coating has been also investigated. Isothermal oxidation test of TiAl turbine blades was conducted. After 600h test at 950°C oxidation test the spallation of scale from the surface of turbine blades without coating was observed. The scale on the turbine blade with aluminide coatings was very thin and spallation was not presented. The gas phase aluminizing technology can be applied in aerospace industry It is very attractive technology for improving of oxidation resistance of turbine blades.

AP-9 Dry Drilling of Austempered Ductile Iron (ADI) with Different Coated Drill, ***W. Mattes*** *(wilmar@senai-sc.b),* SENAI-SC, ALVES, Brazil, *S. Martins*, SOCIESC, Brazil

Due to economic and environmental reasons, dry drilling research have been developed attending the global marketing necessities. However, long tool life and high quality of holes are not easy obtained in dry conditions. Nowadays, many coatings have been developed in order to aid the machining process. Thus, the aim of this work is to evaluate which coating type can improve the dry drilling performance of ADI. Dry drilling tests were carried out with Austempered Ductile Iron (ADI) with 293 hardness Brinell. Carbide drills with different coatings were used as cut tool. The drilling parameter were cut speed of 120m/min, rate feed of 0.2mm and hole depth of 30mm. Three coatings were tested: AlCr, TiAlN and one combination of TiAlN and AlCrN. Based on these results, the better performance of dry drilling of ADI was achieved when TiAlN+ AlCrN coating was used. So, with adequate coating choose it is possible to improve the dry drilling process.

AP-10 The Oxidation of a Air Plasma Sprayed Thermal Barrier Coating, ***P. Smith*** *(peter.smith@cranfield.ac.uk), R.G. Wellman,* Cranfield University, United Kingdom, *R. Jones, M. Wybrow,* Rolls Royce, United Kingdom, *J.R. Nicholls*, Cranfield University, United Kingdom

Since the development of air plasma sprayed (APS) thermal barrier coatings (TBC) and their application in hot oxidising conditions, oxidation has become a key element in understanding coating degradation and eventual failure. The operating conditions of a TBC can be partly recreated using isothermal and cyclic furnaces to replicate the in-service oxidation of a TBC system. The main aim of this project was to study the changing morphology and growth of the thermally grown oxide (TGO) in an APS TBC system, using scanning electron microscopy and energy dispersive x-ray analysis. To do this it was necessary to obtain samples at different stages of oxidation. The samples of the APS TBC were thermally cycled at 1100°C and taken out at increasing time intervals to be analysed. The TGO thickness was measured to obtain the rate of oxidation and to determine the type of oxides present at various stages. The analysis of the TGO shows the initial growth of an alumina layer during the early stages of thermal cycling, however, after further cycling areas of a mixed oxide appear between the alumina and yttria stabilised zirconia (YSZ). At the point of coating failure the mixed oxide has grown to form a continuous layer. The growth rates of both oxide layers were recorded to observe how the mixed oxide interacts with the alumina layer, where the mixed oxide most frequently occurs, due to the undulating morphology at the bond coat-TGO-YSZ interfaces, and to determine the origin of the mixed oxide.

AP-11 Influence of Deposition Parameters on Structure and Oxidation Resistance of Diffusion Aluminide Coatings Obtained by CVD Method on Ni-Base Superalloys, ***L. Swadzba*** *(lucjan.swadzba@polsl.pl), B. Mendala, M. Hetmanczyk, B. Witala, M. Goral*, The Silesian University of Technology, Poland

Chemical vapour deposition (CVD) method plays meaningful role in depositon aluminide coatings on nickel based superalloys. Thank to this method it is possible to deposit aluminide coatings in cooling channels which is difficult using other methods. In this paper result of development and properties of high-temperature coating deposited on superalloys such as single crystal PWA 1484 and polycrystalline Mar M-247, Inconel 100 will be presented. Result of diffusion aluminizing using high-tech ION BOND equipment and "out of pack" equipment will be showed. There will be shown influence of technological parameters such as: temperature, pressure in retort, chemical composition of reactant gases on microstructure, thickness and phase composition of aluminide coatings. Low activity coating of NiAl type containing 20 – 25% mas. and 30μm thickness was developed. Thickness of layer and content of aluminium in coating on the surface is insufficient to fully protect bas e material. Further processes were conduct with using additional source granuls of chemical composition of Ni, Al, Cr and modified by reactive elements. As a result high activity aluminide coatings type of NiAl were developed. Aluminide coatings were investigated by light microscopy, scanning electron microscopy (SEM), electron probe microanalysis (EPMA) and X-ray diffraction analysis (XRD). Result of 23 hour oxidation resistance test of aluminide coatings will be presented. There will be shown influence of chemical composition 3 different nickel based superalloys on properties of aluminide coatings.

AP-12 Characterization of Microstructure and Properties of Plasma Sprayed Ceramic Coatings on Mg Alloys, ***A. Iwaniak*** *(aleksander.iwaniak@polsl.pl), G.J. Moskal, A. Kiebus, T. Rzychon*, The Silesian University of Technology, Poland

This article will be presents results of microstructural investigation and properties characterization of ceramic cotings deposited on magnesium alloy by plasma spraying. The study has been done on AZ91 type alloy with ceramic top coat on the basis of tungsten carbides and NiCr bond coat. First of investigated area is related to quantitative and qualitative description of ceramic top surface, bond coat and magnesium alloy substrate. Second area of investigations is connected to XRD characterization of deposited coating. The purpose of this research was identification of the residual stress in the top coat. Another subject is related to microstructural characterization of coating from their quality point of view. The parameters which identify the quality of the bond coat and the ceramic coating consist: the thickness of the layers; the quality of the connection between the metal base and the bond coat; the presence of cracks and oxides; porosity; globular grains; bond c oat integrity; the roughness of the bond-coat surface; the microstructure of

the ceramic layer – the analysis involves the porosity assessment, randomly oriented cracking and their shape, metallic impurities and globular grains.

AP-13 High Temperature Wear Behaviour of Aluminum Oxide Coatings Produced by Ac Micro Arc Oxidation, *E. Arslan, Y. Totik,* ***Y. Vangolu*** *(yvangolu@atauni.edu.tr), A. Alsaran, I. Efeoglu*, Ataturk University, Turkey

Aluminum alloys are becoming increasingly important, especially in the automotive and aerospace industries. However, these materials tend to have poor wear resistance at atmospheric and high temperature conditions. Aluminum oxide coatings are potentially very effective in developing hard, wear-resistant surfaces. The aim of this present study was to evaluate the high temperature wear behavior of aluminum oxide coatings at different temperatures by using high temperature pin-on-disc tribotester and alumina balls as counterfaces. The aluminum oxide coatings were produced by AC micro arc oxidation (MAO). The structural analyses of the coatings were performed using XRD and SEM techniques. The hardness was measured using microhardness tester. Keyword: Aluminum alloys, Plasma electrolytic oxidation, High temperature, Wear.

AP-14 Thermal Barrier Coatings by Electron Beam-Hysical Vapor Deposition of Zirconia Co-Doped with Yttria and Niobia, ***D.S. Almeida,*** *C.A.A. Cairo,* Centro Tecnologico de Aeronautica, Brazil, *D.A.P. Reis (danielireis@hotmail.com),* Instituto Tecnologico de Aeronautica, Brazil, *V. Henriques, F. Piorino Neto*, Centro Tecnologico de Aeronautica, Brazil

The most usual ceramic material for coating turbine blades is yttria doped zirconia. Addition of niobia, as a co-dopant in the Y_2O_3-ZrO_2 system, can reduce the thermal conductivity and improve mechanical properties of the coating. The purpose of this work is to evaluate the influence of the addition of niobia on the microstructure and thermal properties of the ceramic coatings. SEM on coatings fractured cross-section shows a columnar structure and the results of XRD show only zirconia tetragonal phase in the ceramic coating for the chemical composition range studied. As the difference $NbO_{2.5}$-$YO_{1.5}$ mol percent increases, the tetragonality increases. A significant reduction of the thermal conductivity, measured by laser flash technique, in the zirconia coating codoped with yttria and niobia when compared with zirconia-yttria coating was observed.

AP-15 Effects of Granulometry on Properties of Plasma Sprayed Yttria-Stabilized Zirconia Coatings, ***S. Liscano*** *(lissug@hotmail.com), L. Gil,* UNEXPO, Venezuela, *M.H. Staia,* Universidad Central de Venezuela, *O. León*, UNEXPO, Venezuela

It is well known that the quality and properties of the plasma-sprayed coatings are strongly dependent on the coating microstructures, and this of in-flight particle characteristics and processing parameters. The aim of this work was to determine the influence of granulometry of feedstock powders on porosity and mechanical properties of plasma sprayed YSZ TBCs. NiAl bond coats were deposited on a number of AISI 310 specimens using HVOF spray technique. Furthermore, these specimens were coated, with ZrO_2–8%Y_2O_3 powders with two different granulometry, using air plasma spray technique (APS). Duplex coatings were then characterized using optical microscope, Scanning Electron Microscopy (SEM) coupled with energy dispersive X-ray analysis (EDS), and X-ray Diffraction (XRD). The fracture toughness of these coatings was evaluated using micro-indentation techniques. The results show that as sprayed microstructure is typically porous, and it was evidenced that t he porosity increased with particle size, and this was associated with an increased of partially melted particles, which was directly related to increase of fracture toughness. Cracks were found to initiate and propagate easily in direction parallel to the coating growth directions than along the splat boundaries. EDS analyses revealed that oxidation of aluminum, in the NiAl alloy, occur in the high-temperature plasma-spray stream during deposition.

AP-16 Effect of Hf Alloy Addition on Aluminide Coating Performance, ***B.A. Pint*** *(pintba@ornl.gov), K.L. More, J.A. Haynes*, Oak Ridge National Laboratory

Additions of Hf were made to a conventional Ni-base chromia-forming alloy and an alumina-forming austenitic alloy to improve their high temperature oxidation resistance after aluminizing. The addition of Hf is intended to improve the adhesion of thermally grown alumina on the coating thereby improving oxidation resistance. Coatings were made by a standard low activity chemical vapor deposition process at 1100°C and produced microstructures typical of NiAl coatings. Oxidation resistance is being assessed using thermal cycling at 1100°-1150°C. Adding Hf as an alloy addition to the substrate is one means of incorporating Hf into a simple aluminide coating and is being used to assess the potential performance benefit of adding Hf by a conventional aluminizing process.

Hard Coatings and Vapor Deposition Technology
Room: Town & Country - Session BP

Symposium B Poster Session

BP-1 Deposition of Superhard Nanolayered CrAlBN Thin Films by Cathodic arc Plasma Deposition, ***S.K. Kim*** *(skim@ulsan.ac.kr), V.V. Le,* University of Ulsan, Korea, *J.W. Lee*, KAIST, Korea

Thin films of CrAlBN were deposited on SKD11 tool steel substrate using Cr and AlB cathodes by a cathodic arc plasma deposition system. The influence of nitrogen pressure, AlB cathode arc current and bias voltage on the mechanical and structural properties of the films were investigated. The hardness of the film decreased with the increase of the nitrogen pressure from 1.3 Pa to 9.3 Pa. The hardness of the films increased slightly as the AlB cathode arc current was raised from 35 A to 45 A. A further increase in the AlB cathode arc current beyond 50 A decreased the film hardness. The film hardness increased from -50 V to -200 V. The film exhibited a maximum hardness of 48 GPa at the bias voltage of -200 V.

BP-2 A Cylindrical Form of the Hot Refractory Anode Vacuum Arc (HRAVA), ***S. Muhl*** *(muhl@servidor.unam.mx),* IIM-UNAM, *I. Camps, O. Peña,* Universidad Nacional Autonoma de Mexico, *E. Camps, L. Escobar-Alarcon,* Instituto Nacional de Investigaciones Nucleares, Mexico, *S.E. Rodil*, Universidad Nacional Autonoma de Mexico

We have designed, built and studied a cylindrical version of the Hot Refractory Anode Vacuum Arc (HRAVA) developed by the group of R.L. Boxman of Tel Aviv University, Israel. In the Tel Aviv version the anode and water-cooled cathode are in the form of two parallel plates separated by a few centimetres. After a few minutes of operation the arc and resultant plasma from the hot refractory anode extends radially outwards in all directions from the cathode-anode axis. In our cylindrical system a water cooled cylindrical aluminium cathode surrounds a central anode rod made of high density graphite mounted on a ceramic base which closes one end of the cylinder. A solenoid surrounds the outside of the cathode cable which produces a variable magnetic field along the axis of cylindrical cathode. In this way when a cathode arc is generate using a trigger pulse the magnetic field moves the cathode spot in a circular path around the inside of the cathode. The close proximity of the anode, ~2 cm, ensures that it rapidly is heated to a sufficiently high temperature that the aluminium deposited by the cathode arc is thermally evaporated and an anodic plasma, produced by the thermal emission of electrons, extends out of the open end of the cylindrical setup. The deposition rate depends on the arc current and the magnetic field. The advantage of the present arrangement is that the beam of evaporated material is directed along the axis of the cylindrical cathode. The macroparticle production is dependent on the magnetic field and the design of the ceramic cap to trap the macroparticles has been optimised. In this study we report the variation of the anode temperature, ion current, deposition rate and macroparticle density as a function of the experimental parameters.

BP-3 TiN/Ti Thin Film on Flexible PET Substrate Deposited by RF Magnetron Sputtering, *J.H. Huang (jhhuang@mx.nthu.edu.tw),* ***J.L. Lin***, *G.P. Yu*, National Tsing Hua University at Hsinchu, Taiwan

The objective of this study was to prepare TiN/Ti thin films on PET substrate, with excellent adhesion, high gas anti-permeability and low sheet resistance. TiN/Ti thin films have been deposited on PET substrate by RF magnetron sputtering. The respective deposition durations of Ti and TiN thin films were chosen as the controlling variable in the deposition process. The gas anti-permeability, sheet resistance, surface adhesion and optical properties of TiN/Ti thin films were systematically investigated. In addition, the effects of Ti interlayer, packing factor and film thickness on these properties were also studied. The crystal structure, chemical composition and packing factor of the TiN/Ti thin films were characterized by glancing incidence X-ray diffraction (GIXRD) and Rutherford backscattering spectrometry (RBS), respectively. Water vapor transmission rates (WVTR) of the TiN/Ti thin films were measured using a MOCON instrument. The electrical resistance was determined using a four-point probe. Experimental results showed that the structure of TiN thin films deposited on both PET and Ti/PET substrates were amorphous. The film adhesion for all TiN/PET and TiN/Ti/PET samples was excellent. The Ti interlayer could effectively increase the packing factor of TiN thin films. The gas permeability of TiN/Ti thin films was related to the synthetic parameter of packing factor × total thickness of the coating. The WVTR of the specimens would level off, if the TiN/Ti thin films reached a critical thickness of about 100 nm in this study. The sheet resistance of the TiN/Ti thin films decreased with increasing film thickness. The packing factor of the TiN thin film increased with the thickness of Ti interlayer and hence decreasing the sheet resistance. The results indicated that adding a Ti interlayer could effectively improve

the gas permeability and sheet resistance of the TiN/Ti thin films on PET substrate. The superior performance of the thin films nearly reached the requirements for the applications in flexible LCDs, inorganic solar cell, and thin film battery.

BP-4 Preparation, Structure and Characteristic Study of TiVCrAlN Thin Film, ***D.-C. Tsai*** *(d9366207@mail.nchu.edu.tw), M.-J. Deng, F.-S. Shieu*, National Chung Hsing University, Taiwan

Al-doped TiVCrN thin films were prepared by dc magnetron cosputtering, in which were utilized for TiVCr and Al targets, respectively. Glancing incidence X-ray diffraction revealed a simple face-centered cubic solid solution phase with a (200) preferred orientation for the Al-doped films, in contrast to the undoped ones possessed (111) and (200) preferred orientation predominantly, indicating that Al addition can lead to important enhancement of the adatom mobility and consequently trend to thermodynamic favorable orientation. The attendance of Al input power also play an important role on orientation and grain size. These, in turn, also cause lattice shrinks and grain growth. However, it is observed by transmission electron microscopy that the microstructure morphology seem to be independent form the Al concentration, implying that barrier existing at the boundary is not enough to be overcome. The variation trend of the surface roughness which well conforms with that of the grain size is observed. With increasing Al concentration, the hardness and resistivity increase. In consideration of the variation trend of mechanical and electrical property of the films, the factor is attributed to a competition of grain growth and bonding characteristic.

BP-5 Interlayer Effect on the Detaching Mechanism for Mo-Ru Hard Coatings, ***Y.-I. Chen*** *(yichen@mail.ntou.edu.tw),* National Taiwan Ocean University, Taiwan*, L.-C. Chang,* Mingchi University of Technology, Taiwan*, B.-N. Tsai*, National Taiwan Ocean University

Hard coatings have been used as a protective coating on the top surface of the glass molding die. Damages such as scratch, peeling, atomization and adherent glass scars were the main injuries occurred on the surface of the protective coating, which degraded the surface quality and limited the lifetime of the molding die assembly. In a realistic mass production process, recycling of the damaged coating was necessary for the cost and time issues. Thus, detaching the damaged coating is an important process and chemical stripping was proposed ever. Etching pits in the protective coating and selectively oxidized interlayer provided feasible paths for the stripping. In this study, Mo-Ru coating was used as the protective hard coating, while Ni, Cr, Ti and Ta were used as the interlayers to evaluate the detaching mechanism by using the etchant, ammonium cerium (IV) nitrate – acetic acid solution. The chemicals solved in the etching solution were investigated by an inductivel y coupled plasma optical emission spectroscopy. It is verified that Ni and Cr with lower ionized valances were easily reacted with the etchant. On the other hand, Ti and Ta interalyers were resistant to the etchant and suitable to protect the substrate.

BP-6 Growth of α-$(Al,Cr)_2O_3$ Thin Films by Reactive r.f. Magnetron Sputtering, ***D. Diechle****, M. Stueber, H. Leiste (Harald.Leiste@imf.fzk.de), S. Ulrich,* Forschungszentrum Karlsruhe, Germany*, V. Schier*, Walter AG, Germany

Advanced thin film materials for cutting tool applications require complex property profiles including high hardness, toughness, wear and corrosion resistance. Such materials are expected for example in the Al-Cr-O system. The materials science approach behind this work is to quench Al-Cr-O thin film materials in a metastable corundum-type solid solution structure directly from the vapor phase at deposition temperatures significantly below the temperature range of the thermodynamic stable phase which exists only above 1300°C in the corresponding Al-Cr-O phase diagram. First, we will describe a combinatorial approach using a segmented target consisting of aluminum and chromium plates for the deposition of Al-Cr-O thin films by reactive r.f. magnetron sputtering. This experimental procedure results in the growth of coatings of different composition and microstructure in dependence of the sample positions in relation to the target. For specific deposition conditions stoichiometric, nanocrystalline solid solution strengthened $(Al_{1-x},Cr_x)_2O_3$ thin films were grown in a corundum-type structure. Secondly, we derive from these experiments fixed individual material compositions and use homogeneous metallic targets with an appropriate Al:Cr composition for reactive r.f. magnetron sputtering. Similar coatings grown by these different approaches will be compared with respect to their constitution, microstructure and properties. The deposition experiments are carried out with a Leybold Z 550 PVD machine in an argon–oxygen plasma. The cathode power is set to 500 W in r.f. mode, and the total gas pressure is 0.65 Pa in all experiments. During deposition the substrate temperature is controlled in the range from 180°C to 600°C. In addition, a substrate bias up to -400 V is induced with a second r.f. power supply. Commercial cemented carbide substrates and silicon wafers are coated. The coatings are characterized by determining their thickness, Vickers micro hardness, residual stress, density, chemical composition, constitution (by XRD) and their microstructure (by REM and TEM).

BP-7 Optoelectronic and Structural Properties of ZnO:Ga Thin Films Prepared by Pulsed DC Magnetron Sputtering, ***W.-T. Yen****, Y.-C. Lin (ielinyc@cc.ncue.edu.tw)*, National Changhua University of Education, Taiwan

The ZnO:Ga (GZO) thin films were prepared on a type Corning 1737 glass substrate by pulsed DC magnetron sputtering. The effect of pulse frequency and film thickness on the thin film structural and optoelectronic properties was investigated. After deposition, the deposition rate and thickness of the thin film were measured and analyzed using an a-step profiler. An X-ray diffraction (XRD, CuKα*,* @lamda@=1.54052Å) analysis was performed to investigate the crystallographic structure of the ZnO:Ga film. The surface morphologies of the films were observed with an atomic force microscope. Field Emission Scanning Electron Microscopywas performed to observe the microstructures of the ZnO:Ga films. The electrical properties were obtained by Hall effect measurements. The optical transmittance of the ZnO:Ga films were measured by UV-visible spectrometer. PL measurements were carried out by the excitation of a He-Cd laser with 325nm wavelengths at room temperature.The experimental resu lts showed that a highly c-axis (002) preferred orientation and lowest resistivity are obtained by a pulsed frequency of 10kHz condition. Following an increase in the thickness of GZO thin film, the grain size of GZO thin film and carrier mobility also increased, resulting in the resistivity of GZO thin film decreased. From the XRD spectra, the full width at half maximum spectra gradually broadened out, which predictably could be because the $Ga3^+$ was being replaced by $Zn2^+$, causing internal defects and generating a deformed lattice, resulting in the signal broaden. The lowest resistivity of 2.01×10-4ω-cm for the GZO thin film is obtained under the pulse frequency of 10kHz and thin film thickness of 500nm, this result is better than that from previous studies. In addition, the Rms of surface roughness is 2.9 nm, the optical transmittance in visible region is 86%, the energy gap of GZO thin film is approximately 3.83eV are obtained.

BP-8 Formation, Characterization and Properties of Al-doped Vanadium Pentoxide Nanorods by Chemical Vapor Deposition, ***M.W. Huang****,* National Chung Hsing University, Taiwan*, Y.C Su, L.W Chang,* National Tsing Hua University, Taiwan*, F.-S. Shieu,* National Chung Hsing University, Taiwan*, H.-C. Shih (ya.chiyi@xuite.net)*, Chinese Culture University, Taiwan

Al-doped vanadium oxide nanorods were synthesized on Si（100） and glass substrates by chemical vapor deposition using solid precursors of vanadium oxide powders（V_2O_5 99.5%, 1.185 g）mixed with aluminum powders（Al 99.5%, 0.015 g）in an Ar atmosphere（$5x10^{-2}$ Torr, 10 sccm）at 700℃ for 1 hour. Structure, morphology, and optical property of the nanorods were characterized by SEM, HRTEM, EDS, XRD and XPS. The diameter of Al-doped V_2O_5 nanorods were about 50 to 100 nm and length of several micrometers with an orthorhombic crystal structure growing along direction [020] with sharp diffraction peaks at (010) and (020). XPS patterns showed three conspicuous binding energy peaks of (V) $2p_{3/2}$ at 518 eV, (V) $2p_{1/2}$ at 525 eV, (O) 1s at 530 eV and (Al) 2p at 71 eV. The V $2p_{3/2}$ binding energy centered at 518 eV with an FWHM of 1.2 eV is characteristics of vanadium in the +5 oxidation state which is well consistent with the value of V_2O_5 structure. The electrical properties of a single Al-doped V_2O_5 nanorod was conducted in-situ and show that the current and conductivity both decrease obviously, because V_2O_5 is an n-type semiconductor and Al is a ρ-type doped element, the holes would counteract with electrons which led to the decrease of carrier mobilities.

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BP-9 Characterization of Superlattice CrN/AlN Coating for Semiconductor Packaging Applications, *D.-Y. Wang (dywang@mdu.edu.tw),* ***W.-H. Tzeng***, Mingdao University, Taiwan

The CrN coating deposited by PVD technique has been adopted as the replacement for the conventional hard chrome plating to protect the semiconductor packaging dies for its environment-friendly process and superior tribological properties. To further extend the service life of the packaging dies against the ever-increasing abrasiveness, stickiness, and corrosiveness of the packaging resin, a CrN/AlN superlattice coating was synthesized by using the unbalanced magnetron sputtering technique. This study demonstrated the obvious dependence of the superlattice characteristics with the periodic double-layer thickness, optimized at 4 nm. The XRD analysis revealed the meta-stable cubic structure of AlN layer within the CrN/AlN superlattice matrix, which possesses a B1-NaCl crystal

structure. The typical hexagonal wurtzite crystal structure of the bulk AlN was suppressed by aligning to the cubic CrN lattice along the superlattice boundaries. The microhardness of the superlattice CrN/AlN coating was measured at 28.1 GPa, significantly higher than the monolithic CrN coating. At the periodic thickness of 4 nm, the CrN/AlN superlattice coating exhibited very stable oxidation behavior with a relative weight increase of only 0.2-0.25% after exposure to a 1000-°C TGA test. Results showed that the CrN/AlN superlattice coating is capable of withstanding the abrasive high-temperature service environments up to 800°C without much degradation. Finally, the contact angle measurement revealed the improved hydrophobic property of the CrN/AlN superlattice coating, which allows easy mold- releasing during the prolonged packing operation.

BP-10 Effects of Dopant Ion and Mn Valence State in the $La_{1-x}A_xMnO_3$ (A=Sr, Ba, Ca) CMR Films for Infrared Sensor, ***S.G. Choi**, A.S. Reddy,* Yonsei University, Korea*, H. Ryu, B.-G. Yu,* Electronics and Telecommunication Research Institute, Korea*, H.-H. Park (hhpark@yonsei.ac.kr)*, Yonsei University, Korea

$La_{1-x}A_xMnO_3$ (A are divalent alkaline earth ions such as Ca, Sr, and Ba) colossal magnetoresistance (CMR) films have been received much attention for infrared sensor resistor because they have a high temperature coefficient of resistance (TCR) and a low noise property which are the most important figure of merit to infrared sensor, for example, microbolometer. Electron transport properties in the CMR oxides with perovskite structure are known to depend on the ionic size and oxidation state of the element at the rare-earth elemental site, which influences the bond length and bond character of Mn-O and oxidation state ratio between Mn^{3+} and Mn^{4+}. To understand the relation between structural and electrical properties of Mn-based oxides films, we investigated CMR films by varying the dopant ions and the ratio of the oxidation state of Mn ion.

In this research, the effects of various dopants such as Ca, Sr, and Ba ions and the ox idation state of Mn ion on the structural and electrical properties of CMR films were investigated. For these purposes, $La_{1-x}A_xMnO_3$ films with same tolerance factor and then no internal lattice strain difference in the film were prepared. Also CMR films which have different oxidation state of Mn^{3+} and Mn^{4+} were also prepared by varying the amount of divalent A ions while maintaining the tolerance factor. In this way, a correlation between dopant and the oxidation state ratio of Mn^{3+} and Mn^{4+} in the CMR thin films which affects crystal structure, TCR value and 1/f noise could be systematically investigated.

BP-11 Composition-Constitution-Morphology Relationship of Al_2O_3 Thin Films Deposited by Plasma Assisted Chemical Vapor Deposition, ***J.M. Schneider** (schneider@mch.rwth-aachen.de), K. Jiang, K. Sarakinos, D. Music, J. Mayer,* RWTH Aachen University, Germany*, R. Snyders,* Université de Mons, Belgium*, St. Konstantinidis,* University of Mons-Hainaut, Belgium*, T. Markus*, Forschungszentrum Jülich, Germany

We have studied the correlation between the chemical composition, constitution and morphology of Al_2O_3 using experimental and theoretical means. Plasma-assisted chemical vapor deposition, with an $AlCl_3$-Ar-H_2-Cl_2 precursor mixture, was used to synthesize γ- and α-alumina thin films. These coatings contain from ~1.0 to ~2.0 at.% of residual Cl depending on the constitution, where larger Cl contents are found in γ-alumina. Plasma chemistry, studied by optical emission spectroscopy, suggests that Cl is incorporated in these coatings as a result of incomplete dissociation of $AlCl_3$ precursor. Combining scanning electron microscopy, transmission electron microscopy (TEM), electron dispersive X-ray analysis (EDX) and ab initio calculations, we have studied the formation of the pores found in these films. Pore populations with diameter of about a few tens of nm and in the μm range can be observed in the alumina matrix. Chlorine desorption measurements show chlorine release from γ- and α-alumina films as the temperature is increased above the growth temperature and a second onset temperature at about 1000°C. Based on ab initio calculations, we have established that Cl can be incorporated in both γ- and α-alumina. The Cl content may be larger in γ-alumina, because there are more possible incorporation sites as compared to those in α-alumina. Furthermore two Cl atoms are likely to agglomerate since the total energy is reduced compared to an un-agglomerated configuration. We propose that Cl agglomeration is the first step towards Cl_2 molecule formation and subsequent precipitation of Cl to form bubbles. It can be learned that the Cl_2 incorporation has to be minimized during growth of dense alumina coatings.

BP-12 Characterization of Binary, Ternary and Quaternary Hard Coatings in the Material System V-Al-C-N Produced by Industrial Scale Reactive Magnetron Sputter Deposition, ***Sz. Kolozsvári** (kolozsvari@tzo-gmbh.de), P. Pesch,* TZO Rheinbreitbach GmbH, Germany*, C. Ziebert, M. Stueber, S. Ulrich*, Forschungszentrum Karlsruhe, Germany

Binary, ternary and quaternary hard coatings in the system V-Al-C-N (VN, AlN, VC, VAlN and VAlCN) were deposited by industrial-size/industrial scale reactive d.c. and r.f.-magnetron sputtering in an Ar/N and an Ar/CH_4 plasma from a V target and/or Al target, respectively from an VAl_{20}-target (V target with 20 Al-plugs). For each experiment, Si (100) substrates and polished 1.2379 steel substrates were placed on the rotating substrate table with variable rotation speed and rotation axes. VN coatings were deposited at a constant Ar gas flow of 250mln, while the bias voltage applied to the substrate table was systematically varied between -80 and -200V. VC coatings were deposited at constant Ar:CH_4 gas flow ratio (250:60) and systematically varied bias voltage between -80 and -200V and respectively at constant bias voltage from -170V and varied Ar:CH_4 ratio through changing the CH_4 amount in the plasma. For the deposition of AlN, VAlN and VAlC N coatings the bias voltage, the Ar:N ratio and the total gas pressure were varied. The chemical composition of the obtained coatings was determined by electron microprobe analysis and the crystal structure of the films was characterized by X-ray diffraction. The influence of the process parameter variation on the mechanical properties hardness, reduced elastic modulus and critical load of failure have been studied by microindentation and scratch test. The surface roughness of the as-deposited samples was examined as a function of the initial substrate surface roughness. It was possible to achieve a large variation in the hardness in the range from $700HV_{0.005}$ to $3100HV_{0.005}$ and in the coefficient of friction in the range from 0.2 to 0.7. Finally, the specific conditions for the formation of large-area VN, AIN, VC, VAlN and VAlCN nanoscale coatings in industrial-size coating facilities are described in terms of process engineering.

BP-13 Ultraviolet Photodetector and Gas Sensor Properties of Sb doped SnO_2 Nanowires, ***C.-H. Kuo**, J.-M. Wu (jmwu@fcu.edu.tw)*, Feng-Cha University, Taiwan

The Influence of the Sb (antimony) dopant in SnO_2 nanowires has been addressed to characterize the ultra-violet light photodetector and gas sensors activities. The electrical characterization showed that as-synthesized sensors devices exhibited ohmic contacts. The different quantitative ratio of mixed Sb:Sn powder was prepared to synthesize the Sb doped SnO_2 nanowires by thermal evaporation at the temperature of 900°C. The UV-exposed current to dark current has been investigated. It appeared that the UV on and off ratio was shown to depend on the concentration of Sb dopant. The gas sensor activities of SnO_2:Sb naowires were estimated by introducing the variation concentration of ethanol such as 10 ppm, 100 ppm, 1000 ppm. The results demonstrate the potential of fabricating sensors (nanosensors) exhibited a high sensitivity with quick response and recovery times in room temperature.

BP-14 Influence of Bias Voltage on the Microstructure and Physical Properties of Magnetron Sputtered ZrSiN Nanocomposite Thin Films, *N. Cusnir, **D. Oezer**, C.S. Sandu, R. Sanjines (rosendo.sanjines@epfl.ch), A. Karimi,* EPFL, Switzerland*, J. Patscheider*, EMPA, Switzerland

We report an investigation concerning the influence of ion bombardment on the morphology and physical properties of Zr-Si-N nanocomposite thin films. The films were deposited by reactive magnetron co-sputtering from individual Zr and Si targets. The Si content of films was varied by changing the power applied to the Si target. The increase of ion bombardment energy was obtained by applying a negative potential U_b to the substrate. The evolution of the crystalline structure, grain size and lattice constant was mapped out using grazing incidence X-ray diffraction measurements. The optical properties were studied by spectroscopic ellipsometry, the electrical resistivity was measured by Van der Pauw method between 20K and 300K and the hardness was investigated by nanoindentation. Films deposited at a substrate temperature of T_s= 510 K with U_b = 0 V exhibit (111) preferential orientation. The maximum nanohardness of 30 GPa is reached at 4 at.% Si concentrat ion, these films exhibit a mean crystallite size of about 14 nm and a SiN_x coverage on ZrN grain boundaries of about 0.5 monolayer in thickness. In contrast, films deposited at T_s= 510 K with a bias voltage of U_b= -150 V exhibit less pronounced columnar structure with small crystallites having many crystallographic orientations. The maximum nanohardness of 38 GPa observed in these films is reached at about 1.2 at.% Si while the average grain size is 10 nm and the SiN_x surface coverage is 0.35. These results are compared with our prevue results on ZrSiN films deposited at various substrate temperatures and discussed in terms of atomic diffusion and phase segregation effects due to the ion bombardment.

BP-15 Wear Resistance of PVD Magnetron Sputtered ZrTiBN Thin Films, ***O. Jimenez*** *(o.jimenez@sheffield.ac.uk), M. Audronis, K. Kanakis, A. Leyland, A. Matthews*, University of Sheffield, United Kingdom

It is well known that hard thin films containing transition-metal nitride or boride phases (e.g. ZrN, $ZrB_2$and TiB_2) offer outstanding properties such as high hardness, high melting point and chemical inertness. ZrN and (Zr,Ti)N coatings have already been shown to exhibit good tribological behaviour and are considered as very promising candidates for applications related to improved protection against wear (among many others). In this work thin PVD ZrTi(BN) coatings were successfully deposited onto mirror-polished ASP23 tool steel, using medium frequency (20-350 kHz) asymmetric bipolar pulsed magnetron sputtering (PMS) technology. Coatings were obtained using a rectangular target composed of three pieces ($Zr/TiB_2/Zr$) under different conditions of bias voltage and N_2content-the latter being used as a reactive gas at different flow rates, to promote the formation of hard Zr nitride-containing phases. The phase analysis, morphology and mechanical property evaluation of the coatings were studied by means of glancing angle X-ray diffraction (GA-XRD), cross-sectional scanning electron microscopy (SEM) images and nanoindentation, respectively. The tribological tests were carried out by using a UMT multi-specimen test system, at a low reciprocating-sliding frequency of 5Hz (with humidity and temperature continuously monitored and recorded), with the resulting tribological behaviour and wear mechanisms being analyzed by surface profilometry and by SEM evaluation of the worn surfaces. Coatings were tested at an applied normal load of 1N and over sliding distances of 1000 m and 1500 m against a 4 mm diameter polycrystalline high-purity alumina ball counterface. SEM images revealed that the coatings are fully dense, featureless and defect-free. Phase identification performed by GA-XRD revealed a partially amorphous structure, with (Zr,Ti)N and Zr metal being the main constituents. Hardness values above 20 GPa were measured fo r most of the coatings deposited at different N_2flow rates, while hardness for those containing no nitrogen was found to be significantly lower-due to the absence of hard ceramic phases. Wear resistance was found generally to be good; most of the samples survived 1000 m of sliding distance, exhibiting a moderately low friction coefficient of ~0.4

BP-16 Synergy Between High Temperature and Wear In CrSiN Nanocomposite Coatings Deposited by Hybrid Arc /Magnetron Process, *A. Mège-Revil,* ***P. Steyer*** *(philippe.steyer@insa-lyon.fr),* INSA de Lyon, France*, J. Fontaine, M. Guibert,* Ecole Centrale de Lyon, France*, J.-F. Pierson,* Ecole des Mines de Nancy, France*, C. Esnouf*, INSA de Lyon, France

CrN, Cr2N, CrN+Si (6 at.%) and Cr2N+Si (3 at.%) thin films were deposited on M2 steel by an hybrid process in which Cr was deposited from an arc-evaporated target while Si was magnetron sputtered. XRD and HRTEM showed that the nanocomposite structure was obtained only in the case of CrN+Si. The grains of Cr2N are shrunk by adding Si. Consequently, a large amorphisation of the intergranular regions is observed, preventing the formation of a barrier layer of silicon nitride. As a result, nanohardness measurements confirmed that adding Si in CrN improved the mechanical properties, a phenomenon which is not observed in the case of Cr2N and Cr2N+Si. Isothermal and dynamic oxidation were followed by TGA. The oxidation resistance of CrN was found to be greatly enhanced by the nanocomposite structure. Consequently, no such improvement was observed between Cr2N and Cr2N+Si. In aggressive tribo-oxidative conditions, Cr2N-based coatings were too brittle to sustain the shortest tes t. On the contrary, a coated ball of CrN withstood up to 20 meters of alternated rubbing on mirror-polished steel. Finally, the CrN+Si coated ball once again proved its multifunctionality with a Specific Wear Energy three times more important than that of CrN at room temperature and twice at 150 and 300°C.

BP-17 Mechanical and Electrochemical Properties of CrZr-Si-N Coatings, ***Y.S. Kim****, G.S. Kim, S.Y. Lee (sylee@kau.ac.kr), S.C. Oh*, Korea Aerospace University, Korea

For many years, chromium nitride films have been extensively used as a protective hard coating in the various mechanical industries due to their good wear resistance, high thermal stability as well as good corrosion resistance. However, in spite of their excellent properties, the CrN films show inadequate properties for some applications as high speed machining, or at high temperature or in severely corrosive conditions because of the limitations of binary system. Recently, in order to improve the mechanical and chemical properties of the CrN, the Cr-based ternary nitride coatings such as Cr-W-N, Cr-Al-N, Cr-Si-N and Cr-Zr-N have been developed and their excellent properties are reported in many papers. However, investigations on the synthesis of Cr-based quaternary nitride coatings are very limited. In this study, quaternary CrZr-Si-N films with various Si contents ([1] at.%) were synthesized by closed field unbalanced magnetron sputtering with vertical magnet ron sources and their crystalline structure, morphology, mechanical and electrochemical properties a function of Si content were investigated. Preliminary results showed that the characteristics of the CrZr-Si-N films such as hardness, surface morphology, friction coefficient and cross-section structure were very similar irrespective of the Si content ([2] at.%), i.e. all films have high hardness of 32~33 GPa, very smooth surface roughness of 1.1~0.7 nm (Rms value), very low average friction coefficient of 0.22~0.23 and dense microstructure. Detailed experimental results included electrochemical properties will be presented. [1] [1] ≥6.4[2] ≥6.4.

[2]**BP-18 Phonon Anomalies in Multiferroic $BiFeO_3$ Epitaxial Thin Films Prepared by Using Pulsed Laser Deposition**, *M.K. Singh (mksingh100@yahoo.com), S. Dussan,* ***G.L. Sharma****, R. Katiyar*, University of Puerto Rico

Ferroelectromagnetic materials, also known as multiferroics, exhibit ferroelectric (or antiferroelectric) properties in combination with ferromagnetic (or antiferromagnetic) properties. $BiFeO_3$ (BFO) is known to be only is known to be the only material that exhibits multiferroism at room temperature. BFO thin films were grown on (111) STO substrates by employing pulsed laser deposition (PLD) method. The average thickness of these films, as estimated using field-emission scanning electron microscopy, was 300±3 nm. To examine the structure of the PLD-grown BFO film on a STO (111) substrate, θ-2θ x-ray diffraction (XRD) was carried out. The pattern reveals purely $[111]_c$-oriented rhombohedral BFO reflections. Raman spectrum of (111) oriented $BiFeO_3$ (BFO) thin film was studied in temperature range between 27 ^{0}C – 1000 ^{0}C. Observed Raman modes at 136, 162, and 212 367, 550 cm^{-1} show anomalous changes in frequencies, line w idth and integrated intensity between span of temperature range 250- 400 ^{0} C i.e. around magnetic phase transition (T_N). The sign and magnitude of such anomalous behavior appears to be an experimental evidence of perturbation antiferromagnetic ordering coupled with octahedral tilting reveals strong spin-phonon coupling around T_N. The soft mode behavior of A_1 Raman modes at 136 and 162 cm^{-1} was observed directly and reveals a decrease of the Curie temperature (T_C) of the strained film, which was originally suggested by M. S. Kartavtseva and co workers [Thin Solid Films, 515 (2007) 6416].

BP-19 Characteristics of Silicon-Nitride Films Deposited by Internal Linear Inductively Coupled Plasma Source, ***G.H. Gweon****, K.N. Kim, J.H. Lim, G.Y. Yeom (gyyeom@skku.edu)*, SungKyunKwan University, Korea

Silicon-nitride film have been widely used in a various of important applications from semiconductor possibly to flat panel display, such as passivation layers for diverse microelectronics, a gate dielectric material for thin film transistor (TFT), and as anti-reflection (AR) coating for solar cell. Especially, due to their chemical inertness, excellent dielectric properties, and thermal stability, many researchers have been studied to develop high quality silicon-nitride films using a various type of plasma sources. In this study, to obtain high quality silicon-nitride films, internal linear inductively coupled plasma source was used. The internal linear antenna (U-type) was made of 10 mm diameter copper tubing covered by quartz tubing of 25 mm diameter and antenna was connected to the power supply while the other end was connected to the ground. In addition to, we carried out the deposition of silicon nitride thin films by using internal linear ICP source and investig ated the effect of the ratio of NH_3 to SiH_4 on the properties of thin film, such as deposition rate, optical properties, relative composition, and surface morphology. The gas mixture of $SiH_4/NH_3/Ar$ was fed to the chamber where SiH_4 and NH_3 gases were used as the reaction gases and Ar gas as the ignition gas. Also, the compositions and binding states of the films were measured using X-ray photoelectron spectroscopy (XPS; VG Microtech Inc., ESCA2000). The binding states were also measured by a Fourier transform infrared spectrometer (FTIR; Bruker, IFS-66/S). The surface morphology of thin films was measured by a field emission scanning electron microscope (FE-SEM; Hitachi S-4700).

BP-21 Boron-10 Coating of Textured Semiconductor Surfaces for Neutron Detector Integrated Circuits[1], ***C.C. Klepper*** *(kleppercc@ieee.org), O.R. Monteiro, R.C. Hazelton, J.J. Moschella, J.M. Williams, E.P. Carlson, M.D. Keitz*, HY-Tech Research Corporation

A new coating process is emerging for infill of high aspect-ratio vias and trenches in semiconductor substrates with dense, amorphous boron in the production of solid-state neutron detectors. The technique uses a heated-cathode, filtered vacuum arc source of boron plasma. Electrical biasing of the substrate is pulsed, with pulsed shaping and duty cycles programmed to control the infill process, while limiting coating of the ridges between the trenches. The full ionization intrinsically produced by the source allows for precise control of the energies of the boron ions by means of this bias programming, in which the voltage is varied periodically in time to alternate coating deposition at low ion energies and sputtering of already coated material by energetic ions. Boron infill of ~2 μm wide, ~3:1 aspect ratio trenches in Si has been achieved. Application to higher aspect ratios looks promising. The process will work for C, B or similar light infill materials,

but it is of particular interest for isotopically pure ^{10}B, known for its exceptionally high cross-section for nuclear reaction with thermal neutrons. Unlike the case of metals, such as Cu, these light materials require substantial bias voltage levels, well in excess of 500V, to achieve the needed self-sputtering during the sputtering phase of the bias cycle. This is one of the areas where this technique substantially departs for the earlier developed copper infill technique.[2]This and other unique aspects of filtered boron vacuum cathodic arc operation will also be discussed.

[1]Work supported by the Domestic Nuclear Detection Office under contract No. HSHQDC-07-C-00121 (Phase I SBIR)

[2] O.R. Monteiro, JVST-B 17, no. 3 (1999).

BP-22 Preparation and Mechanical Properties of Reactively Sputtered Complex $Al_xCoCrCuFeNi$ Oxide Films, *T.K. Chen, C.S. Lin,* ***M.S. Wong*** *(mswong@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

Complex oxide films of multi-element $Al_xCoCrCuFeNi$ (the molar ratio of x =0-2) were prepared by reactive sputtering of homogeneous alloy targets and co-sputtering of an alloy target and an Al target. . The as-deposited films are nanocomposite in nature and comprise single cubic-(c-) spinel crystalline phase. The film hardness increased with aluminum content reaching a maximum hardness of 22.6 Gpa for $Al_2CoCrCuFeNi$ oxide film. After annealing at 500°C, the hardness enhanced to 26 Gpa. These hardness values are among the hardest available hard oxide materials. Post-annealing at higher temperature dramatically decrease film hardness mainly due to the precipitation of softer second phase and the cracks induced by the precipitates. Further enhancement in film hardness are attempted by adding more Al into the film and by applying substrate bias during film deposition.

BP-23 Nucleation and Growth of Diamond by Pulsed Liquid Injection CVD Using Tequila as Precursor, ***L.M. Apatiga*** *(apatiga@servidor.unam.mx),* Campus Juriquilla, Mexico, *J. Morales*, Universidad Autonoma de Nuevo Leon, Mexico

Tequila, identified as silver tequila 100 % agave (pure tequila), was used as carbon source in the synthesis of diamond. By using a Pulsed Liquid Injection Chemical Vapor Deposition reactor, diamonds of 200-400 nm in diameter were deposited at 850°C on silicon substrates. According to Fourier Transform Infrared Spectroscopy studies tequila is formed basically by ethanol and water, as well as certain chemicals, among them higher alcohols, fatty acids, esters, etc. produced by the fermentation and distillation processes, during its elaboration. Tequila has the right concentration of carbon, hydrogen and oxygen atoms to form CVD diamond, its composition falls into the diamond growth zone, inside the triangle of the ternary C-H-O gas phase diagram. The deposited diamonds were characterized by Scanning Electron Microscopy and Raman spectroscopy.

BP-24 Ion Energy Distribution and Langmuir Probe Studies in an Ar/O_2 Discharge During Al_2O_3 Deposition, ***S.L. Mensah*** *(smensah@uark.edu),* University of Arkansas, *H.H. Abu-Safe,* Lebanese American University, Lebanon, *M.H. Gordon*, University of Arkansas

Local plasma parameters and ion energy distributions for plasmas used to deposit low-temperature alpha-Alumina without a chrome template layer were investigated as a function of power, pressure, and oxygen partial pressure in the vicinity of the substrate using a Langmuir probe and energy-resolved mass spectroscopy. The thin films were deposited by a mid-frequency AC reactive magnetron sputtering system. In this work, power ranged from 4 to 8 kW, at pressures 2, 5 and 8mTorr with oxygen partial pressure ranging from 20-80%. Monitored species include Ar@super+@, O@super+@, O@sub2@@super+@, and Al@super+@. @The EQP results show that, at constant total pressure, changes in the ratio of the partial pressures of Ar/O@sub2@have a negligible effect on the energy distributions of the species, but we noticed an effect on the energy distribution of the species when the total pressure is changed. Similarly, changes in pressure influence the characteristics of the alumina films de posited. Analytical characterization of the thin film shows a correlation between the changes in pressure and phase and morphology of the films. Alpha-alumina phase is predominant when low total pressures are coupled with high oxygen partial pressure and high power. Here we correlate the plasma properties to the differences noted in the deposited thin films to explain why alpha alumina films are obtained.

BP-25 Characterization and Mechanical Properties of Magnetron Sputtered Iron Nitride Coatings, *H.-R. Stock (stock@iwt-bremen.de),* ***I. Eisbrecher***, *H. Teuber*, Stiftung Institut fuer Werkstofftechnik, Germany

Iron nitrides produced by thermochemical procedures like gas or plasma nitriding have interesting properties. For example iron nitride surfaces are machinable with single crystal diamond tools, whereas cutting of pure α-iron surfaces cause severe wear of the diamond tool. We used a d.c. magnetron sputtering unit (CemeCon, CC800/9) with an austenitic manganese steel target (X120Mn12, AISI A128 grade A). Argon and nitrogen as reactive gas were added by mass flow controllers. As substrates polished steel samples were used. With a target power of 3000 W layers of 7 µm thickness could be deposited within two hours. X-ray diffraction reveals the existence of the γ'-phase Fe_4N. To achieve good adhesion a titanium interlayer was deposited prior to the iron nitride layer by means of a titanium target. The coatings were optimized in respect to hardness and adhesion and characterized regarding to chemical composition, morphology, microhardness and topogra phy by electron microscopy, ultra microhardness testing, atomic force microscopy and glow discharge optical emission spectrometry, respectively.

BP-26 Multilayer Chromium Based Coatings Grown by Direct Liquid Injection CVD, ***F. Maury*** *(francis.maury@ensiacet.fr),* ENSIACET, France, *A. Douard, S. Delclos, D. Samelor, C Tendero*, CIRIMAT, France

There is a great interest for multilayer hard coatings because they exhibit enhanced properties resulting from their nanostructuration. Such coatings are generally constituted of carbide and nitride and they are deposited under very low pressure by plasma and PVD processes. These vacuum techniques permit the growth of heterostructures with nanometric thick individual layers and sharp interfaces, which are two requirements for advanced performances. However, both to develop more economical processes and with the goal of continuous deposition applications, the CVD processes operating under atmospheric pressure or near atmospheric pressure are particularly attractive. In this paper we show that the combination of pulsed direct liquid injection and the use of metalorganic precursor (DLI-MOCVD) is a promising route for the growth of nanostructured multilayer coatings. Chromium metal as well as chromium carbide and nitrides single coatings have been deposited at 500°C by thes e processes using liquid solution of bis(benzene)chromium as Cr molecular precursor. Then, CrC_x/CrN and Cr/CrN nanostructured multilayer coatings with a bilayer period as low as 25 nm have been grown. Structural characterizations and preliminary mechanical properties of these metallurgical coatings are presented and discussed.

BP-27 Characteristics and Homogeneity of Direct and Pulse Current Electrodeposited High Phosphorus Content Ni-P Coatings, ***J.C. Wu***, *H.S. Huang, K.J. Chang, F.B. Wu (fbwu@nuu.edu.tw)*, National United University, Taiwan

High phosphorus content Ni-P binary alloy coatings were prepared by direct current (DC) and pulse current (PC) electroplating techniques using Brenner type solution. The characteristics and homogeneity, including microstructure, phase, composition, deposition rate, and thickness uniformity, with respect to plating parameters for both coatings were evaluated for comparison. A high averaged P content of 24at.% could be achieved in both DC and PC Ni-P coatings deposited under current densities ranged from 50 to 125 mA/cm^2. Using a high current density of 100 mA/cm^2 for DC plating, the deposition rate was raised significantly, yet the elemental distribution of P decreased drastically down to 12at.% in the vicinity of substrate edge. Through the modification of the substrate edge geometry and decrease of current density, a high P content Ni-P coating with excellent coverage could be retained. On the contrary, the coating deposited by PC plating showed a super ior thickness uniformity and deposition rate. The laminar structure and severe pinhole distribution in the Ni-P coating edge due to hydrogen reduction of DC plating could be improved by the pulsating modulation. In addition, the plating mechanisms for cathodic reactions of both DC and PC plating techniques were intensively discussed.

BP-28 Effect of Pulse Plating with Ultrasonic Agitation on the Corrosion and Wear Properties of Eco-friendly Trivalent Chromium Layers Prepared in Chromium Sulfate Bath, *S.C. Kwon (sckown@kims.re.kr),* Korea Institute of Materials Science, Korea, ***J.J. Lee***, *Y. Choi*, Sunmoon University, Korea

Eco-friendly trivalent chromium layers was prepared in a modified chromium sulfate bath by pulse plating with ultrasonic agitation to replace hexavalent hard chromium coating in industrial fields. Deposition rate of the trivalent chromium layers by ultrasonic pulse plating was about 0.021mm per hour, which was faster in a direct current (D.C) plating than pulse and ultrasonic pulse plating at a same total on-time. Micro-hardness of the ultrasonic pulse plating are about 856 Hv , which is lower than those of pulse plating (864 Hv) and direct current plating (915 Hv). Wear resistance of the trivalent chromium layer prepared by ultrasonic pulse plating is higher than that of the chromium layer prepared by a direct current plating. The Ultrasonic agitation during pulse plating resulted in increasing neutral salt fog spray life and wear resistance, that is related to smaller crack size and more broad size distribution of the trivalent chromium layers.

BP-29 BGA Cutter Improvement Utilizing Nano-TiAlN Coating Layers Synthesized by Cathodic Arc Ion Plating Process, ***S.H. Huang***, National Chiao Tung University, Taiwan, *T.-E. Hsieh (tehsieh@mail.nctu.edu.tw),* National Chiao Tung Univesity, Taiwan, *C.-W. Chen*, Gigastorage Corporation, Taiwan

Various Ti/Al concentration ratio targets were used in a filtered cathodic arc ion plating system (FCAIP) to deposit the multiple TiAlN layers on silicon wafer substrate and WC cement BGA cutters at various modulation wavelengths(@LAMDA@). Transmission Electron Microscopy (TEM), grazing incidence x-ray diffraction (GIXRD), engery dispersive x-ray spectrometer (EDS), nanoindentation and CNC BGA router were used to evaluate the characteristics of TiAlN layers and performance of BGA cutters. TEM analysis revealed the modulation wavelengths of nanolayers were less than 10 nm at various rotation speed in FCAIP chamber. TEM and GIXRD analyses showed the crystal structure of TiAlN layer with the maximum hardness was NaCl(B1) structure using the target with the composition Ti@0.5Al@0.5. The maximum hardness of multiple TiAlN layer measured by nanoindentation was 43 GPa, while the TiAlN monolayer exhibited the maximum hardness 30 GPa in the same chamber. The TiAlN-coated BGA cut ter deposited at the optimized condition exhibited the twice longer life or higher machining speed in comparison with the ordinary one.

BP-30 Mechanical Properties and Oxidation Resistance of (Cr,Al)N Based Films Synthesized by Radio-Frequency Magnetron Sputtering Method, ***H. Hasegawa*** *(hasegawa@mech.okayama-u.ac.jp), T. Miyake,* Okayama University, Japan, *S. Kunitsugu,* Industrial Technology Center of Okayama Prefecture, Japan, *K. Ohashi, S. Tsukamoto*, Okayama University, Japan

Metastable nitride have been widely used in applications such as cutting tools, protection wear and machinery components, because of their superior tribological, chemical and physical properties. (Cr,Al)N has gained much attention as a substitute for (Ti,Al)N, and has been investigated with respect to, microstructure, mechanical properties, thermal stability, and cutting performance. Recently, we developed the newly (Cr,Al)N based films by adding foreign metal such as Y and Si, which are expected to lead formation of stable oxide and amorphous at elevated higher temperature.

In this study, (Cr,Al,Y)N and (Cr,Al,Si)N were synthesized by radio-frequency magnetron sputtering method with differing N_2/Ar ratios. Microstructure and surface morphology is characterized by X-ray diffraction method, scanning probe microscopy and electron microscopy. Oxidation resistance was evaluated by thermo gravimetric analyzer with measurement of weight gain in dry air enviro nments. In addition, abrasive wear tests were conducted using a Calotest type apparatus. A rotating steel ball was pushed against the sample surface, and the wear volume was measured by a surface profiler.

BP-31 Tribological Characterisation of TiN_x Coatings Synthesized by Cathodic ARC Evaporation Technique, ***J. Menghani*** *(jvm@med.svnit.ac.in), K.B. Pai, M.K. Totlani,* SVNIT Surat, India, *N. Jalgoankar*, Multi-Arc India Ltd., India

Hard Ceramic coatings play continuously increasing grows in field of tribology as well as decorative applications. Stoichiometric titanium nitride (TiN) is actually one of the most important technological coating materials, not only because of its excellent tribological properties, but also due to its good chemical stability. It is certainly, in tribological terms, the most explored PVD hard thin film material and most extensively used in industry. It is used in a wide range of applications, which vary from protective material for machine parts and cutting tools to diffusion barriers in semiconductor technology]. In the past, properties of substoichiometric titanium nitride (TiN_x) have been studied by comparably few researchers. In the present investigation TiNx thin films of varying thickness (1.5 µm,2.0 µm ,2.5 µm , 3.0µm and 4.0 µm) is deposited by Cathode arc evaporation technique. The compositional characterization was carried out by using Philips X-pert pro XRD, Wear testing was carried out using Pin on disc winducom testing machine, normal load applied was 5N,speed 500rpm and for 20 min. Microstructural analysis before and after wear testing was carried out using SEM(Hitachi 3400S).The effect of thickness on wear properties is discussed.

BP-32 NiTi Memory Alloy Sculptured Thin Film by Glancing Angle Electron Beam Evaporation Technique, ***K. Kazmanli*** *(kursat@itu.edu.tr), L. Trabzon, M. Urgen, G. Gurluk*, Istanbul Technical University, Turkey

In this study NiTi alloy sculptured thin films have been studied. The silicon wafer substrates were coated with NiTi alloy by electron beam deposition system. Because as-coated samples showed low crystal-formation, DSC investigation was conducted to find the crystal formation temperature of NiTi thin film. Crystal structure of the coatings was determined by means of X Ray Diffraction investigation. In order to produce films with different morphology (such as inclined, spiral and zig-zag column shapes), vapor flux angles and substrate rotation speed were changed systematically. All coatings were heat treated at the temperature determined from DSC investigation and characterized by using scanning electron microscope and ultra-micro-hardness tester.

BP-33 High Temperature Oxidation Resistance of Multicomponent Cr-Ti-Al-Si-N Coatings, ***C.-Y. Hsiao*** *(netass.tw@yahoo.com.tw), Y.-Y. Chang, D.-Y. Wang,* Mingdao University, Taiwan, *W. Wu*, National Chung Hsing University, Taiwan

The high temperature oxidation resistance of the Ti-Al-Si-N and Cr-Ti-Al-Si-N coatings was studied. These coatings were deposited on silicon substrates by using a cathodic-arc deposition system with lateral rotating arc cathodes. Chromium, titanium and Al-Si alloy cathodes were used for the deposition of Cr-Ti-Al-Si-N coatings with different alloy contents. For the high temperature oxidation test, the coated samples were annealed between 700°C to 1000 °C in air. In this study, field emission scanning electron microscope (FESEM), transmission electron microscope(TEM) and X-ray diffraction(XRD) using Bragg-Brentano and glancing angle parallel beam geometries were used to characterize the microstructure of the as-deposited and annealed films. The composition and chemical bonding of the deposited and annealed Cr-Ti-Al-Si-N coatings were evaluated by X-ray photoelectron spectroscopy (XPS). The oxidation kinetics of the deposited coatings showed parabolic behavior, indicating that the diffusion process occurred during oxidation. It has been found that the high temperature oxidation behavior of the deposited films is correlated with the alloy content and nanocomposite structure. It indicated that the Cr-Ti-Al-Si-N with higher Cr, Al, and Si contents possessed superior oxidation resistance than Ti-Al-Si-N. The different oxidation mechanisms of the deposited Cr-Ti-Al-Si-N at high temperature will be developed.

BP-34 Optical, Morphological and Electrochemical Properties of Niobium Oxide Thin Films, ***G. Ramírez,*** *S.E. Rodil (ser42@iim.unam.mx), S. Muhl,* Universidad Nacional Autónoma de México, *J.J. Olaya,* Universidad Nacional de Colombia, *M. Rivera,* Universidd Nacional Autónoma de México, *E. Camps, L. Escobar-Alarcon*, Instituto Nacional de Investigaciones Nucleares, México

Niobium Oxide thin films were deposited by unbalanced reactive magnetron sputtering under different conditions of pressure and oxygen/argon flow ratio. The films were characterized to obtain their physical, structural, optical and electrochemical properties using X-ray diffraction (XRD), Rutherford backscattering spectroscopy (RBS), variable angle ellipsometry, nanoindentation ad atomic force microscopy. Combining the results of XRD and RBS, we can conclude that the films were amorphous having the $Nb_2O_2$5 stoichiometry. Although the pressure and O_2/Ar flow ratio were varied between 2 to 4 Pa and 1/10 to 3/10, respectively, no large changes in the film properties were obtained The mechanical and optical coatings did not show significant variations between the different deposition conditions; all the films were transparent with a bandgap about 3.4 eV and the hardness was around 4.5 Gpa. The morphology of films, measured with AFM, showed that the surface roughness varied between 0.6 to 2.9 nm, without any clear trend. Nevertheless, concerning the electrochemical properties, the response of the films to a DC polarization in a 0.85% NaCl solution was significant different. The parameters used to compare the electrochemical response were the polarization resistance and the corrosion resistance obtained from a Tafel Analysis.

BP-35 Structure Characterization and Properties of Silicon Carbonitride Films Deposited by Reactive Magnetron Sputtering, ***X.M. He*** *(xiaoming_he@amat.com), M. Nastasi,* Los Alamos National Laboratory, *K.C. Walter*, Physical Optics Corporation

Amorphous silicon carbonitride (a-SiCN) films were synthesized on Si (100), glass and metal substrates by reactive radio-frequency magnetron sputtering of a Si_4C target with a mixture of Ar and N_2 gases. The composition, morphology, and chemical bonding of the films were characterized using ion beam analysis techniques, atomic force microscopy (AFM), infrared (IR), and X-ray photoelectron spectroscopy (XPS). The hardness, stress, optical and tribological properties were measured from the a-SiCN films of different compositions. The results show that the a-SiCN films have a hybridized bonding structure that is mainly comprised by the Si-C, Si-N, and C-C bonds, and the composition and bonding components of the films can be modified by adjusting the reactive N2 concentration through the sputtering depositions. The binding energy of the Si-C, Si-N, C-C and C-N bonds is not affected by the variations of Si, C and N incorporations, but the properties of the coatings can be enhanced by the reasonable balanced amounts of each bonding component. The a-SiCN films with optimal combinations of the composition and bonding structure can be synthesized to have high harness (> 26 GPa), high density (> 2.35 g cm^{-3}), improved optical properties, and enhanced wear resistance.

BP-36 Effects of Low-Temperature Duplex Coatings on Corrosion Behavior of Austempered Ductile Iron, ***C.-H. Hsu*** *(chhsu@ttu.edu.tw), C.-Y. Lee, K.-L. Chen, K.-C. Lu*, Tatung University, Taiwan

Austempered ductile iron (ADI) is an attractive engineering material due to its excellent strength, toughness and the low cost. In general, the austempering is isothermally treated about in the temperature range from Ms to 450°C, thus the traditional surface modification of high temperature can not be available to treat ADI. This study utilized electroless nickel (EN) and cathodic arc deposition (CAD) technologies, with the known advantage of low processing temperature, to treat the ADI substrate. The eligibility of applying the EN and CAD-CrTiAlN duplex coatings on ADI, along with the coating properties, such as structure, roughness, and adhesion were evaluated and analyzed. Moreover, polarization tests were performed to further understand the effect of the coatings on the corrosion resistance of ADI. The results showed that the unique microstructure of ADI did not deteriorate after EN and CAD treatments. With regards to the corrosion resistance, the duplex coat ed specimens performed better than that of the uncoated and monolithic EN or CrTiAlN coated ones in 3.5 % NaCl aqueous solution.

BP-37 Thermal Evolution, Mechanical and Corrosion Properties of Al Implantation into TiSiN Nanocomposite Coatings by MPII Hybrid System, *C.-L. Chang (clchang@mdu.edu.tw),* ***L.-Y. Tseng****, C.-W. Wu, Y.-C. Liu, W.-Y. Lin*, MingDao University, Taiwan

An as-deposited TiSiN film was post-treated with high-energy ion bombardment following the metal plasma ion implantation (MPII) process, involving an accelerated vacuum-arc metal plasma source with multiple charge states. In this study, the modification of the surfaces of energetic Al-implanted TiSiN films on the thermal evolution, mechanical and corrosion properties using metal plasma ion implantation (MPII) was investigated, by varying ion energy and dose. The microstructure and chemical states of aluminum, implanted on the surface layer of TiSiN films, were examined, as functions of ion energy and dose, by nanoindenter, Auger electron spectroscopy, X-ray photoelectron spectroscopy and X-ray diffraction. TiSiN and Al-implanted TiSiN coated samples were oxidized in air using a conventional furnace at different temperature for 2 h. The phase transformations and surface morphology of coatings were observed by utilizing X-ray diffractometer and AFM. The corrosion behavior of coatings was investigated using polarization and immersion tests. Polarization test of coatings was carried out in the solution of 3.5 wt.% NaCl. For immersion test, the coatings were exposed under H_2SO_4 environment for 24 h. The results turned out that better thermal evolution, mechanical properties and corrosion resistance of Al-implanted TiSiN coatings than that of TiSiN, which can be enhanced by Al implantation using MPII method.

BP-38 Using High Work Function Ni Metal to Improve the Stress Reliability of $CaCu_3Ti_4O_{12}$ MIM Capacitors, ***L.-C. Chang*** *(lcchang@mail.mcut.edu.tw),* Mingchi University of Technology, Taiwan*, C.-H. Yang, H.-L. Kao,* Chang Gung University, Taiwan*, F.B. Wu*, National United University, Taiwan

The RF sputtered $CaCu_3Ti_4O_{12}$ (CCTO) MIM capacitors were fabricated at 400°C using low-cost and high-work function Ni electrode to improve leakage current for VLSI backend integration. This stress degradation is especially a concern in CCTO materials, which also leads to high leakage currents because of the small conduction band discontinuity (δE_C) with respect to Si. We report improvements in the thermal leakage current by using Ni as a high-work-function top electrode for CCTO capacitors. This avoids sacrificing the overall value by using a multilayer or laminate structure and results in better voltage linearity, which is important for memory integrated circuits. Here we describe the stress reliability of the nonvolatile memory characteristics of hi-κ MIM capacitors.

BP-39 Fabrication and Characterization of HF-PECVD Silicon Films and Application for Thin Film Solar Cells, *S.-Y. Lien (syl@mdu.edu.tw),* ***C.-T. Shen****, C.-C. Wang, C.-H. Chao, K.-W. Weng, C.-F. Chen*, Mingdao University, Taiwan

The high frequency plasma enhanced chemical vapor deposition (HF-PECVD) is a well applicable deposition technique for large area and high rate deposition for silicon solar cell application. This paper presents the properties of a-Si:H films and high efficiency of p-i-n solar cells prepared using RF (27.1 MHz) excitation frequency. The influence of the power (10-40 W) and pressure (20-50 Pa) used during the deposition of absorber layers in p-i-n solar cells using pure silane on the properties of the films and solar cells is investigated. We summarizes the power and pressure effect on properties and growth mechanism of a-Si:H films. It was found that the a-Si:H films prepared under various deposition conditions show widely various deposition rate, optical-electronic properties and microstructure. After optimum the deposition parameters, the amorphous silicon based thin film silicon solar cells with efficiency of 7.2 % have been fabricated by HF-PECVD. These are very encouraging results for future fabrication of high efficiency thin film solar cells using by HF-PECVD.

BP-40 Phase Transformation, Thermal Stability and Indentation Behavior of Ni-P-Based Interlayer Enhanced CrN Composite Coatings, *Y.Y. Li, Y.C. Hsiao, J.C. Wu,* ***F.B. Wu*** *(fbwu@nuu.edu.tw)*, National United University, Taiwan

In present study, CrN/NiP and CrN/NiPAl composite coatings were fabricated by magnetron sputtering technique. The sputtering of Ni-P-based interlayers was fulfilled by the electroplating of patterned Ni-P thick films on Cu and Al disc substrates. The microstructure of the duplex coatings was controlled by the process temperature during sputtering and post annealing procedures. According to phase identification, the CrN coating was successfully deposited under process temperature from 350 to 550°C. A preferred CrN(200) orientation was observed for the CrN coating deposited on the NiP binary alloy interlayer under 350°C process temperature. The diminishing of the preferred orientation of CrN coating was accompanied by the phase transformation of the NiP layer as the deposition temperature was increased. On the contrary, the amorphous phase of the NiPAl interlayer retained until process temperature was raised to 450°C and no significant related preferr ed orientation was found for the CrN layer. It was believed the introduction of Al in NiP not only increase the thermal stability of the duplex coating, but improve the thermal resistance during CrN coating deposition. In addition, through loading-unloading nanoindentation analysis, significant creep behavior was observed when the microstructure of the interlayer was amorphous or fully crystallized. With adequate heat treatment, the creep of the composite coatings could be reduced by the precipitation and crystallization of the Ni-P-based interlayer.

BP-41 Superhard Coatings Prepared by Pulsed Magnetron Sputtering, ***M. Keunecke*** *(martin.keunecke@ist.fraunhofer.de), K. Weigel, K. Bewilogua,* Fraunhofer Institute for Surface Engineering and Thin Films, Germany*, W. Kölker, P. Jaschinski*, Cemecon AG, Germany

Hard coatings like TiN, TiAlN, titanium diboride or boron carbide are in use as hard and wear resistant tool coatings. These coatings often will be prepared by PVD techniques like arc evaporation or d.c. magnetron sputtering. Typical micro hardness values of such hard coatings deposited with d.c. magnetron sputter are more or less in the range of 30 GPa. As a clear advancement compared to d.c. magnetron sputtering processes the pulsed magnetron sputter deposition technique could be verified. Different hard coatings were prepared using the pulsed magnetron sputter technique in a CC800/9 batch coater equipped with 4 targets. Coatings prepared with the pulsed sputter process showed a significant increase in hardness. The highest hardness values were reproducibly measured with up to 50 GPa. Beside the hardness other mechanical properties like resistance against abrasive wear and adhesion were measured. Cross sectional SEM images showed the growth structure of the coatings. The chemical composition of the coatings was investigated by microprobe measurements. It could be stated that with a pulsed magnetron sputter process the property range of the hard coatings could be extended especially with respect to the hardness reaching for some coatings even the superhard (> 4000 HV) region.

BP-42 TiCrSiN and TiCrAlSiN Coatings Deposited by a Hybrid PVD Techniques, ***D. Yu*** *(iamydh@163.com), C. Wang, X. Cheng, F. Zhang*, Guangdong University of Technology, China

TiCrSiN and TiCrAlSiN coatings are synthesized onto stainless steel and cemented carbide substrates and Si wafers using a hybrid method of hollow cathode deposition (HCD) and magnetron sputtering (MS) techniques. In the current study, chemical composition, crystal structure and mechanical properties like microhardness, adhesion and friction coefficient are characterized. As the Cr increase, the TiCrSiN coatings decrease microhardness. The adhesion improves with the increase of Cr content. The average friction coefficient of TiCrSiN and TiCrAlSiN coatings are about 0.5. And the TiCrAlSiN coating shows promising application in the field of high speed milling hardened steel technology.

BP-43 Evaluation of the Electrochemical Behaviour of Tantalum Oxide Thin Fims for Biomedical Applications, ***P.N. Rojas****, S. Muhl (muhl@servidor.unam.mx), S.E. Rodil*, Universidad Nacional Autónoma de México

The corrosion resistance of materials in the body fluids is an essential factor to determine the lifetime of medical implants. Tantalum oxide films were deposited on medical grade stainless steel using an rf-magnetron sputtering system working at three different powers (50, 150 and 200 W) and different working pressures (0.67, 2, 4 Pa) These films were evaluated using Potentiodynamic polarization (PP) in 0.89% NaCl (7.4 pH) in order to determine the conditions that lead to the best corrosion resistance. The effect of the deposition conditions on the film physical properties was also evaluated by means of electron microscopy, energy dispersive analysis,

ultraviolet-visible spectroscopy, ellipsometry and infrared spectroscopy and correlated to the electrochemical parameters. Based on these results, one single deposition condition was chosen to make a further evaluation by electrochemical impedance spectroscopy (EIS) and Potentiodynamic polarization using three simulated body fluids; 0.89% NaCl solution, Hartman (ringers-lactate) and Gey´s solution, as a function of the immersion time.

BP-44 Microstructure and Properties of Laser-Cladded Cr-Ni Coatings Prepared on Steel, *A. Iwaniak (aleksander.iwaniak@polsl.pl),* ***E. Augustyn****, J. Adamiec*, The Silesian University of Technology, Poland

This article will be presents results of microstructural investigation and properties characterization of Ni-Cr coatings deposited on carbon steel by flame spraying and diode laser cladding processes. Ni-Cr coatings were deposited on steel substrates by flame spraying using of commercial powders Metco, next was cladding by using 2kW diode laser. The phases of clad layer were investigated by an optical microscope, scanning electron microscopy (SEM), X-ray diffractometer (XRD), electron probe microanalysis (EPMA) and energy-dispersive spectrometer (EDS). If proper cladding parameters are used, the clad layers will have a good surface shape, sound metallurgical bonding with base metal, low dilution, and the effect of heating on heat-affected zone metal can be limited. A clear dependence of the micro-cracking susceptibility on the base preheating temperature is observed for Ni-Cr coatings prepared by direct laser melting on the preheated steel. The number of cracks decreas es with the preheating temperature and for preheating for 300°C no cracks are observed. It was found that the laser cladded zone has a higher microhardness value compared with that of the flame spraying treated material. This is a result of the significant reduction in grain size in the case of laser cladding. Unlike the flame spraying cladded zones, the laser treated material is free of micropores and microcracks.

BP-45 Development of Temperature Sensor Thin Films to Monitor Turning Processes, *W. Tillmann,* ***E. Vogli*** *(evelina.vogli@udo.edu),* TU Dortmund University, Germany*, K. Pantke, D Biermann*, Institute of Machining Technology, Germany

Increasing demands on the chipping process require a fundamental analysis concerning the design as well as the material selection for cutting insert and its wear protection. By employment of novel wear resistance thin coatings the cutting inserts can be effectively protected from wear. Apart from the monitoring the wear and the cutting forces the knowledge of the developed temperatures during cutting process is essential and necessary. In this work an innovative technology was employed to measure in-situ the temperature development during cutting process. The measurement was based on the Seebeck-effect. Coating adhesion was systematically analyzed and optimized by varying of pretreatment conditions. Furthermore the design of masks was enhanced and finally turning experiments were carried out to scrutinize the efficiency of deposited temperature sensors in cutting tests.

Optical Thin Films
Room: Town & Country - Session CP

Symposium C Poster Session

CP-1 Preparation of TiO_2 Photoelectrode by Spray Pyrolysis Technique for the Photovoltaic Application, *M.F. Hossain, S. Biswas, M. Shahjahan,* ***T. Takahashi*** *(takahash@eng.u-toyama.ac.jp)*, University of Toyama, Japan

Among the new generation photovoltaic cell, dye-sensitized solar cells (DSCs) have been the subject of intense study on account of their high conversion efficiency and low cost. Significant progress regarding the efficiency of DSSC devices was made possible by employing nano-porous crystalline TiO_2. Among the various deposition techniques, spray pyrolysis technique has also proved to be a simple and inexpensive method particularly useful for large area applications. In the technique, different processing parameters can be varied to control structure and morphology of the thin film. In this present work, porous TiO_2 thin films were fabricated. by using spray pyrolysis technique. The solution containing titanium (IV) isopropoxide as a titanium source, acetone and ethanol was sprayed onto SnO_2 :F coated glass at different substrate temperatures of 300-500 C.For the fabrication of solar cell, cis-dithiocyanato-bis(2,2'-bipyridyl-4,4'-dicarboxylate) rut henium(II) dye was used along with carbon paste electrodes on SnO_2:F coated glass as a counter electrode. All the TiO_2 photoelectrodes were characterized by XRD, Raman and UV-visible spectroscopy. Surface morphology of the sample was studied with FE-SEM and AFM. Incident photon-to-current efficiency (IPCE) was also calculated for all the DSCs with different TiO_2electrodes. The amount of dye incorporation was found to be highly dependent on the microstructure and the thickness of the film, as apparent from optical measurements. The variation of IPCE and photoelectric conversion efficiency with solar cells of different TiO_2 films is discussed with the analysis of different microstructure of the TiO_2 thin films.

CP-2 Effects of Oxygen Flow Ratios and Annealing Temperatures on Raman and Photoluminescence of Titanium Oxide Thin Films Deposited by Reactive Magnetron Sputtering, *C.K. Chung (ckchung@mail.ncku.edu.tw), M.W. Liao,* ***C.W. Lai***, National Cheng Kung University, Taiwan

Titanium oxide thin films were deposited on the Si (100) substrate by dc reactive magnetron sputtering at 3-15 % oxygen flow ratios (O_2/(Ar^+O_2)) at room temperature, and then annealed by rapid thermal annealing (RTA) at 350, 550, and 750°C for 2 min in atmosphere. The bonding and luminescence behavior of as-deposited and annealed titanium oxide thin films were analyzed by Raman and photoluminescence (PL) spectroscopy, respectively. Raman spectra results indicate that titanium oxide peaks are weak in the as-deposited films but the pronounced peaks of the mixed anatase and rutile phases were detected after RTA at 350-750°C. The Raman intensity of rutile phase increases with increasing oxygen flow ratio together with annealing temperature. The PL spectra of post-annealed titanium oxide films show a wide FWHM peak in wavelengths of 365- 550 nm. The peak can be fitted into three Gaussian peak at ~380 nm (3.26 eV), ~410 nm (3.02 eV), and ~440nm (2.81 eV). The peak positio n at ~380 nm corresponds to energy bandgap of the anatase phase through electron-hole pair recombination while that at ~410 nm is emitted from the rutile phase. The peak around 440 nm is attributed to oxygen defects. The intensity ratio of the rutile peak to anatase peak increases with increasing oxygen flow ratio. It is in consistent with Raman results. In addition, the relationship between the PL shift of oxygen defects peak and oxygen flow ratio is discussed.

CP-3 Transparent Thermally Stable Poly(etherimide) Film as Flexible Substrate for OLEDs, *C. Legnani, W.G. Quirino,* CeDO - Inmetro, Brazil*,* ***M. Cremona*** *(cremona@fis.puc-rio.br),* PUC-Rio, Brazil*, V.L. Calil,* CeDO - Inmetro, Brazil*, G.F. Moreira, C.A. Achete, C. Vilani*, Dimat - Inmetro, Brazil

Due to their applications in many different areas and the simplicity of manufacturing, Organic Light Emitting Diodes (OLEDs) hold great promise in research dedicated to the development of new optoelectronic and photonic devices. These organic devices, mainly small molecule vacuum-deposited ones, can also be fabricated onto plastic film substrates, as for example the polyethylene terephthalate (PET) and the poly carbonate (PC), for the development of flexible OLEDs (FOLEDs). Polyimides are well known for their excellent thermal and chemical stability and good mechanical, optical and electrical properties[1]. Their strength and heat and chemical resistance are so great that these materials often replace glass and metals, such as steel, in many demanding industrial applications. In this work, a Poly(ether imide) film, PEI, was used as flexible substrate for the fabrication of FOLEDs. The degradation temperature for this material, as reported in the literature, is about 526°C allowing its application in hostile environments. The polymeric film used in this work was obtained by inversion phase by evaporation solvent in nitrogen atmosphere or in vacuum at 80°C, forming a ~0,5 mm thick film. The DSC analyses confirmed the high, 252°C, transition glass temperature (Tg) of this substrate. The optical transmittance is about 86% at 550 nm. In order to achieve the necessary conductive properties indium tin oxide (ITO) thin films were deposited onto PEI at room temperature using a r.f. magnetron sputtering with an r.f. power of 40W, at pressure of 2,5 mPa in Ar atmosphere with a subsequent thermal treatment at 250°C. Resistivity, mobility and carrier concentration of as-deposited ITO films were $3.96x10^{-4}$ Ohm-cm, 19.5 cm^2/V.s and $8.0x10^{20}$/cm^3, respectively, and the properties of the post annealed at 250°C were $3.28x10^{-4}$ Ohm-cm, 25.6 cm^2/V.s and $9.9x10^{20}$/c m^3, respectively. The mobility and the carrier concentration increase, leading to a decrease in the sheet resistance value. The low resistivity and high stability of the new substrate open the possibilities to develop devices that work in extreme conditions.

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CP-4 Holographic Grating Formation in PVB Doped Polymer Dispersed Lilquid Crystal Based on PUA, ***E.H. Kim*** *(udam99@changwon.ac.kr), Y.G. Jung, U. Paik*, Changwon National University, Korea

Holographic polymer dispersed liquid crystals (HPDLCs) are a relatively new class of materials that have potential device applications in displays and optical shutters. Polymer matrix used in HPDLCs should have high transmittance, adhesive, compatibility with LC, mechanical property and

thermal stability to enhance stability of LC droplet and durability as well as electro-optical performance of HPDLC films. In present study, we incorporated different contents of polyvinyl butyral (PVB) into the conventional polyurethane acrylate (PUA) system at a specific composition. PUA ologomer, PVB and LC having simular solubility parameters shows clearly homogeous mixture before photpolymerizaion until the content of PVB is up to 10 wt%, resuling in high diffraction efficiency and good phase separation. As the content of PVB is increased, the hardness, elastic modulus and thermal stability are improved due to the expansion of distant of crosslinks and entanglement by PVB molecular with a high molecular weight relatively compared to PUA oligomer. Off-state diffraction efficiency is increased gradually with increasing PVB content owing to the increase of elasticity of polymer matrix, which implies that phase separation between polymer and LC are augmented. However, the increase of viscosity by adding PVB causes the deterioration of diffraction efficiency, showing lower diffraction efficiency at PVB content of 10 wt% than 1 wt%. Also, HPDLC films having low driving voltage (<50 V), fast response time (<6 ms) and high diffraction efficiency (>70 %) could be fabricated with 1 wt% PVB at 40 wt% LC.

CP-5 Study of Structural and Optical Properties of TiO_2:Tb Coatings Prepared by High Energy Reactive Magnetron Sputtering, *D. Kaczmarek,* ***J. Domaradzki*** *(jaroslaw.domaradzki@pwr.wroc.pl),* Wroclaw University of Technology, Poland*, Z.J. Radzimski*, Silicon Quest International

From a review of the literature published during last decade a growing pursuit of obtaining dense crystal structure of different coatings with novel properties may be noticed. Specifically, many papers address titanium dioxide (TiO_2) thin film prepared by using different deposition methods. Because of industrial applications, the most important in this field seems to be vapor physical deposition methods, such as magnetron sputtering. Typically, enhanced energy per molecule, which is needed for obtaining a dense structure in the sputtering process, could be assured by appropriate selection of pressure, substrate temperature, argon/oxygen ratio and power of plasma discharge. This work is focused on structural and optical properties of TiO_2 thin films doped with terbium. The thin films have been prepared by high energy reactive magnetron sputtering. The method was modified to achieve dense nanocrystalline high temperature stable rutile form of the TiO_2 fi lm. Thin films were deposited from a mosaic Ti-Tb target sputtered under oxygen plasma (without argon) at a pressure $<10^{-1}$ Pa. Besides using only O_2 as a reactive and working gas and a low pressure, enhanced energy of particles during the film growth was maintained by increasing the target temperature and by using 164 kHz unipolar pulses with peak amplitude of 1800 V for powering the magnetron source. Using X-ray diffraction, X-ray photoelectron spectroscopy and atomic force microscopy it has been shown that by doping with Tb at a specified amount of 0.4 %, thin films had dense anatase structure. Moreover, this phase was stable even after additional annealing up to 1000 K. Optical properties were studied by the application of optical transmission and photoluminescence measurements (PL). The thin films were transparent in a wide range of optical spectra. The most intense PL spectra were recorded for coatings with the Tb amount of 2.6% where the Tb^{3+} were embedded into TiO_2 – rutile matrix. The energy transfer from TiO_2 nanocrystallites to Tb ions that occurred in the thin films was also discussed.

CP-6 The Carbon Effects on the Phase Transformation of Visible-Light Responsive Carbon Containing TiO_2 Nanoparticles, ***Y.-H. Cai,*** *S.-J. Cai,* National Dong Hwa University, Taiwan*, V. Yeh, S.-B. Wu,* Natioanl Dong Hwa University, Taiwan*, C.-L. Cheng (clcheng@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

Visible-light-responsive titanium dioxide (TiO_2) has attracted increasing attention recently. While commercially available TiO_2 responses only in the UV region, visible-light-responsive TiO_2 allows using the visible region of the solar light for photocatalytic reactions. A new carbon containing visible-light-responsive mixed-phase TiO_2 has been proposed and proved to be efficient[1]. Enhanced photocatalytic effects were found when anatase mixed with carbon covered rutile phases in the TiO_2 nanoparticles[2]. In this study, we discuss the phase transformation mechanism on the mixed-phase TiO_2 using in-situ Raman mapping and temperature programmed desorption (TPD) methods. The mixed phase was found due to carbon facilitated anatase/amorphous to rutile phase transformation at lower temperature. Evidence on the carbon facilitated anatase/amorphous to rutile phase transformation at lower temperature (200 °C) was demonstrated throu gh in-situ observation of Raman mapping on the transformation. In a further effort to understand the phase transformation, pure anatase phase TiO_2 with carbon admixture was employed to study the phase transformation. While the pure anatase phase transformed to ruitle phase at temperature high than 800 °C, the amorphous TiO_2 will phase transform facilitated by the carbon inclusion at relatively lower temperature. A possible oxygen deficiency mechanism leading to the phase transformation will be discussed as evidenced from the TPD observation.

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CP-7 Characteristics of Indium Zinc Tin Oxide Thin Film Prepared on Flexible Substrates Using the Hetero-Target Sputtering System, ***D.H. Kim,*** *Y.S. Rim, H.-W. Choi, K.-H. Kim (khkim@kyungwon.ac.kr)*, Kyungwon University, Korea

The Indium Zinc Tin Oxide (IZTO) thin films for transparent thin film transistor (TTFT) were deposited on polycarbonate (PC) and polyethersulfone (PES) and glass substrates at room temperature by facing targets sputtering (FTS). Two different kinds of targets were installed on FTS system. One is ITO (In_2O_3 90wt.%, SnO_2 10wt.%), the other is IZO(In_2O_3 90wt.%, ZnO 10wt.%). As-deposited IZTO thin films were investigated by a UV/VIS spectrometer, an X-ray diffractometer (XRD), an atomic force microscope (AFM) and a Hall Effect measurement system. As a result, we could prepare the IZTO thin films with the resistivity of under 10^{-4} [Ω•cm] and IZTO thin films deposited on glass substrate showed an average transmittance over 80% in visible range (400~800 nm) in all IZTO thin films except in IZTO thin film deposited at O_2 gas flow rate of 0.1 [sccm].

CP-8 Effect of Thermal Acceleration on the Degradation of Gallium doped Zinc Oxide Thin Film, ***J. Kang,*** *M. Lee (mhlee@kicet.re.kr), H. Choi, W. Seo,* Korea Institute of Ceramic Engineering and Technology, Korea*, D.Y. Lee*, Daelim College of Technology, Korea

Transparent conducting Oxides (TCO) thin films such as indium tin oxide (ITO) and zinc oxide (ZnO) have been widely studied for their practical applications as transparent electrodes, solar cell and various opto-electronics. However, because of the high cost and scarcity of indium, transparent conducting ZnO has recently attracted much attention as a promising alternative material. Undoped ZnO thin films are not stable especially at high temperature, doping the zinc oxide can reduce this disadvantage. ZnO doping is achieved by replacing Zn2+ atoms with atoms of element of high valence such as aluminum, gallium. But, Gallium element has more advantages than Al element because the size of Ga ion as impurities is similar to size of zinc ion, minimizing lattice defect. Through several researches, electrical properties of doped ZnO thin film has secure closely ITO. However, TCO Thin films go through various thermal processes and environmental condition where these could be a ffected by heat. In this research, the effect of thermal degradation on Ga doped zinc oxide (GZO) thin film was studied in higher accelerated conditions of temperature. GZO thin film was prepared by R.F sputtering using ZnO target doped with 5wt% of Gallium. The prepared GZO films were exhibited thickness of 150nm, resistance of 30~40Ω/sq and transmittance of 90%. The percentile increment of resistance by thermal acceleration exceeded 15% after 26.9hr, 2.8hr, 0.1hr at 200C, 250C, 300C, respectively. Surface roughness (Ra) of GZO film increased to 10times by thermal condition. The acceleration factor and the activation energy of degradation were calculated.

CP-9 Linear and Nonlinear Optical Properties of CdSe/PMMA Nanocomposite Filters with Mechanical Property Improvement, *G. Chen,* University of Arkansas*, Y.A. Wang,* Ocean NanoTech, LLC*., M. Xiao,* ***M. Zou*** *(mzou@uark.edu)*, University of Arkansas

Hybrid composites of CdSe nanocrystals embedded in poly(methyl methacrylate) (PMMA) matrices were prepared by casting methods which disperse the quantum dot powder and PMMA in chloroform solution. X-ray diffraction (XRD) measurements of the CdSe/PMMA nanocomposites show broad pattern for cubic CdSe. The particle size of CdSe estimated from XRD measurements is 8.5 nm. UV-vis absorption spectra and refractive index measurements of the nanocomposites show that the linear optical properties of the nanocomposite films are strongly dependent on the CdSe nanocrystal concentration in the PMMA polymer. The room temperature nonlinear optical properties of the nanocomposites were investigated using a single-beam Z-scan technique with femtosecond laser pulses at the wavelengths of 794 nm. The experimental data reveals that the optical nonlinearity varies with the input laser energy as well as the concentration of the CdSe nanocrystal in the polymer. In addition, mechanical property characterization results show that both the Young's modulus and the hardness of the nanocomposite film increase as the concentration of CdSe nanocrystal in the PMMA polymer increases. The results of this study

indicate that CdSe/PMMA nanocomposite films, having favorable nonlinear optical properties while in the mean time possessing improved mechanical properties than pure PMMA, are promising material for optical filter applications.

CP-10 Influence of Sputtering Gas on the Properties of Reactively Sputtered Tungsten Oxide Films, ***A.K. Chawla****, S. Singhal, R. Chandra (ramesfic@iitr.ernet.in), H.O. Gupta*, Indian Institute of Technology Roorkee, India

Due to high refractive index and chemical stability characteristics, tungsten oxide (WO_3) thin films were used in a wide range of optical, electrical and chemical applications such as electrochromic devices, solar cells, photo catalysts, antireflective coating and chemical sensors. Considering the field of optical applications, the basic requirements of fabricated thin films are homogeneous, non-porous, high packing density and low optical loss. Depending on the deposition conditions and synthesis techniques films may present considerably different structural, optical and electrical behavior, and consequently different electro chromic behavior. With DC magnetron sputtering thin film properties can be improved by controlling the reactive gas atmosphere. Bandgap values from 2.6 to 3.4 eV, reported for polycrystalline and amorphous WO_3 thin films, only allow for absorption in the near ultraviolet and blue region of the solar spectrum. Bandgap modifications woul d be necessary to extend the light harvesting capability to a wider portion of the solar spectrum. Bandgap modification of WO_3 films through doping techniques is proposed. In this work, nitrogen doping was used to modify structural and, optical, properties of the material in the presence of two inert gases (Argon and Helium). Tungsten oxide films were produced using reactive dc magnetron sputtering. Substituting Helium gas in place of argon results in a decrease in the particle sizes and thus affects the band gap values. Crystal structure and surface morphology were studied by X-ray diffraction (XRD) and atomic force microscope (AFM), respectively. We find that the atomic mass of the sputtering gas significantly affects the primary crystallite size as well as the surface morphology and texture. Optical properties were studied using UV-Vis-NIR spectrophotometer in the visible spectrum.

CP-11 Modulation of Luminescence Emission Spectra of N-Doped Ga_2O_3 Nanowires by Thermal Evaporation, *H.-C. Shih (hcshih@mx.nthu.edu.tw),* ***L.-W. Chang****,* National Tsing Hua University, Taiwan*, M.W. Huang, C.-F. Li,* National Chung Hsing University, Taiwan*, J.-W. Yeh*, National Tsing Hua University, Taiwan

In this study, we have synthesized N-doped Ga_2O_3 nanowires on a p-type Si (100) substrate with N_2 (50 sccm) and O_2 (1 sccm) at 800°C through a two-step evaporation to modulate the spectra of the luminescence emission. Both TEM and XRD analyses confirmed that N-doped Ga_2O_3 is monoclinic with a uniform mean diameter of 40 nm and a length up to several tens of micrometers. As determined by selected area diffraction (SAD), the growth direction of N-doped Ga_2O_3 nanowires is [002]. The optical properties of the N-doped Ga_2O_3 nanowires were studied by photoluminescence (PL) at the room temperature, exhibiting a red-light (738nm) and near infrared-light (805nm) emissions as a function of the nitrogen dopant in response to Ar-Kr laser excitation at 532nm. The results serve to reinforce the potential of N-doped Ga_2O_3 nanowires for optoelectronic device applications.

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CP-12 Characteristics of ITO-ZnO:Al Thin Films Grown by Combinatorial Pulsed Laser Deposition, ***V. Craciun*** *(valentin.craciun@inflpr.ro)*, National Insitute for Laser, Plasma and Radiation Physics

Doping or alloying materials to obtain new and better properties are well-known processes employed to meet the challenges of newer and more demanding applications. Since it is not yet possible to predict, based on first principles, the exact amount of doping or alloying element needed to optimize a specific property of the new phase or material studied, various strategies have been developed. The combinatorial method, where two or more sources of atomized materials that are spatially separated are simultaneously or alternatively used for deposition on a single substrate is a very good example of such a strategy. We have used the combinatorial pulsed laser deposition method to obtain transparent and conductive oxides by mixing indium tin oxide with pure or aluminum doped zinc oxide. By changing both the direction and the flux of the atomized incoming species, one has control upon the lateral and vertical compositional gradients of the growing thin film, while by simply alternating the deposition targets one can obtain multilayers with different periodicities both vertically and laterally, along the surface. This approach allowed us to control the microstructure and properties of the deposited films. Since ZnO exhibits a stronger tendency to grow textured than ITO, by firstly depositing a few monolayers of ZnO on the substrate a template is created, resulting in the growth of a textured mixed compound. The cubic ITO is forced to adopt a textured growth along its (222) axis that possesses a similar symmetry as the hexagonal (0002) axis of ZnO. By changing the ratios of the ITO to ZnO fluxes we also observed a continuous change of the lattice parameter along the growth axis, between those of pure ZnO and pure ITO. Moreover, the crystalline grain sizes and stress levels were also dependent on the local chemical composition. Similar trends were observed for the optical band-gap, optical transmission, and electrical properties, which were found to also depend on the ZnO to ITO ratios.

CP-13 Characterization of Anomalous Luminescence Properties from Self-Ordered Porous Anodic Alumina with Oxalic Acid Electrolytes, ***Y.-R. Chen*** *(m9624027@stmail.cgu.edu.tw), C.-H. Fang,* National United University, Taiwan*, J.-C. Wang, T.-E. Nee*, Chang Gung University, Taiwan

In recent years, self-ordered porous anodic alumina (PAA) film have been extensively used as templates, mask, or host materials to synthesize various nanostructures, and building blocks for developing nanoscale devices. PAA film produced by the anodization technique has a nanoscale porous structure in mass production and the pore height and diameter are controllable and was applied in the fabrication of visible spectral range optical devices. In order to characterize the luminescence properties, we have formed the self-ordered PAA films which evaporated onto silicon substrates. In this work, the anomalous luminescence properties of carrier confinement in PAA films have been investigated by introducing oxalic acid electrolyte into the anodization technique. The temperature-dependent photoluminescence (PL) spectra were measured to characterize the recombination mechanisms. From the PL spectra of PAA films, it has found the asymmetrical luminescence profile in the blue emi ssion region. It was used the Gaussian function to divided into two subbands which originate in two kinds of different oxygen-deficient defect centers, i.e., F+ (oxygen vacancy with only one electron) and F (oxygen vacancy with two electrons) centers, respectively. The density of the F centers is the largest on the surface, followed by a gradual decrease with an increase in the pore wall depth and electrolyte concentration. However, it was observed the reverse trend of the F+ centers. In strong contrast to a commonly expected trend of uniformly reduced non-radiative recombination with decreasing the lattice temperature, anomalous low-temperature PL growing and declining is observed between the F and F+ centers. The rate equation models are invoked to corroborate the anomalous temperature behaviors. All the calculations are agreement with the experimental observations. The temperature-dependent lifetimes for the PAA films will be discussed in detail as well.

CP-14 Enhancement of Light Reflectance and Thermal Stability in Ag-Mg Alloy Contacts on p-Type GaN, ***Y.H. Song****, G.H. JUNG, J.H. Son, J.-L. Lee (jllee@postech.ac.kr)*, Pohang University of Science and Technology (POSTECH), Korea

Recently, flip-chip and vertical-structure designs were exploited in GaN-based LEDs to improve the light extraction efficiency. In these configurations, highly reflective p-Ohmic contacts must be essential for increasing light extraction efficiency. Silver (Ag) has been widely used for a reflective ohmic contact due to its high reflectance (>95%) for visible light and low electrical resistivity. However, agglomeration has been observed at high temperature and considered as a weak point of Ag contact. The agglomeration of Ag film affects the electrical resistivity and optical properties of device operation. Therefore, preventing Ag from oxidation and/or agglomeration is a key aspect in obtaining high quality Ag-based ohmic contacts. Until now, several efforts to improve the thermal stability of Ag-based ohmic contacts have been demonstrated such as Ag-Al alloy and Ag-Cu alloy contact. However, degradation of electrical and optical properties was still found in these allo y-based ohmic contacts. In this presentation, we investigated Ag-Mg alloy contact as a method to prevent agglomeration of Ag contact on GaN at elevated temperatures. Changes in surface morphology and micro-structure in both Ag and Ag-Mg alloy contacts (0.1 wt.% Mg) were investigated using AFM, SEM, and XRD. Interfacial reactions between contact metals and GaNwere analyzed using depth profiles of secondary ion mass spectroscopy and synchrotron radiation photoemission spectroscopy. Based on these experimental results, the effects of Mg addition to Ag on the electrical and optical properties of the contact are proposed. Ag-Mg alloy contact was developed for obtaining low contact resistivity and high reflectance ohmic contacts on p-type GaN. Specific contact resistivity of 2.0 x 10-5cm2 and light reflectance of 82% on

sapphire were achieved from Ag-0.1 wt.% Mg alloy contact after annealing at 400oC in air. Because Mg atoms in Ag matrix have high diffusivity and low oxidation Gib bs formation energy, Mg atoms in Ag matrix move to surface of Ag-Mg alloy contact. This suggests that oxidized-Mg overlayer can be formed on the surface during the thermal annealing process in an air ambient. These oxidized-Mg overlayer could act as a diffusion barrier for excessive incorporation of oxygen into the Ag layers with the transformation into MgO. As a result, oxidized Mg atoms on Ag-Mg alloy contact induced the good thermal stability in Ag-Mg alloy contact, preventing Ag agglomeration.

CP-15 Electrochromic Tungsten-Titanium Oxide Films Deposited by Co-Sputtering using a Pulsed Sputtering Deposition System, ***K.-W. Weng***, Mingdao University, Taiwan, *S. Han (shenghan@ntit.edu.tw),* National Taichung Institute of Technology, Taiwan, *Y.-C. Chen*, National Chung Hsing University, Taiwan

Titanium doped tungsten oxide films deposited by co-sputtering metallic titanium and tungsten using a pulsed sputtering deposition system. The pulse power was varied in the range 100 to 500 W keeping the gas flow rate and pulsed frequency. The films are amphorous or microcrystalline depending on the pulse power. The XRD results show that for low pulse power the films present an amphorous tungsten trioxide, while for high pulse power (about 400 W) they present a mixture of a more crystalline. The XPS examination showed the existence of +6 valence tungsten ions and +4 valence titanium ions. The as deposited films with 400 W are transparent with transmittance exceeding 70% in the near-IR region. An in situ three electrode cell with a 0.1 M lithium perchlorate/propylene carbonate electrolyte was used. It is a good electrochromic behavior at an optimal pulse power 400W.

Carbon and Nitride Materials: Synthesis-Structure-Property Relationships
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Symposium D Poster Session

DP-1 Silver-Carbon Nanoparticles Produced by High-Current Pulsed Arc, ***F. Maya*** *(fermr@correo.unam.mx), M. Miki-Yoshida,* Centro de Investigación en Materiales Avanzados (CIMAV), México, *S. Muhl, O. Peña*, Universidad Nacional Autónoma de México

Great interest has been focused on Ag-Carbon material systems due to the possibility of obtaining Ag nanoparticles encapsulated by crystalline or amorphous carbon. Silver exhibits excellent thermal and electrical conductivity and has been extensively used in catalysis, electronics, photonics, photography, biological labeling, and surface-enhanced Raman scattering. Composite nanostructures using nanoscale silver as the core have been prepared by many research groups. For example, Ag/C nanocables and Ag/C nanoparticles have been synthesized in the presence of PVP; silver/cross-linked poly (vinyl alcohol) coaxial nanocables have also been prepared. In this work we have used a High-Current Pulsed electric arc in Argon between 3.3mm graphite and 1.5mm silver electrodes to produce silver-carbon nanoparticles at different gas pressures. These nanoparticles were analyzed by TEM, SEM, Optical absorption and XRD. Firstly, we measured the relation between the deposition rate and pressure and found that the deposition rate increased when the pressure decrease. The XRD analysis showed that the silver is crystalline; to confirm this the nanoparticles were analyzed by TEM. The Electron Diffraction again showed that the silver part of the nanoparticle is crystalline. Furthermore, it was established that the particles consisted of a crystalline silver nucleus coated with amorphous carbon. The size of the particles varied from ~150nm to less than 5nm depending on the gas pressure (800-100 Torr). Additionally, small quantities of cristalline graphite and amorphous carbon were found in the produced deposits. By EDS we studied the composition and structure of the deposits as a function of the gas pressure, as well as, the duration of the arc.

Finally, the analysis of the optical absorption spectra showed that the nanoparticles are not spheres but rather spheroids (an ellipsoid with two equal axes). Both, the aspect ratios ε (the ratio of the largest to the smallest axes) and the equivalent radii r_v(radius of a sphere having the same volume) are dependant on the gas pressure. This is interesting since by changing the aspect ratios, it is possible to tune the frequency of the surface plasmon resonance (SPR). This SPR tunability makes these nanoparticles potentially interesting for many optical and optoelectronic applications.

DP-2 Optical and Electron Field Emission Properties of Silver Doped Diamond Like Carbon Films Deposited by RF Reactive Sputtering Technique, ***Sk.F. Ahmed*** *(faruquekist@gmail.com), M.W. Moon, K.R. Lee*, Korea Institute of Science and Technology, Korea

Diamond-like carbon (DLC) film has been extensively studied due to their remarkable properties and potential applications. The ratio of sp^2/sp^3 carbon atoms is one of the most important factors determining the quality of the DLC films, which can be changed by incorporating different elements into DLC matrix. Various attempts have been made to dope DLC films with different elements such as boron, phosphorus, nitrogen, sulfur, silicon and tin etc. and the doping effects of these elements have been extensively investigated. Among them silver incorporated diamond like carbon (Ag:DLC) has been an interesting research field of diamond-like carbon owing to its potential for solving some of the major drawbacks of pure DLC films. Ag incorporation in the DLC films reduce surface free energy and residual internal stress without sacrificing the hardness, increase hydrophobic properties and improve hemocompatibility and antibacterial properties for biological applica tion. In this work we have reported the effect of silver incorporation on the optical and electron field emission properties of DLC films deposited by the RF reactive sputtering technique. The chemical binding energy and the compositions of the films were investigated by X-ray photoelectron spectroscopy (XPS) studies. Optical transparency and optical band gap decreased with the silver incorporation to the DLC film. Optical band gap calculated from transmittance spectra decreased from 2.55 to 1.95 eV with a variation of Ag concentration from 0 to 12.5 at. %. The field emission measurements showed that the threshold field and effective emission barrier were reduced by silver doping and the emission current strongly depends on the silver doping percentage. The threshold field was found to decrease from 6.8 to 2.6 V/μm with a variation of Ag atomic % from 0 to 12.5. The field enhancement factor was calculated and we have explained the emission mechanism.

DP-3 Temperature Programmed Desorption and Spectroscopic Studies of Surface Functionalized Nanodiamond Particles Prepared for Bio-Applications, ***S.-J. Cai***, *Y.-C. Chiu,* National Dong Hwa University, Taiwan, *V. Yeh, C.P. Chen,* Natioanl Dong Hwa University, Taiwan, *C.-L. Cheng (clcheng@mail.ndhu.edu.tw)*, National Dong Hwa University, Taiwan

Nanometer-sized diamond (ND) has been identified to be a promising and bio compatible nanoparticle for bio applications. It can be easily conjugated with interested bio molecules; and the functionality of the conjugated bio molecules can be preserved. Despite various methods have been proposed for surface functionalization, there have been limited studies on the functionalized nanodiamond surfaces. In this study, we employed both spectroscopic (IR and Raman) and surface (Temperature Programmed Desorption, TPD) methods to investigate the surface properties of surface functionalized nanodiamonds. The creation of molecular functional groups on nanodiamond surface was achieved chemically on ND of various sizes (5-500 nm in diameter). In the first case, strong acid treatment methods create ND-COOH functionalized ND surfaces; the second case, ND-$(CH_2O)_n$ are created on the nanodiamonds' surface chemically for further conjugation with bio molecules. The surface fu nctional groups are characterized with the above mentioned methods. Various chemical bonding serves as marker to confirm the formation of the functional groups. The surface C=O stretching frequency was studied in the ND-COOH system. This frequency is ~1820 cm^{-1} for particle size ~500 nm, and down shifts to 1725 cm^{-1} with decreasing particle size to 5 nm as a result of hydrogen bonds formation between the COOH groups in the particles surface; and the C=O stretching is strongly size- and temperature-dependent. Desorption of water molecules by TPD experiment confirm the formation of hydrogen bonds. The observed red-shift on the C=O stretching for 100 nm diameter nanodiamond from 1816 cm^{-1} to 1804 cm^{-1} when temperature increased from room temperature to 600 °C was attributed to water desorption. We also developed method to characterize the functionalized ND-$(CH_2O)_n$ using infrared spectroscopic methods. The temperature desorp tion experiments confirmed the covalent bonging of the designed functional group on nanodiamond surfaces. This work provides understanding of nanodiamond surface and the spectroscopic properties of the functionalized nanodiamond surfaces.

DP-4 Preparation and Evaluation of the Porous Graphite/Carbon Composites for Lithium-ion Battery with High Rate Charging, ***M.L. Lee***, National Tsing Hua University, Taiwan, *J.M. Chen,* Industrial Technology Research Institute, Taiwan, *J.-W. Yeh, H.-C. Shih (hcshih@mx.nthu.edu.tw)*, National Tsing Hua University, Taiwan

The electric vehicle (EV) is believed to be one of the most important industries in this century, and the lithium ion polymer battery should be the main choice because of its performance. Since high rate charging of lithium-ion battery is the major problem of this electric device. Recently, graphite material has popularly taken the place of commercial meso-carbon

micro-beads (MCMB) in the anode material of the lithium-ion polymer batteries. The study utilized the spray dry technique to manufacture the porous graphite/carbon and by controlling the vaporizing of solvent in different rates to synthesize varied structures of the porous graphite/carbon composites for lithium-ion polymer batteries with high rate charging. The composite graphite performs significantly batter than MCMB in surface area e.g., the MCMB is ~4 BET (m^2/g) and that of the porous graphite/carbon composite is ~40 BET (m^2/g). SEM shows the morphology of porous graphite/carbon composites a nd that some other tests are done for checking the electrochemical properties of those graphite/carbon materials. The porous structure of the graphite material contributes to the diffusion of the lithium-ion while charging and discharging in a battery. By increasing the efficiency of high rate charging and discharging, the porous graphite/carbon material is popularly used in lithium ion polymer batteries for the electric vehicle or high power electric devices. [1] [1]J.Li,X.Wang, Q.Huang, S.Gamboa, P.J.Sebastian, J.Power Source 158(2006)784[2]C.Lin, B.N.Popov, H.J.Ploehn, J.Electrochem, Soc, 149(2002)167.

[2]**DP-5 Wetting Behavior of a Droplet on Dual Rough Surfaces Coated with Hydrophobic and Hydrophilic DLC Films**, ***T. Cha****, J.W. Yi, M.W. Moon (mwmoon@kist.re.kr),* Korea Institute of Science and Technology, Korea*, H.Y. Kim,* Seoul National University, Korea*, K.R. Lee*, Korea Institute of Science and Technology, Korea

Solid surface wettability could be controlled by a chemical treatment of the material surfaces or modification of the surface topology. The chemical treatment of material surfaces causes to change the surface energy, affecting the wetting behavior like contact angle between a water droplet and material surfaces. Furthermore the increase of the surface roughness due to the presence of the micro- or nano-texture amplifies the hydrophobicity or even hydrophilicity of the materials. Here we investigated the effect of dual roughness and chemical composition on both extreme wettabilities of superhydrophobic and superhydrophilic natures. We used the dual rough structures, imitated from the morphologies of lotus leaves by combining the micro- and nano-sacle structures[1] chosen hydrophobic DLC films (HMDSO - hexamethlydisiloxane) and hydrophilic oxygen treatment for extreme wettabilities. On the superhydrophobic surfaces, we observed the high static contact angle and l ow contact angle hysteresis. We then performed the systematic experiment that a water droplet on the surfaces was evaporated slowly and we have monitored the variation in contact angle of the droplet and the transition from Cassie-Baxter state to Wenzel state in high pressure. On the other hand, we observed the static contact angle and spreading velocity of a water droplet on superhydrophilic surfaces. Finally, switchable behaviors of wettabilities between superhydrophobic and superhydrophilic natures were demonstrated with repeat of HMDSO coatings and O2 plasma treatment and various wetting pattern applications were valid by using reversible wetting characteristics.

[1]ar, K. Bewilogua, K. H. Oh, K.-R. Lee, Chem. Phys. Lett. 436 (2007) 199.

DP-6 Structure-Property Relationships of Galvanic Nickel Coatings Codeposited with nanoDiamond particles, ***O. Shenderova*** *(oshenderova@itc-inc.org),* International Technology Center*, D.L. Schulz, R.A. Sailer,* North Datota State University*, G.E. McGuire, W. Mecouch*, International Technology Center

Galvanic nickel coatings have been in use for decades and have many desirable properties including wear and corrosion resistance and relative ease of application. Recently, work has been performed to enhance the tribological properties of nickel-based coatings by incorporation of diamond nanoparticles as a wear-resistant component of this functional coating.1 In this previous study, detonation nanodiamonds (DNDs) with primary particle size 5nm produced by detonation of carbon-containing explosives were used. The polydispersed DNDs with average aggregate size ~150nm were then employed as a hard filler in nickel electroplated films giving Ni-DND composite coatings. It was found that the presence of the DNDs significantly affected the properties of the coatings in an intriguing fashion. Toward that end, Ni-DND coatings exhibited improved microhardness and wear resistance when compared to Ni-only control coatings but the latter showed better erosion resistance.@super 1@ In another series of experiments@super 2@ it was revealed that the type of the steel substrate, its roughness and conditions of electroplating (current density, substrate purification, Ni adhesion layer formation) significantly influence the microhardness and tribological properties of the galvanic coatings. These observations warrant a better understanding of the structure of the Ni-DND coatings and the effect on various mechanical properties. The work presented in this paper relates to an investigation of the structure/tribological properties of Ni-DND composite coatings. Characterization data to be discussed includes wear rates, hardness profiles and composite structure as measured by pin-on-disk, nanoindentation and high resolution SEM test methods.@paragraph2@super 1@I. Petrov, P. Detkov, A. Drovosekov, M.S. Ivanov, T. Tyler, O. Shenderova, N.P. Voznecova, Y.P. Toporova, D. Schulz, Diamond & Related Materials 15 (2006) 2035–2038.

@super 2@R.A. Sailer, O.Shenderova, D.L. Schulz, W.Mecoach, G.McGuire, in preparation

DP-7 Metal-Containing Diamond Like Carbon (DLC:Me) and AlN (AlN:Me) Metallo-Dielectric Nanocomposites, *G.M. Matenoglou (gmatenog@cc.uoi.gr), H. Zoumbos,* University of Ioannina, Greece*, A. Lotsari,* Aristotle University of Thessaloniki, Greece*, Ch.E. Lekka, D.F. Anagnostopoulos,* University of Ioannina, Greece*, Ph. Komninou,* Aristotle University of Thessaloniki, Greece*, G.A. Evangelakis,* ***P. Patsalas***, University of Ioannina, Greece

Pulsed Laser Deposition (PLD) from sectored targets is a versatile technique, which is able of growing nanocomposite films. Of special importance are nanocomposites consisting of a nitride or a carbonaceous dielectric matrix incorporating metal nanoparticles, which are promising materials for various applications in mechanical protection, electronics and plasmonics. In this work we study the structure of PLD-grown, metal-containing diamond like carbon DLC:Me (Me=Ag,Cu,Ti,Zr,W,Mo) and AlN:Me composites (Me=Ag,Cu,Au) vs. various parameters, such as the metal content, nanoparticle size and distribution and the working pressure. The films have the form of a dielectric matrix (DLC or AlN) incorporating metal nanoclusters of 3-10 nm diameter. The density of the a-C and AlN matrices are studied by means of X-Ray and Neutron Reflectivity (XRR/NR). The composition of the films and the crystal structure of the inclusions have been determined by in-situ auger electron spectroscopy and high-resolution transmission electron microscopy, respectively. The optical properties of the films have been determined by optical reflectance spectroscopy. We show that the incorporation of the metal nanoparticles may severely alter the structure and properties of the matrix and a simple rule of mixture does not apply. Thus, we investigate the interactions at the matrix-nanoparticle interface using ab-initio calculations. Finally we demonstrate the tailoring of the optical properties of DLC:Me and AlN:Me nanocomposite films by varying the metal nanoparticles' size and content.

DP-8 Preparation and Characterization of Nb-Based Hard Coatings for Osteosynthesis Purposes, ***M. Braic*** *(mbraic@inoe.inoe.ro), C.N. Zoita, V. Braic,* National Institute for Optoelectronics, Romania*, T. Petreus*, University of Medicine and Pharmacy, Romania

One major task of modern biology and materials science is the design of new biomaterials able to improve the adaptation of artificial implants to living tissues and markedly increase their service life. The complexity of the problem is due to the fact that none of the materials currently used to produce implants possesses all properties of the bone tissue. New biomaterials as TiAlV alloys, were developed and largely used. However, the toxicity of vanadium and the side effects caused by the presence of aluminium ions restrict their use in medicine. In order to prevent the release of toxic Al and V ions, different thin films with barrier effect were deposited on the metallic substrates. The aim of this paper is to present the results obtained in the deposition of the graded NbCN films on TiAlV alloy. The films were deposited by the magnetron sputtering technique in a reactive nitrogen and methane atmosphere, at three deposition temperatures (300°C, 400°C a nd 500°C), using an AJA ORION system and the same substrate bias. Elemental and phase composition, microhardness, adhesion, surface roughness and residual stress as well as the corrosion were investigated. The corrosion tests in artificial physiological solution revealed a higher corrosion resistance of the coated samples, the deposited films representing an efficient diffusion barrier for the ions release from the Ti6Al4V substrates. In order to demonstrate the biocompatibility of the coatings, in vitro tests were carried out, using the direct contact method in a stabilized cellular line (CCl 81 VERO) as medium. The morphology and behaviour of cellular cultures in different growth stages in the presence of different coated samples were analyzed by electron microscopy, their observed cellular activity and cells morphology showing no sign of cytotoxicity effect.

DP-9 Effect of Oxygen Plasma Treatment on Bonding States for Columnar Structured a-CN_x Thin Films Prepared by a Reactive Sputtering, ***M. Aono*** *(aono@nda.ac.jp), S. Kikuchi, N. Kitazawa, Y. Watanabe*, National Defense Academy, Japan

Amorphous carbon nitride (a-CN_x) thin films were deposited on silicon single crystal substrates by rf-reactive sputtering method using a graphite target, and after deposition the films were exposed to oxygen plasma so as to be modified on their surface. Effect of oxygen plasma treatment on bonding structures of the film surface has been studied. A substrate temperature was varied from room temperature (RT) to 853 K. Oxygen gas of about 16 Pa was discharged by rf-power and oxygen plasma was generated. Plasma treatment time was kept at 30 sec. The chemical bonding states and film composition were analyzed by X-ray photoelectron

spectroscopy (XPS) and Fourier transform infrared spectroscopy (FT-IR). The films thickness was calculated from images of scanning electron microscopy (SEM) and ellipsometer. XPS study has revealed the films have NO_2 and NO_3 bonding structures when the films are deposited with a deposition temperature over 673 K. After exposure to oxygen plasma, carbon in the film surface was etched selectively and this phenomenon was observed in all the films. However, NO_3 bonding state was remarkably increased after oxygen plasma treatment for the films deposited at high deposition temperature.

DP-10 DLC Coated Spinal Disks with Predictable Long Term Adhesion, ***R. Hauert*** *(Roland.Hauert@empa.ch), C.V. Falub,* Empa, Switzerland*, G. Thorwarth,* Swiss Federal Institute for Materials Testing and Research (EMPA)*, B. Weisse, U. Müller,* Empa, Switzerland*, M. Parlinska-Wojtan,* Swiss Federal Institute for Materials Testing and Research (EMPA)*, C. Voisard,* Synthes GmbH, Switzerland*, M. Tobler*, IonBond AG, Switzerland

In spinal disk replacement, articulating implants increasingly contribute to restore full spine flexibil-ity. Release of wear debris has been observed with all commercially available metal-on-polyethylene and metal-on-metal implants. Theoretically, over a long term, this may increase the risk of allergic reactions, hypersensitivity, or failure of the device. In view of their extremely low wear, application of Diamond Like Carbon (DLC) coatings to medical implants seems feasible. Yet, the body environment has shown to cause unpredictable DLC layer delaminations, so far barring an adoption for the MedTech field. The key to this problem rests in the design of an interface layer stable in-vivo, which is the aim of this presentation. Interface reaction layers between DLC and CoCrMo implant materials have been analyzed by XPS depth profiling. The presence of an about 6 nanometer thick reactive interface layer consisting of cobalt-, chrome- and molybdenum- carbides formed at the beginning of deposition could be shown. We will show that the interface reaction layer determines the adhesion lifetime of the coating, which, in the case of DLC on CoCrMo, can be predicted. Wear and friction tests have been performed on a newly developed simulator using calf serum as a lubricant. As long as layer adhesion is guaranteed, a wear mass reduction by a factor of 20 is observed with respect to the uncoated implants, whereas no change in the coefficient of friction by the DLC coating was observed.

DP-11 Structural and Optical Properties of Ultrananocrystalline Diamond / InGaAs/GaAs Quantum Dot Structures, ***C. Popov*** *(popov@ina.uni-kassel.de), A. Gushterov, L. Lingys, C. Sippel, J.P. Reithmaier*, University of Kassel, Germany

The combination of the unique properties of ultrananocrystalline diamond (UNCD) films and of semiconductor quantum dot (QD) structures could significantly improve the performance of different electronic and optoelectronic devices, where e.g. good thermal management and advanced mechanical parameters are required. In the current work quantum dot InGaAs/GaAs heterostructures have been grown by molecular beam epitaxy (MBE) with different densities between 1.6 x 10^{10} cm^{-2} and 1.6 x 10^{11} cm^{-2} controlled by the deposition temperature. These structures were overgrown with UNCD by microwave plasma chemical vapor deposition (MWCVD) using methane/nitrogen mixtures. Scanning electron microscopy (SEM) reveals that without ultrasonic pretreatment the diamond nucleation density on QD structures is low and only separate islands of UNCD are deposited, while after pretreatment thin closed films are formed. From the cross-section SEM images a growth rate of ca. 3 nm/min is estimated which is very close to that on silicon at the same deposition conditions. The UNCD coatings exhibit a morphology consisting of two types of structures as shown by atomic force microscopy (AFM). The first one includes nodules with diameters between 180 and 350 nm varying with the density of the underlying QDs; the second is formed by a kind of granular substructure of these nodules with diameters of about 40 nm for all QD densities. The optical properties were investigated by photoluminescence (PL) spectroscopy before and after the deposition of UNCD. The PL signals of QD structures overgrown with UNCD remain almost unchanged with respect to the peak positions and widths, revealing that the UNCD/QD structures retain the optical properties of uncoated InGaAs/GaAs quantum dots.

DP-12 To Diagnose the Characterization of Diamond Films Grown on SiO_2 Layer, ***P-Y. Chen*** *(PLin121@ms.ccafps.khc.edu.tw)*, I-Shou University, Taiwan

Continuous diamond film growth on SiO_2 was achieved by an effective pretreatment. Carbon implantation and diamond powder abrasion were used to enhance diamond nucleation on SiO_2 film. The possibility of using two gas mixtures (CH_4-CO_2 and CH_4-H_2) in order to achieve the growth of continuous diamond films on SiO_2 layer. The diamond film growth rate obtained using the CH_4-CO_2 gas mixture was 0.6 μm/h which is roughly three times higher than that of the CH_4-H_2 gas mixture. CH_4-H_2 gas mixture had a lower etching reaction rate than that of CH_4-CO_2 gas mixture. The higher etching rate of CH_4-CO_2 gas mixture was consider to be due to a higher concentration of active carbon radicals or carbon-containing species in the plasma. Scanning electron microscopy (SEM) and optical emission spectroscopy (OES) indicated that active carbon radicals and carbon-containing species are major cause of pits arising at the surface. Using CH_4-H_2 gas mixture at a low microwave power gave the optimum reaction conditions for depositing diamond films on SiO_2 layer surface.

DP-13 Deposition of Amorphous Carbon-Silver Composites, *O. Garcia-Zarco, Z. Montiel,* ***S.E. Rodil*** *(ser42@iim.unam.mx),* Universidad Nacional Autónoma de México*, M. Camacho-López*, Universidad Autónoma del Estado de México

Composites of Amorphous carbon films and silver were deposited by co-sputtering, where the target (10 cm diameter) was of pure graphite with small inclusion of pure silver (less than 1 cm^2). The films were deposited under different power, from 40 to 250 W, and different target-substrate distances. The substrate was earthed and rotating in order to obtain an uniform distribution of the silver content (less than 10 at%). The addition of the Ag piece into the target increased the deposition rate of the carbon films, which could be related to the higher sputter yield of the silver, but there seem to be also a contribution from a larger emission of secondary electrons from the Ag that enhances the plasma and therefore the sputtering process becomes more efficient.

Scanning electron micrographs acquired using backscattered electrons showed that the silver was segregated from the carbon matrix, forming nanoparticles or larger clusters as the power was in-creased. The X-ray diffraction pattern showed that silver was crystalline and the carbon matrix remains amorphous, although for certain conditions a peak attributed to fullerene-like structures was obtained. Ellipsometric spectroscopy was used to determine the variation in the optical properties as the silver con-tent and distribution was changed; at low power conditions, the Plasmon resonance of the Ag nanoparti-cles dominates the spectra. Finally, we used Raman spectroscopy to understand the bonding characteristics of the carbon-silver composites, finding that there are variations in the D/G ratio, which can be correlated to the observed structure and X-ray diffraction results.

DP-14 Enhancement of Deposition Rate and Droplet Reduction in T-Shape Filtered Arc Deposition System for DLC Preparation, ***M. Kamiya*** *(kamiya@arc.eee.tut.ac.jp), T. Yanagita, H. Tanoue, S. Oke, Y. Suda, H. Takikawa,* Toyohashi University of Technology, Japan*, M. Taki, Y. Hasegawa,* Onward Ceramic Coating Co., Ltd., Japan*, T. Ishikawa,* Hitachi Tool Engineering, Ltd., Japan*, H. Yasui*, Industrial Research Institute of Ishikawa, Japan

T-shape filtered arc deposition system (T-FAD) is powerful tool as a physical vapor deposition (PVD) system for preparing various kinds of diamond-like carbon (DLC) films with high quality, especially hydrogen-free hard DLC, named tetrahedral amorphous carbon (ta-C). Most of macro-particles (droplets) emitted from the graphite cathode are removed from the cathodic arc plasma when the plasma beam passes thorough the T corner of the filter duct. However, further droplet reduction is required.

In the present study, in order to increase the deposition rate and decrease the number of droplets on the film, the electric field was applied in the T-shape droplet-filter duct. The ion current in front of the substrate, deposition rate, and number of droplets on prepared DLC film were measured as a function of the electric field strength. As a result, the ion current had a maximum at the electric field generated by applying the same voltage as the filtered carbon-arc-pl asma potential. The deposition rate increased by approximately 10%, and thus the number of droplets was reduced by same level. Similar experiment was carried out for carbon-argon mixture plasma.

DP-15 Ashing of DLC Film by Oxygen Plasma Beam Converted from Filtered Carbon-Cathodic-Arc, ***H. Tanoue*** *(tanoue@arc.eee.tut.ac.jp), M. Kamiya, S. Oke, Y. Suda, H. Takikawa,* Toyohashi University of Technology, Japan*, M. Taki, Y. Hasegawa,* Onward Ceramic Coating Co., Ltd., Japan*, T. Ishikawa,* Hitachi Tool Engineering, Ltd., Japan*, H. Yasui*, Industrial Research Institute of Ishikawa, Japan

Diamond-like carbon (DLC) film has a great advantage as a protective film to realize the reuse of cutting tools and press molds, since it can be removed from the workpiece by ashing treatment. RF oxygen plasma is usually used for ashing DLC film. However, RF plasma ashing has some problems; the tiny piece of film residue, mirco-arcing at edges of workpieces, difficulty of uniform ashing for workpieces with complex shape or curvature. It is better to use beam-form-plasma to solve such problems. Therefore, in the present study, oxygen plasma beam was generated by converting from the filtered cathodic carbon-arc-plasma and feasibility of the ashing of the DLC film was examined. First, the converted oxygen plasma was analyzed by optical emission spectroscopy. The hydrogen-free DLC films were prepared on superhard alloy substrate by T-shape filtered arc deposition system (T-

FAD). The oxygen plasma beam was irradiated to the DLC film. The biases of uni-polar pulse (repetition frequency, 10 kHz; duty, 20%) and DC were applied to the substrate. The ashing rate of the DLC film and surface roughness of the substrate were measured at various bias conditions. Micro-Raman spectroscopy was used to confirm whether DLC film was removed.

DP-16 Characterization of High sp^3 Diamond-Like Carbon Films Synthesized by Cathodic Arc Activated Deposition Process, ***W.-C. Lin***, *D.-Y. Wang (dywang@mdu.edu.tw)*, Mingdao University, Taiwan

The diamond-like carbon (DLC) film has been widely used in the cutting and forming industries for its superb properties of high hardness, low friction coefficient, high wear resistance, and chemical inertness. In this study, an amorphous hydrogenated carbon film (a-C:H) was synthesized by using a cathodic-arc activated deposition (CAAD) process. The CAAD process consisted of a PVD stage, where a metallic and nitride transition layers were deposited, and a follow-up CVD stage, where the super-hard CrxC/DLC gradient coating was deposited. During the later CVD stage, the energetic metal plasma catalyzed the decomposition of the hydrocarbon gas (C_2H_2) and induced the formation of the final gradient carbon film. In order to maintain the sp^3/sp^2 ratio of the DLC film with specific tribological properties, the deposition parameters have to be regulated through a control mechanism. Results of this study demonstrated that the carbon bond ratio of DLC is closely related to the fl ow ratio of the reactive gas $C_2H_2/(C_2H_2+N2)$. The formation mechanism of metal carbides and DLC during the CAAD process was analyzed by using optical emission spectroscopy, XRD, and ESCA. The tribological tests, electron microscopy, and Raman spectroscopy were employed to characterize the microstructure and carbon bond properties of DLC coatings.

DP-17 On Mechanism of Self-Arrangement of Nanosized Diamond Particles Under Sintering, ***O.O. Bochechka*** *(abochechka@mail.ru), G.S. Oleynik, A.V. Kotko*, National Academy of Sciences, Ukraine

The results of TEM investigation for formation of 50-80 nm single crystals with decahedral and icosahedral faceting produced under high pressure (4-7 GPa) and temperature (1600-2000°C) in the system of 1-7 nm diamond particles of detonation synthesis are discussed. The appearance of such crystals occurs in the absence of diffusion mass transfer and is determined by the self-arrangement process, that is, by the oriented association of the initial diamond nanoparticles. A mechanism of the self-arrangement is proposed, which includes several successive elementary stages such as i) the formation of contacts between particles along morphologically flat facet surfaces; ii) selection of contacting particles and contact planes formed due to the realization of recognition mechanism (this determines the oriented growing together and/or homoepitaxy of independent particles and so the formation of single crystals of regular habitus. The oriented interaction of diamond part icles containing surface layers of non-diamond carbon[1] seems to be possible as it is known that transfer of structural information of a substance can proceed through layers of foreign substances); iii) transformation of non-diamond carbon into diamond. The mechanism of the transformation under the action of one-dimensional periodic potential arising due to the altering of layers of substances with different forbidden energy gaps is possible[2]. A diamond particle with a surface 5-10 Å thick layer of non-diamond carbon is, in fact, a fragment of the superlattice whose constituents differ in the forbidden gap value: it is equal to 3.5 eV for diamond (particle core) and to zero for carbon with sp^2- hybridization.

[1] Aleksenskiy A.Ye., Baidakova M.V., Vul A.Ya., et al. FTT. 2000. T 42. N 8. S.1531-1534.

[2] Zavaritskaja T. N., Karavanskij V. A., Melnik N. N., Pudonin F. A. Pisma v JETF. 2004. T. 79. N 6.S.340-343.

DP-18 Nanoscratch Characterization of Self-Assembled Monolayers using Molecular Dynamics, ***T.-H. Fang*** *(fang.tehua@msa.hinet.net), C.-N. Fang*, National Formosa University, Taiwan

The nanoscratch properties of self-assembled monolayers (SAMs) on an Au surface are investigated by molecular dynamics (MD). This study uses the tight-binding second-moment approximation (TB-SMA) potential to describe the interaction force between Au and Au atoms. Due to the complexity of the structure of SAMs, the coarse grain equivalent scheme is treated as a single-spherical molecule to an equivalent SAMs chain. For the description of the intramolecular interactions in a SAMs chain, bond stretching potential, bending potential, and the torsion potential were adopted. The non-bonded and SAMs on an Au surface was adopted Lennard-Jones potential. The results showed that when the scratch depth of the sample increased was observed during scratching process, the friction force and the normal force were increased. The scratch areas showed break away, and the frictional coefficient can be defined as a value of the friction force and the normal force.

DP-19 A New Globular Nanocarbon Material and Production Fire Method Therof, *V.G. Suschev,* ***V.A. Marchukov*** *(marvalal@mail.ru), V. Y. Dolmatov*, FSUP SCTB Technolog, Russia

A new thermal-oxidative gas-cycle (fire) production method for nanosized porous globules of high-purity carbon (GNC Globular Nanocarbon) by means of combustion of methane in special conditions using chlorine as an oxidizing agent has been developed.

A distinctive feature of this method is the combustion process organization in such a way as to almost all thermal energy of oxidation is consumed to the target process of chemical synthesis of complex carbon structures without effluent of carbonic acid gas and nonutilizable heat to the environment. The method allows to solve a problem of obtaining of uniform structural nanosized carbon particles using controlled high-speed chain chemical reactions proceeding in conditions wide of the equilibrium. General foundations being a basis of the method are guided by a theory of formation of permolecular structures in nonequilibrium systems developed by Professor Aleskovsky V.B. (St.Petersburg, Russia). The method is a way of obtaining of nanosized metal – carbon compounds.

A primary nanomaterial obtained by means of plasma synthesis represents an aggregate of uniform carbon microporous globules with the size of 20-30 nm coated with surface chlorine atoms chemically combined with carbon (nanocarbochloride - GNC-Cl). This material is a progenitor of a new class of nanocarbon products composed of a nanosized porous structural carbon core and a purposefully-formed shell containing functional groups combined with carbon atoms. Joint name of this class of products is GNC–globular nanocarbon.

After reductive and oxidative thermal treatment with hydrogen, methane, water or other special reagents one can obtain high-active forms of high-pure nanocarbon materials (GNC-A) with the characterization specific surface >1000 m2/g, the carbon content >99,5% and practically zero-ash content. GNC-A is characterized by an uniformity of primary nanoglobules of pure carbon and unique combination of high dispersivity of particles with their microporous structure.

The zero-ash content, high carbon basis purity, easiness of modification of surface structure make this new material very promising for all traditional directions of application of carbon nanoproducts. As for potential fields of application of GNC, these are analogous to the well-known ordered carbon structures such as fullerenes, nanotubes, ultradispersed diamonds. But, in comparison with them there is an advantage: after creation of industrial production GNC can be cheaper by a factor of 10 or 100 than above–listed analogues.

DP-20 New Approaches to Industrial Production of Detonation Nanodiamonds, ***V. Y. Dolmatov*** *(alcen@peterstar.ru), V.A. Marchukov, M.V. Marchukov*, FSUP SCTB Technolog, Russia

At present a modern industrial production of detonation synthesis nanodiamonds (DND) is created on the bases of FSUP «Special engineering and design bureau.

The detonation synthesis is based on the technique of explosion of TNT-RDX charge in aqueaous solution of a strong reducing agent. That allowed to redouble the yield of diamond-containing blend (DB) and DND, to decrease the content of incombustible impurities in the DB 2-3 times, to transform a main part of the incombustible impurities into a soluble form of complex salts.

The DB obtained in the form of an aqueous suspension is filtrated from coarse and fine impurities, subjected to magnetic separation and centrifugated to give a dense and readily-removed sediment. Further the moist DB is directed to the chemical purification stage where the DB is added to ~ 60% HNO3, subjected to strong dispersion, filtrated from difficult-to-remove impurities and pumped up to the system of continuous complex contour plug-flow titanium reactors. The process temperature is ~230-240°C, pressure is 8 to 10 Mpa, a stay period of reaction mass in the high temperature zone is 30-40 minutes. In this conditions the DB is purified from non-diamond carbon and incombustible impurities being as complex salt kind. As a result, a number of residuary incombustible impurities in the DND is ~0,07-0,10 wt.%. Such product can be used in any technologies of application. After chemical purification the obtained nitric-acid suspension of DND is released from nitric oxides and washed out from waste HNO_3. Then the weak-acidic suspension of DND is treated with ammonia and again added to the zone of high temperature and pressure to desorb nitric oxides and HNO_3 from internal pores of DND-agglomerates. The formed ammonium salts are decomposed at the high temperature. The obtained re-purified DND are washed out from an excess of ammonia and directed to a thickening stage to give 8-12 % aqueous suspension of pure DND (1st commodity form of DND as an aqueous suspension). In order to obtain a dry ultrafine granular product (2nd commodity form) the DND-suspension is directed to a spray-type drying stage.

DP-21 On Some Characteristics of Germanium Carbide Thin Films Deposited by rf Magnetron Sputtering, ***V. Braic*** *(vbraic@inoe.inoe.ro), C.N. Zoita, L. Braic*, National Institute for Optoelectronics, Romania

Germanium carbide films proved lately to exhibit attractive properties, as a material with low stress, low light absorption and good adhesion on many infrared substrates such as ZnS, Si and Ge, till to an improved wear resistance to severe abrasive environments. In addition, these films exhibit the band gap tunability over a wide range, in conjunction with a high thermostability. Up to now, some optical, electrical and structural properties have already been reported for the $Ge_{1-x}C_x$ films prepared by CVD, ARE, glow discharge and reactive sputtering methods.

The aim of this paper is to study the influence of carbon content on the characteristics of germanium carbide films, obtained by the RF magnetron sputtering method. The films were obtained on Si(001) and quartz substrates at two deposition temperatures (200°C and 700°C) and two RF substrate bias values (100 V and 300 V). Pure C and Ge targets were used, in a deposition atmosphere consisting of a mixture of Ar and CH_4, at different partial pressure ratios. The films were analyzed by quantitative AES and RBS for the elemental composition, XPS for the near-surface microchemical composition, XRD for structural analysis, AFM for surface morphology, mechanical profilometry for thickness and film stress measurements. Optical investigations (UV-Vis-NIR films transmission, FTIR analysis and Raman spectroscopy) and Hall mobility measurements were also carried out. It was found that the films with low Ge content present a reduced crystallinity, the high deposition temperature promote the growth of larger grain, while higher methane partial pressures determine the reduction of the observed grain size. It clearly resulted that the optical and electrical properties are strongly dependant on the crystallographic and chemical composition of the films, so that films with tuned properties can be obtained by a careful selection of the deposition parameters.

DP-22 High Adherence of DLC Films on Iron Based Material, ***V.J. Trava-Airoldi*** *(vladimir@las.inpe.br),* INPE - Instituto Nacional de Pesquisas Espaciais, Brazil*, L.V Santos, L.F. Bonetti,* National Institute for Space Research - INPE, Brazil*, P.A. Radi,* INPE - Instituto Nacional de Pesquisas Espaciais, Brazil*, R.P.C.C. Statutti*, National Institute for Space Research - INPE, Brazil

Since three decades ago diamond-like carbon (DLC) films have attracted considerable interest over due to their high hardness, low friction coefficient, high wear resistance, high thermal conductivity, high elastic modulus, and chemical inertness. More recently the real possibilities of DLC deposition inside of long iron based tube, give to scientific and development areas an special opportunities to solve some problems related to transportation of aggressive liquids on petroleum and other minerals. The major disadvantage of hard DLC film deposition is a relatively low adhesion of these films on iron based substrates. To overcome the low adhesion problems of these films on metallic substrates, different coating concepts have been proposed, such as thin metal interlayer, surface implantation, multilayer coating, variation of the self-bias voltage, and the use of surface thermal treatments. In this work, an enhanced process of plasma carbonitriding has been hardly studied in order to prepare the iron based material to be coated. A convenient gradient of carbon concentration in the bulk of the substrate as a function of the pressure, gases component and substrate temperature represent the most important part of this study in order to get best adherence. Also, an enhanced asymmetrical bipolar PECVD DC pulsed power supply system has been used. A good quality of DLC coatings with low friction coefficients, low total stress, high degree of hardness, good adherence, and high uniformity at a reasonable growth rate were deposited. The quality characterization and trybologic/mechanical properties were carried out by using Raman scattering spectroscopy and a multi function trybometer system, respectively.

DP-23 Diamond Thin Films: an Insight on the Growth Mechanism, ***J.C. Madaleno*** *(jcmadaleno@ua.pt), S.Z. Rotter, J. Gracio*, University of Aveiro, Portugal

During the last decades, polycrystalline diamond films have been successfully deposited by chemical vapor deposition (CVD) on a wide variety of foreign substrates. The deposition method includes some kind of seeding procedure (whether it may be mechanical scratching, ultrasonic treatment or bias-enhanced nucleation), followed by the diamond deposition in appropriate systems. This nucleation procedure enriches the substrate surface with diamond seeds that start to grow when the substrate is exposed to the proper growth conditions, and eventually coalesce, closing the diamond film. The film starts to thicken, and diamond deposition follows a columnar growth mode.

A novel nucleation process (NNP) has been proposed by Rotter . This method induces the formation of an optimal nucleation film (which includes the right seed density and a special carbon film, formed prior to the seeding) that plays a very important role in the growth of the diamond layer at the very first steps of the diamond formation. The growth takes place in a lateral direction until the initial grains coalesce. Eventually this lateral growth mode is replaced by the standard columnar growth mode. This method has been used to coat successfully a wide variety of substrates with complex 3-D shapes. In some cases, a clear influence of the substrate can be seen in an early stage of growth. In this paper, we will characterize thin diamond films growth by NNP on different substrates by Scanning Electron Microscopy, Raman spectroscopy and X-Ray Diffraction, and discuss the role of the initial carbon film deposited on the substrate's surface, prior to the seeding procedure. Exciting data that can provide an insight to the real growth mechanisms involved in diamond will be presented. Finally a new model for the chemical vapor deposition of diamond thin films will be proposed and discussed.

This method is expected to open new ways for using diamond thin films as an encapsulation layer of various small structures, for thermal management applications, for tribology, etc. In addition, it makes diamond deposition possible on a variety of unsuspected abstracts. Note: Requested an Oral Session.

DP-24 Development of Si-Doped Hydrogenated Amorphous Diamond-Like Carbon (a-C:H:Si) for Tribological Applications in Humid Environment, ***H.-R. Stock*** *(stock@iwt-bremen.de), B. Hilker*, Stiftung Institut fuer Werkstofftechnik, Germany

Due to their excellent tribological properties, such as low friction and high wear resistance, amorphous hydrogenated carbon (a-C:H) films are known as a solid lubricant. These a-C:H films were doped with silicon (a-C:H:Si) to expand the possible applications of the a-C:H:films. Silicon is known for reducing the internal stress and increasing the thermal stability. We also investigate the influence of different friction partners on wear and friction mechanism. For depositing the films onto polished steel discs we used dc pulsed magnetron sputtering (Cemecon CC800/9). A transition layer was created to improve adhesion by decreasing the voltage of a chromium target, using nitrogen gas flow and increase the voltage of a graphite target. The final a-C:H:Si layer were deposited by sputtering from graphite targets with silicon inserts and reactive deposition from acetylene. The resulting films have a coating thickness of 2 µm and show good adhesion. By varying different pa rameters like bias voltage and acetylene flow as well as modify the amount of silicon content in the top layer we investigate the friction coefficients in humid environment. The tribological properties were analyzed by a pin-on-disc test in water. Under an applied load of 20 N the coated samples were slid against pins of different materials. This materials were for example 100Cr6, a-C:H:Si or CrN_x. The a-C:H:Si films showed low friction coefficients in water against a-C:H:Si coated 100Cr6 pins of $\mu = 0.06$. These tribological tests illustrate the potential of Si doped a-C:H films to effectively reduce friction and wear in humid environment.

DP-25 Annealing Effect on the Structural, Mechanical and Electrical Properties of Titanium-Doped Diamond-Like Carbon Thin Films, ***Y.-H. Lin*** *(d9531810@oz.nthu.edu.tw), H.-D. Lin, C.-K. Liu,* National Tsing Hua University, Taiwan*, Y.-C. Chen,* National Chung Hsing University, Taiwan*, Y.-S. Chang, H.-C. Shih*, National Tsing Hua University, Taiwan

Abstract Diamond-like carbon (DLC) films are a form of amorphous carbon containing a significant fraction of sp3 bonding, which are well known for their superior physical and chemical properties of high hardness, chemical inertness, optical transparency, thermal conductivity and biocompatibility.[1,2] Recently, metal-doped DLC films have stimulated great interests and been widely investigated owing to their extraordinary microstructure of nanocrystalline metal carbide precipitated in the amorphous carbon matrix.super 3,4 In this study, titanium-doped diamond-like carbon (Ti-doped DLC) thin films with Ti compositions of 1.1 at. % were synthesized on a Si substrate by a process combining filtered cathodic vacuum arc (FCVA) and metal vacuum arc (MeVVA) system. This study discusses on the effect of annealing temperature on the microstructure, surface roughness, hardness and electrical resistivity of the resulting films. The Raman spectra reveal that the degree of graphitizatio n of Ti-doped DLC thin films increases with increasing annealing temperature from 100 to 600 °C. Because the degree of graphization of the thin films increases, the hardness of the thin films improved, and the electrical resistivity of the Ti-doped DLC thin films decreases from 0.038 to 0.006 Ω cm.

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Tribology and Mechanical Behavior of Coatings and Thin Films
Room: Town & Country - Session EP

Symposium E Poster Session

EP-1 The Oliver and Pharr Method for Coatings and Physical Scratch Test Analysis for Layered Materials, ***M. Fuchs*** *(info@siomec.de), N. Schwarzer*, Saxonian Institute of Surface Mechanics, Germany

In the work it will be presented how the classical Oliver&Pharr method, widely used for the analysis of indentation experiments, has to be extended for layered materials. Thereby a completely analytical mathematical approach[1] is applied, not only evaluating the substrate effect and correcting it, but also allowing the extraction of the yield strength of the coating material in addition to its Young's modulus.

In the second part it will be presented how the extended Hertzian theory[1], can be applied to scratch tests on layered materials, in order to extract more physical information from the latter. By taking into account, that with lateral forces additional boundary effects like indenter tilting, mixed loads of sticking-sliding areas are coming into play a much more profound simulation and modelling of the classical scratch test is possible. The methods also work perfectly for multilayer and gradient coatings and in principle allows the extraction of the mechanical parameters of any of the constituents of even very complex coating structures. Especially with a new multi-path scratch method combined with additional classical nanoindentation measurements one can benefit from such a more comprehensive and sophisticated analysis. So, it will be shown how the true initial failure can be detected and clearly defined critical stress values can be assigned to the coating material.

The new methods will be demonstrated on a variety of examples in combination with a special new analysing module woven into the software package FilmDoctor[2].

[1]N. Schwarzer: "The extended Hertzian theory and its uses in analysing indentation experiments", Phil. Mag. 86(33-35) 21 Nov - 11 Dec 2006 5153 - 5767

[2]FilmDoctor: Special version for "next generation surface tester analysis", www.siomec.de/downloads/FilmDoctor

EP-2 The Stucture and Properties of Nano-Crystalline ZrTiN Thin Films : Effect of Ratio of Ti/Zr, ***Y.-W. Lin*** *(james722@itrc.org.tw), J.H. Huang, G.P. Yu*, National Tsing Hua University, Taiwan

ZrTiN thin films were deposited by reactive magnetron sputtering based on our previous optimum coating conditions (substrate bias, substrate temperature, system pressure, nitrogen flow etc.) for TiN and ZrN thin films. The ratios of Ti/Zr were tuned by changing target power of Ti and Zr. This study investigated the effect of ratio of Ti/Zr on microstructure and mechanical properties of thin films. With tailoring target power of Ti and Zr to find out the optimum processing parameters, the observed stable nano-crystalline structure and chemically inert characteristic of ZrTiN were the dominant features for the successful demonstration of good properties of thin ZrTiN films. In terms of phase formation, three types of coatings were considered: (a) a ZrTiN single-phase solid solution, (b) ZrTiN interlacing nucleus of TiN or ZrN in the matrix, and (c) nanocomposite ZrTiN films including a significant fraction of TiN or ZrN phase. The films were characterized using X-ray diff raction (XRD), transmission electron microscopy (TEM) and nano-indention. The hardness of ZrTiN film was 37 GPa, and a strong inclination to ZrTiN (111) preferred orientation was observed, when the ratio of Ti/Zr were 1. Furthermore, the target power were 400 Watts and 200 Watts for Ti and Zr, the ZrTiN thin film have been observed to find the X-ray diffraction patterns of ZrTiN (111) and TiN (022), and the film's hardness was 26 GPa. The thickness of ZrTiN films measured by scanning electron microscope (SEM) was greater than 900 nm. Atomic force microscopy (AFM) was used to measure the surface roughness. The ratio of N/Ti/Zr and composition of ZrTiN thin film were analyzed by X-ray Photoelectron Spectroscopy (XPS) and Rutherford Backscattering Spectrometer (RBS).

EP-3 Effect of Surface Polishing on the Abrasion Performance of a Chromium Carbide-Based Coating Deposited by VPS, ***Z. Marcano*** *(zina.marcanocordova@gmail.com),* Universidad Central de Venezuela*, J. Lesage,* University of Lille, France*, E.S. Puchi-Cabrera, M.H. Staia*, Universidad Central de Venezuela

Thermal sprayed chromium carbide-based coatings are being widely used for a variety of wear resistance applications involving sliding, abrasion and erosion over a wide range of temperatures up to 900°C. The thermal sprayed carbide coatings are in general surface finished by machining or grinding after the coating process. Therefore, it is imperative to understand the influence of the finishing operation on the characteristics and performance of carbide coatings. In the present study, low stress abrasion wear resistance is compared for 6 test conditions: three disc rotation speeds and two surface finishes (as-sprayed and polished coatings). The abrasion tests were carried out using a three-body solid particle rubber wheel test rig using silica grits as the abrasive medium. A commercially available $Cr_{20}Ni_{9.5}C$ coating powder (1376T, Praxair) was deposited industrially by means of a vacuum plasma spraying (VPS) system. The incremental volume wear loss of the coating deter mined as a function of time shows that lower values were obtained for the polished coatings in comparison with the as-sprayed Cr_3C_2-NiCr coatings. The results also prove that the abrasive wear volume increases with the rotation speed of the disc. The SEM study of the worn surfaces revealed that the wear was mainly due to the selective removal of the binder as consequence of both plastic deformation and fatigue, due to the repeated action of the abrasive particles followed by the undermining of the carbide particles, resulting in their eventual pullout. Some evidence of microcutting could also be noticed, indicating the removal of the binder phase by this mechanism. Also, very little carbide grain fracture and material removal by delamination were observed.

EP-4 Structural and Mechanical Properties of Graded and Multilayered $Al_xTi_{1-x}N$/CrN Coatings Synthesized by a Cathodic-Arc Deposition Process, ***Y.-Y. Chang*** *(yinyu@mail2000.com.tw), C.-P. Chang, C.-Y. Hsiao, D.-Y. Wang*, Mingdao University, Taiwan

Graded $Al_xTi_{1-x}N$ and multilayered $Al_xTi_{1-x}N$/CrN coatings were synthesized by cathodic-arc evaporation with plasma enhanced duct equipment. Chromium and TiAl alloy cathodes were used for the deposition of $Al_xTi_{1-x}N$/CrN coatings. During the coating process of graded $Al_xTi_{1-x}N$, an $Al_{0.63}Ti_{0.37}N$ top layer was deposited on an interlayer of $Al_xTi_{1-x}N$/CrN, which was obtained by regulation of cathode power. With different cathode current ratios ($Al_{0.67}Ti_{0.33}N$/Cr) of 0.75, 1.0, and 1.25, the deposited multilayered $Al_xTi_{1-x}N$/CrN coatings possessed different chemical contents and periodic thicknesses. The nanolayer thickness and alloy content of the deposited coating were correlated with the emission rate of alloy cathode materials. In this study, field emission scanning electron microscope (FESEM), and X-ray diffraction using Bragg-Brentano and glancing angle parallel beam geometries were used to characterize the microstructure and the residual stress of the deposited films. High resolution transmission electron microscope (HRTEM) and scanning transmission electron microscope (STEM) were used for nanolayered structure analyses of the multilayered $Al_xTi1-xN$/CrN coatings. The composition of deposited graded $Al_xTi_{1-x}N$ and multilayered $Al_xTi_{1-x}N$/CrN coatings were evaluated by a wavelength dispersive X-ray spectrometer (WDS). Hardness, Young's modulus and fracture toughness of the deposited coatings were determined by nano-indentation and Vickers indentation methods. The effect of alloy content (Al, Ti, and Cr) on the microstructure and mechanical properties of $Al_xTi_{1-x}N$/CrN coatings were studied.

EP-5 A Study of the Reciprocating Sliding Wear Performance of Plasma Surface Treated Titanium Alloy, ***G. Cassar*** *(mtp06gc@sheffield.ac.uk),* University of Sheffield, United Kingdom*, J.C.A. Batista-Wilson, S. Banfield,* Tecvac Ltd*, J. Housden,* Tecvac Ltd.*, A. Leyland, A. Matthews*, University of Sheffield, United Kingdom

The use of accelerated wear tests, particularly on laboratory test machinery, is extremely popular within the surface engineering research community. The level of success of novel tribological coatings and treatments is often assessed by one or more of the following techniques: pin-on-disk, rubber-wheel abrasion, block-on-ring, micro-abrasion (free and fixed ball), ball-on-plate impact and reciprocating-sliding wear tests. Many of the available test devices and methodologies are described in the international or national standards such as ISO, DIN or ASTM. However, many of these techniques owe their origin primarily to the characterisation of bulk materials with relative uniform hardness and elastic properties and may therefore be less applicable to functionally-graded and surface-engineered materials, particularly in the case of materials with intrinsically poor tribo-mechanical properties. Linearly reciprocating sliding wear testing of duplex treated Ti-6Al-4V alloys was carried out in this study. Wear volumes were correlated to changes in friction force (and the resultant measured coefficient of friction) which are often indicative of breakdown of the surface treatment. Surface micro profilometry and both optical- and electron- microscopy were used to characterize the wear scars produced. The analysis of test parameter variability (depending on the different surface treatment processes investigated) is presented. The results obtained suggest that the use of total material volume lost (after a pre-set period), of time in order to obtain average wear rates, may be incompatible with the tribo-behaviour of (particularly) duplex-treated substrates. The results obtained here indicate that, in order to differentiate and assess the relative improvements (or lack of) in wear behaviour attained by using surface treatments it is insufficient to compare linear wear rates alone. A clear comparison is only possible if testing is carried out in steps of increasing sliding distance, until the

treated/deposited layers have been completely removed. Also, it is shown that the number of repeated tests necessary (for a given treatment and chosen test condition) to evaluate clearly the treatment and/or coating can vary substantially depending on the observed test progression; typically this necessitates the greatest number of repeats around the point of wear at which the ball counter-face contact area is in transition from the treated layers to the bulk.

EP-6 In-Situ Raman Tribo-Spectrometry Technique for High Temperature Sliding Contacts, ***J. Bultman*** *(john.bultman@wpafb.af.mil),* UDRI/Air Force Research Laboratory, *C. Muratore,* Air Force Research Laboratory/UTC, Inc., *A. Safriet,* UDRI/Air Force Research Laboratory, *A.A. Voevodin*, Air Force Research Laboratory

An in situ Raman tribo-spectrometer was designed and constructed for use during high-temperature wear tests with ambient temperatures of up to 800°C. The instrument was used to determine the oxidation thresholds for common solid lubricants such as MoS_2 and WS_2, and to identify the reaction pathways in novel, temperature-adaptive coating materials that rely on catalysis and/or tribo-oxidation to provide low friction contact interfaces throughout a broad temperature range. The probe was also useful for identifying wear mechanisms in multilayered coatings. Finally, the system was compatible with smart tribological coatings for use at high temperature, which use rare earth-doped diffusion barrier layers to produce a characteristic spectral signature when illuminated by laser light to indicate the extent to which the coatings have been worn while in use. The key element of the Raman system was a remote, high-temperature probe with temperature-resistant focus ing and collection optics consisting of two quartz lenses coupled to a commercial Raman spectrometer via fiber optics. The probe allowed focusing of the laser light on the wear track and collection of scattered light from the surface with the same optics, which were air-cooled to reduce aberrations (mirage effects) due to extreme heating. The probe was suitable for use in a variety of wear test geometries, or for use in actual applications in addition the pin-on-disc tests that were the focus of the current work. Advantages and limitations of the technique with examples of its use for MoS_2, WS_2, chameleon YSZ-MoS_2-Ag, and smart Er and Sm doped YSZ/MoS_2 coatings are provided.

EP-7 Tribological Properties of Cr Ceramic Films With W-C-N Coating by DC Reactive Magnetron Sputtering, ***C.W. Chu*** *(chuhose@mail.mirdc.org.tw), J.S.C. Jang, H.W. Chen*, University of I-Shou, Taiwan

This paper discussed the applications of Cr,Cr2N and CrWCN thin films on the PCB（printed circuit board）drill at high rpm (400krpm).CrWCN is a prominent material for protective coatings due to its excellent oxidation and adhesive resistance properites. Experimental results of drill sample:UC ψ0.25*4 are shown as follows: the hardness of Cr2N is 15Gpa,the frictional coefficient is 0.5.When W is doped on Cr2N,the hardness increased to 15GPa and the frictional coefficient decreased to 0.3.When C2H2 gases pass through, the hardness increased to 25GPa and the frictional coefficient decreased from original 0.3 to below 0.2.When the three thin films applied on the micro drill of the 24,000 PCB holes,CrWCN thin film shows the excellent properties and the wear resistance and contact angle are better than the other two thin films.It will have the minimum flank wear and tool life is 4-5 times longer than the uncoated samples.

EP-8 Influence of the Superficial Roughness on Cr-N Coating Hardness Values Measured at Low Indentation Depths, *R.D. Mancosu,* ***C. Godoy***, *S. Goulart-Santos (sgoularts@gmail.com), T. Ornelas*, Universidade Federal de Minas Gerais, Brazil

Thin films are used in industry to modify mechanical characteristics of materials surface. As these coatings have very small thickness, in the order of micrometers, new techniques to measure mechanical resistance are required. The ISO 14577-4 Standard establishes criteria for thin films hardness measurements using instrumented indentation test. According to this Standard the indentation depth should be smaller than 50 % of the depth of the contact of the indenter with the test piece at maximum force (hc). Another methodology, described by Fischer-Cripps, indicates that the indentation depth should not exceed 10 % of the coating thickness. As the indentation depths allowed are very strict, the influence of surface roughness becomes very important during the coating hardness determination. In this work we have studied the restrictions of instrumented hardness measurements in Cr-N coatings deposited on nitrided and non-nitrided AISI 1045 steel. Changes on thickness and rou ghness were produced for this study. We have analyzed the coatings hardness for 3 different thicknesses (3, 5 and 15 μm) with the same roughness, Sa = 0,090 μm. When the ISO standard criterion is applied, the coating hardness values have been influenced by the substrate hardness. When the Fischer-Cripps criterion was applied, we observed that concluded it was very restrictive for our systems and its utilization can difficult the analysis when the roughness is high. In a second analysis, we measured the coatings hardness with 2 different roughnesses, and thickness equal to 5 μm. In this case, Cr-N coating was deposited on a mirror polished surface and another previously nitrided. The ISO Standard establishes that the indentation depth should be higher than 20 Ra. This criterion forces the penetration to be very large, 1,4 μm for the polished surface, and 2,2 μm for nitrided one. For that reason the penetration should be higher than 25 % coating thickness, for th e flatter surface, and 45 % for the rougher surface. Those depth values are higher than that indicated for Fisher-Cripps and near to the ISO Standard methodology. We concluded: i) using ISO Standard criteria the coating hardness had been influenced by the substrate hardness and ii) using Fisher-Cripps criterion with ISO Standard roughness criterion it is impossible to determine the hardness.

EP-9 Solid Particle Erosion Performance of HVOF WC-Co and WC-Co-Cr Coatings Deposited onto SAE 1045 Steel, ***Y.Y. Santana*** *(yucelys.santana@ucv.ve), J.G. La Barbera-Sosa, A. Bencomo, E.S. Puchi-Cabrera, M.H. Staia*, Universidad Central de Venezuela

Thermally sprayed cermet coatings have emerged as a viable solution for a wide range of wear resistance applications. The tungsten carbide coating is frequently used for many applications related to systems such as gas and steam turbines, aero engines, etc. in order to improve the service life of their components. Solid particle erosion under extreme conditions, i.e. high hardness and strength of abrasives, high velocity and pressure, etc. is a serious problem for the industrial equipment. The main objective of this paper is to study the solid particle erosion behavior of both WC-12Co and WC-10Co-4Cr coatings deposited by HVOF thermal spray technique. A detailed microstructural and mechanical characterization study of these coatings was carried out in order to relate their morphology, roughness, hardness, fracture toughness and residual stresses to their tribological performance. X-ray diffraction techniques were used to determine the surface residual stresses of the co atings, whereas the incremental hole drilling technique allowed the analysis of the non-uniform through-thickness residual stresses present in the coatings. Dry erosion tests were carried out at 30° and 90° angle of impingement, respectively, using as erodent SiC particles with ~ 60 μm of diameter, at a velocity of 83.4 m/s and a feed rate of 2 g/min, according to the ASTM G-76 standard. The eroded surfaces were examined so that the erosion mechanisms of the coatings could be determined. It was found that the erosion rate for both coated systems was higher when the erosion test was carried out at an angle of 90°, leading to a brittle wear type mechanism. The results also indicate that, for the experimental conditions carried out in the present study, the WC-10Co-4Cr coating exhibited a higher erosive wear resistance as consequence of a smaller W_2C brittle phase content and a higher through-thickness residual compressive stresses values, wh e n compared to the WC-12Co coating.

EP-10 Nano-Laminated a-C:H Coating on SKD-Tools for Dry Micro-Stamping, ***T. Aizawa*** *(aizawa@asiaseed.org),* AsiaSEEd-Institute, Japan, *E. Iwamura,* Arakawa Chemical Co. Ltd., Japan, *K. Itoh*, Seki Corporation Co., Japan

In parallel with increase of market demand for tiny, fine electrical parts, needs for desk-top micro stamping is much increased. These electrical parts dislike any residues including the lubricating oils since they often become a cause of nuisance for contamination. Various types of coated tools are developed to make dry micro-stamping; dry micro-stamping with severe clearance between work-sheet and tools is never easy task to be solved by conventional mono-layered coating. On the basis of successful application of nano-laminated DLC coating to WC (Co) tools, this technique is further developed to protective coating of SKD-steel tools in dry micro-stamping. Through nano-indentation test, the effect of bi-layer thickness and sublayer thickness ratio on the mechanical properties is discussed to optimize the coating conditions for protective coating of SKD-tools. In second, XRR (X-ray reflection) method is used to define the average density of nano-laminated a-C:H coating as well as its distribution in the multi-layered alignment as a trial to make reliability analysis of nano-laminated coating system. Finally, the micro-stamping test is performed to investigate the endurance of nano-laminated a-C:H coated SKD tools in the bending and ironing step of progressive stamping. Successful dry micro-stamping up to 10, 000 time continuous steps directly proves that this nano-laminated a-C:H coating should be effective to be working as a reliable, protective coating in practice.

EP-11 A Critical Analysis of the Use of Nanohardness as a Method to Evaluate Wear Resistance of Composite Materials, *C. Godoy (gcgodoy@uaigiga.com.br), R.D. Mancosu,* ***S. Goulart-Santos***, *E. Viera Jr.*, Universidade Federal de Minas Gerais, Brazil

It is known that the material hardness is function of the measurement method. In the static indentation tests, the measure of hardness is given by load per unit area of impression. The hardness is associated to an image and the residual plastic impression in the specimen is a function of the applied

load. In nanoindentation testing, the depth of penetration beneath the specimen surface is measured while the load is applied. The area of contact size is determined from the geometry of the indenter. For indentation depth < 6 μm, an exact area function for a given indenter is required. In this case, the determination of material hardness became more a mathematical analysis procedure than a specific experimental procedure. ISO 14577 Standard defines nanohardness as the hardness measured where the total indentation depth (h) is ≤ 0.2 μm. In nanotechnology time, this hardness is fundamental to evaluate the mechanical resistance in such low depth. Therefore, it must be used with rigorous analysis. In composite materials, the proprieties can differ with the depth. In duplex treatments, plasma nitriding followed by coating deposition, we obtain such kind composite material. In the Archard's equation the wear rate (Q) depends on the value of hardness of the softer material, in this study this material was a Cr-Al-N coated/nitrided AISI 4140. A constant wear rate cannot be expected during the wear process because the hardness modifies with the depth. The density also modifies because the density of coating and substrate are different. Thus, it is not indicated to determine the worn volume using the mass loss during the wear process. On the other hand, the use of nanohardness implies the assumption that only the superficial mechanical resistance has a function in wear resistance. Which hardness we must use? In this work, the worn volume was determined by 3D profilometry for four systems: uncoated, nitrided, Cr-Al-N coated and nitrided/Cr-Al-N coated AISI 4140 steel. The smaller worn volume was observed on nitrided/Cr-Al-N coated steel, confirming the importance of a deep hardening for wear resistance. Nanohardness measurements were not able to indicate this result. The determination of the dimensional wear coefficient, k, was also very important, because of its independence with the measure of the hardness. The nitrided/Cr-Al-N coated steel with higher macrohardness presented the lower value of k.

EP-12 Impact Properties of TiN, TiAlN and TiSiN PVD Coatings at Ambient and Elevated Temperatures, *K.-D. Bouzakis (bouzakis@eng.auth.gr), M. Pappa, S. Gerardis, G. Katirtzoglou, S. Makrimallakis, G. Skordaris,* Aristoteles University of Thessaloniki, Greece, ***R. M'Saoubi***, Seco Tools AB

The impact behavior of TiN, TiAlN and TiSiN coated cemented carbide inserts at ambient and elevated temperatures was investigated. Nanoindentations were conducted, supported by FEM algorithms to determine the stress-strain constitutive laws of the examined films. Moreover perpendicular impact tests at various temperatures were carried out. The impact imprints were evaluated with the aid of scanning electron microscopy graphs and a developed algorithm to determine the coating failed area ratio. Thus the change of the impact behaviour of the examined coatings at various temperatures was established. The experimental results indicate a non-linear PVD film wear resistance versus the applied temperature. By FEM-based simulation of this procedure, taking into account the test conditions and duration up to the film damage initiation, the critical stresses associated with the coating fatigue strength were calculated. According to the obtained results, the TiAlN film possesses a n enhanced impact resistance compared to the TiN coating. Moreover, TiSiN coatings show a significantly improved capability to withstand effectively the applied loads, compared to TiAlN films, even at elevated temperatures during the impact test.

EP-13 Production of a Multicomponential Layers on a 99.8% Purity Iron by the Two State Boro-Nitriding Process: Microstructural and Mechanical Characterization, ***I. Campos*** *(icampos@ipn.mx), O.A. Gómez-Vargas,* Instituto Politécnico Nacional, México, *U. Figueroa-López,* Tecnológico de Monterrey, México, *M. Ortiz-Domínguez*, Instituto Politécnico Nacional, México

Nowadays, there are a number of termochemical processes for improving surface mechanical properties. Boriding have been positioning as a one of the processes which report high performance in the improvement of mechanical properties (hardness and wear strength) as well as corrosion resistance on several alloy systems; i.e. ferrous and non ferrous alloys@super 1@,@super 2@. It has been seen that paste boriding process can be used in high production lines in comparison with the powder-pack boriding@super 2@.

On the other hand, surface treatment by nitriding can produce a set of high surface hardness and good wear resistance@super 2@. However, it is not recommended to apply this treatment in some high carbon steels because the carbon tends to diffuse at the surface of the sample, and the nitride layer formed on the substrate leads to flaking and spalling when a mechanical load is applied.

This study analyzed the production of multicomponential boro-nitriding layers at the surface of 99.8% high purity iron@super 3@ by two stage process consisting of paste boriding step followed by a powder nitriding process. Characterization of the boro-nitriding samples were made by Knoop microhardness testing, Energy Dispersive Spectroscopy (EDS), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM).

The diffusion of boron and nitrogen in the two stage process depends of several factors, although in the boriding stage boron potential seems sensible to the preparation of boriding paste as well as the particle size. Additionally, the preparation of the sample before and after of boriding stage plays an important role because is possible to minimize surface imperfections and unnecessary compound on the surface after boriding stage. The product consist of a well define Fe@sub x@B@sub y@/B@sub x@N@sub y@/diffusion zone multicomponential layers which seems as a interesting system where a combination of wear and corrosion strength are main properties in service.

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@super 2@I. Campos, G. Ram@aa i@rez, U. Figueroa, J. Mart@aa i@nez, O. Morales Appl. Surf. Sci. 253 (2007) 3469-3475. @paragraph2@super 3@Fan-Shiong Chen, Kuo-Liang Wang. Appl. Surf. Sci. 115 (1999) 239-248.

EP-14 Effect of Spraying Distance on the Microstructure and Mechanical Properties of a Colmonoy 88 Alloy deposited by HVOF Thermal Spraying, ***J.G. La Barbera-Sosa*** *(jose.labarbera@ucv.ve), Y.Y. Santana,* Universidad Central de Venezuela, *N. Cuadrado, J. Caro,* CTM Centre Tecnològic, Spain, *P.O. Renault, E. Le Bourhis,* Université de Poitiers, France, *M.H. Staia, E.S. Puchi-Cabrera*, Universidad Central de Venezuela

The present work has been conducted in order to determine systematically the influence of the spraying distance on the microstructure and mechanical properties of a Colmonoy 88 alloy deposited by means of HVOF thermal spraying onto a SAE 1045 steel substrate. The spraying distance varied between 380-470 mm and the evaluation of the deposits characteristics and properties was carried out both on their surface and on cross section. The microstructural study was conducted by means of SEM and image analysis techniques, which allowed the characterization of the unmelted particles volume fraction and the apparent porosity of the coatings. Also, X-ray diffraction techniques were used to determine the surface residual stresses of the coatings employing the $\sin^2\psi$ method. For this purpose, an analysis of the (311) plane of the dominant Ni phase, at an angle of $2\theta \sim 93.2^{\circ}$, was conducted. On the other hand, both the hardness and elastic modulus of the coati ngs were determined according to the analysis earlier advanced by Oliver and Pharr (1992; 2004), employing the indentation load versus penetration depth curves derived from instrumented indentation tests conducted with a Berkovich indenter. The yield strength of the coatings was also estimated from the above curves following the methodology suggested by Zeng and Chiu (2001), as well as from spherical indentation tests and the Hertz equations commonly used in contact mechanics. The microstructural analysis indicated a significant increase in the unmelted volume fraction as the spraying distance increases. Such particles were found to give rise to a decrease in the lamellas cohesion and the development of interlamellar microcracks, leading to a decrease in the elastic modulus of the coatings. Both hardness and elastic modulus showed an anisotropic behavior. Such properties were found to be higher on the cross section of the coating than on the deposition plane. A satisfactory c omparison between the predicted and experimental values of the coatings yield strength was observed for all the conditions investigated.

EP-15 Effect of Damage Accumulation in Co-Depletion Layer on the Fatigue Debonding Behavior of Diamond Coated WC-Co, ***S. Kamiya*** *(kamiya.shoji@nitech.ac.jp), A. Ueda,* Nagoya Institute of Technology, Japan, *H. Hanyu,* OSG Corporation, Japan, *J.C. Madaleno, J. Gracio*, University of Aveiro, Portugal

Cobalt-cemented tungsten carbide (WC-Co) coated with chemically vapor deposited (CVD) diamond is one of the most common styles of diamond tools. This technique allowed the realization of precise and complex tool geometries with the extreme wear resistance against severely abrasive materials. The most serious drawback of such tools could be sudden debonding of diamond coating after a certain period of use in machining even without any trace of wear, which causes unacceptable damage to the workpieces. However, no clear observation on the mechanism of such delayed debonding was reported at this moment. Therefore there is no efficient method to predict the lifetime before such sudden debonding of diamond coatings.

In the previous studies, we surveyed fatigue debonding behavior of CVD diamond films deposited both on silicon[1] and WC-Co[2] substrates and subjected to repeated mechanical loading. In the former case, fatigue cracks were found on the substrate surface whenever debonding was observed. In both cases, stress-fatigue lifetime before debonding (S-N diagram) appeared to be independent of film thickness when appropriate stress components were plotted to represent the state of damage on the substrate surface.

Therefore the attention in this study is newly focused on damage accumulation in the Co-depletion layer on the WC-Co substrate surface, which has to be introduced for a successful diamond deposition and should be the most susceptible to fatigue. The elastic plastic properties of Co-depletion layer are surveyed by indentation on the bare substrate surface. Fatigue damage of substrate surface under repeated indentation loading is also investigated and correlated to the debonding behavior of diamond coatings with different film thicknesses. Finally, possibilities for fatigue lifetime prediction on the basis of substrate surface damage accumulation process and optimization of Co-depletion layer for a longer fatigue lifetime is discussed.

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EP-16 Effect of Microstructure on the Mechanical Properties of In-Situ Deposited Carbon Monolayer on the Si(100) at High Temperature Under Ultra High Vacuum, *C.K. Chung (ckchung@mail.ncku.edu.tw),* ***S.T. Hung****, C.W. Lai*, National Cheng Kung University, Taiwan

In this article, the effect of substrate temperature on mechanical property of carbon nanofilms using nanoindentation in dynamic contact measurement mode (DCM) was investigated. The carbon monolayer was deposited on the Si (100) substrate at room temperature and at high temperature of 400 to 700°C by ultra high vacuum ion beam sputtering (UHV IBS). Raman spectroscopy was utilized to characterize bonding behavior of carbon monolayer for the variation of graphite peak (G-peak) and disorder-induced peak (D-peak). The amorphous C microstructure is stable up to 500°C. However, ID/IG ratio increases with increasing substrate temperature from RT to 500 °C due to graphitization effect for the increases sp^2 bonds. The new phase of c-SiC was formed together with the remained C at 600°C. Complete C and Si reaction was found at 700°C from Raman spectra without any C peak. The formation of SiC at higher substrate temperature on the surface of carbon monolayer leads to hardne ss enhancement. Also, a nanoweb-like morphology of the c-SiC was observed on the surface of film from the SEM image. With regard to the mechanical properties, the hardness and Young's modulus of films decrease with increasing substrate temperature from RT to 500°C due to more sp2 bonding formed. And then the hardness and Young's modulus of films increase with increasing substrate temperature from 500 to 700°C due to the formation of nanostructured SiC phase.

EP-17 Effects of Si Addition on the Mechanical Properties and Cutting Performance of Nanocrystalline Cr-Si-C-N Coatings Prepared by a Hybrid Coating System, ***M.C. Kang*** *(kangmc@pusan.ac.kr), J.H. Jeon, K.H. Kim, J.S. Kim*, Pusan National University, Korea

Hard coatings are known to improve the performance of cutting tools in aggressive machining applications, such as high-speed machining. Unfortunately, the development of cutting tool for high-speed machining is not enough in machining of difficult-to-cut materials. The Cr–Si–C–N coatings, characterized as a nanocomposite nanosized (Cr and Si)(C and N) crystallites embedded in amorphous phase of Si_3N_4 and SiC, was successfully synthesized on WC-Co substrates by a hybrid coating system of AIP and sputtering method. Microhardness value of the Cr–Si–C–N coatings by nanoindentation was ¨43 Gpa, which was a much largely increased one compared with Cr–C–N coatings and Cr–Si–N coatings. In addition, the average friction coefficient of the Cr–Si–C–N coatings largely decreased with increasing Si content and compared with CrN, Cr(C, N), and Cr–Si–N coatings. The cutting performances of Cr-Si-CN coated tool for the high-hardened material (AISI D2 steel; HRC50) were investigated under various high-spindle speeds. The reliable on-the machine system for measurement of tool wear was introduced in this work.

EP-18 Evaluation of Thin PVD Coatings Using Rooms and High Temperature Nano-Testing, ***N.M. Renevier*** *(nrenevier@uclan.ac.uk)*, University of Central Lancashire, United Kingdom

There is an increasing demand for understanding nanostructured coatings properties not only at room temperature but also at high temperature near real application condictions. Recent developments in high-temperature nanoindentation[1] have shown increased interest amongst the research community as a step to get closer to real problems or modelling. Measurement methods have been developed for evaluating the hardness and modulus of Physical Vapour Deposited (magnetron sputtering and cathodic arc) coatings when the temperature is raised from room temperature to 400 degrees. Additional nano-testing capabilities have been used to characterise nano-structured properties, this includes nano-scratch, nano-impact, nano-wear. Atomic Force Microscopy and Scanning Probe Microscopy have been used as a mean of surface analysis. Additional techniques such as Scanning Electron Microscopy, Energy Dispersive Spectroscopy and X-Ray Diffraction techniques have been used to complement the analysis. Physical parameters including temperature or loading curve, coatings deposition technique, coating composition and microstructure have been investigated and a comprehensive database has been produced and results reported in this paper.

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EP-19 Mechanical and Tribological Properties of Graded Ti(BN-MoS_2) Based PVD Coatings, ***I. Efeoglu*** *(iefeoglu@atauni.edu.tr), A. Çelik, A. Alsaran, I. Kaymaz, F. Yetim*, Ataturk University, Turkey

In the present investigation, Ti(BN)+Ti(MoS_2) graded solid lubricant coatings were deposited by magnetron sputtering from separate Ti, TiB2, BN, and MoS_2 target. X-ray diffraction, microhardness tester, scratch tester and pin-on-disc tribometer were used to evaluate structural, mechanical, frictional, and adhesion properties. In the work, described here changes in the adhesion and friction-wear life exhibited by nine different coatings deposited under variants of deposition parameters have been investigated. It is found that there is good correlation between the critical loads and friction-wear life of the deposited thin films under different deposition parameters.

EP-20 Effect of the Target Shuttering on the Characteristics of the Ta-Si-N Thin Films by Reactive Magnetron Co-sputtering, *C.K. Chung (ckchung@mail.ncku.edu.tw),* ***T.S. Chen****, N.W. Chang, S.T. Hung*, National Cheng Kung University, Taiwan

The nanocomposite Ta-Si-N coatings were deposited using a reactive magnetron co-sputtering with and without alternating shutter control at different N2 flow ratios (FN2% = FN2 / (FAr+FN2)) of 3-20%. The evolution of microstructure, composition, surface morphology and nanomechanical properties of different Ta-Si-N thin films were characterized by X-ray diffraction, energy dispersed spectrum (EDS), scanning electronic microscopy (SEM) and nanoindentation, respectively. The microstructure of nanocomposite Ta-Si-N thin film with and without target shuttering is amorphous-like which has nanocrystalline grains embedded in an amorphous matrix at 3-10 FN2%. The amorphous-like Ta-Si-N without target shuttering control is transformed into the polycrystalline phase at 20 FN2% while the Ta-Si-N film with target shuttering is still in amorphous-like microstructure due to the increased silicon content. The morphology of amorphous-like Ta-Si-N films is smoother than the polycrystallin e Ta-Si-N film at 20 FN2%. The nano-hardesses of all Ta-Si-N films measured by nanoindentation were between 10.3 and 18.5 GPa. The hardness of Ta-Si-N films without shutter control increases with increasing FN2% to 10 % and then drop at 20 FN2%. In contrast, the hardness of Ta-Si-N films with shutter control was increased with increasing FN2% to 20 %. The maximum hardness of Ta-Si-N films without shutter control occurs at 10 % while that with shutter control is at 20 %. Amorphous-like Ta-Si-N films have much higher hardness and smooth morphology compared to polycrystalline films.

EP-21 Mechanical Properties Characterization of Multilayered Nano Thin Films by Atomistic Simulations, ***J.-C. Huang*** *(jc-huang@mail.tnu.edu.tw)*, Tungnan University, Taiwan

The molecular dynamics simulation method was used to investigate the mechanical characteristics of multilayered nano thin films. The NPT ensemble principle and COMPASS potential function were employed in the simulation. The multilayered nano thin film contained the Cu and Ni thin films in sequence. The average elongation of the Cu/Ni multilayered nano thin film in different axis was simulated under different applied stress. The corresponding stain could be obtained when the elongation was divided by the original lattice constant. Similarly, the strains under different stress field were also achieved. The ideal stress-strain relationship of the Cu/Ni multilayered nano thin film in different axis can be calculated based on the classical mechanics linear law. The Young's modulus of thin film in different axis was also obtained based on the slope of the simulated stress-strain curves. It was concluded that the Young's modulus of Cu/Ni multilayered nano thin film differe d in different axis.

EP-22 Phase Analysis of Alumina Coating by X-Ray Diffraction (XRD), Transmission Electron Microscope (TEM), Secondary Electron Microscopy (SEM) and Micro Indentation, ***W. Engelhart*** *(wolfgang.engelhart@walter-tools.com),* Walter AG, Germany*, W. Dreher,* NMI Natural and Medical Sciences Institute, Germany*, V. Schier,* Walter AG, Germany*, O. Eibl*, Eberhard Karls University Tübingen, Germany

Bulk alumina has a lot of structural degrees of freedom. This gives a variety of different phases which are well studied. The thermodynamically most stable phase is defined by the minimum of the Gibbs Energy. This function depends on a variety of external variables e.g. the interaction energy with

the substrate, the energy of grain borders, or even the external pressure. The well known alpha phase is not the minimum of the Gibbs energy for all ambient conditions. Some of these phases are synthetically fabricated with physical vapor deposition (PVD). The growing process depends sensible on the depositions conditions. For the design of the coating are special phases and grain size selected to increase the lifetime of cutting tools. A detailed phase analysis is necessary for the understanding of the wear mechanism. However it is a challenge to indentify the nanostructure clearly and in detail.

The one micrometer thick coating on tungsten carbide is a nalyzed with x-rays in the gracing incidence geometry (GIXRD). The FWHM of the diffraction pattern gives a number for the correlation length for one crystallographic orientation. The peak to background ratio is taken into account to evaluate the x-ray diffraction data. The combination of TEM bright and dark field images gives a microscopic picture of the real space structure and a first understanding of the homogeneity of the growing process. For industrial applications the film homogeneity is one of the basic demands. For a local analysis of the chemistry energy dispersive x-ray (EDX) in the SEM and TEM is the suitable method. We analyzed several different positions and compared the intensity ratio of the signal for special lines. The structure and the change of the structure is studied with selected area diffraction. As a result a model for the film is given. The model correlates with the mechanical properties, e.g. the hardness measured by micro indentation experiments.

EP-23 Galling and Wear Characteristics of Some Commercial PVD Coatings as Evaluated by Tribological Testing, ***J. Ericsson****, M. Olsson (mol@du.se)*, Dalarna University, Sweden

The increasing use of high strength steels in a variety of mechanical engineering applications has illuminated problems associated with galling in sheet metal forming operations. Galling is a tribological phenomenon associated with transfer of material from the steel sheet to the tool surface during forming resulting in seizure of the tool/steel sheet contact and extensive scratching of the steel sheet surface. As a result, a number of concepts have been developed in order to reduce the tendency to galling in sheet metal forming, including the development of new dry lubricants, new forming tool steel grades and improved surface engineering treatments such as the deposition of low friction CVD- and PVD-coatings. In the present study the potential performance of a number of commercial PVD coatings, including CrN, (Ti,Al)N and various DLC-based coatings, in the forming of hot and cold rolled high strength steel as well as electro and hot-dip galvanized high strength steel has been evaluated using pin-on disc testing and mono and multi pass sliding tests under dry as well as lubricated contact conditions. Post-test examination of the tribosurfaces using FEG-SEM and EDX analysis was performed in order to evaluate the mechanisms controlling the tendency to material transfer and wear.

EP-24 Tribological Properties of Cr_2N Ceramic Films with Tungsten Dopants, ***C.S. Wu*** *(csw@mail.mirdc.org.tw),* National Kaoshiung University, Taiwan*, C.W. Chu*, University of I-Shou, Taiwan

With the miniaturization of the dimensions of the current micro-drill for machining flexible printed circuit board (PCB) , the rotational speed has reached up to 30 krpm.The miniaturization of the micro-drill makes the rework of itself become more and more difficulty. Surface coating is one of the methods to prolong the service life of the micro-drill and Cr2WN is a prominent material for the protective coating which could apply on the micro-drill due to its excellent oxidation and adhesive resistance properties.Some experimental data use the UCψ0.25×4.0 D2 as the drill sample are in the following: the hardness increased with the increase of the W content,the hardness increased from 17.76GPa to 26.0Gpa.However,when W contents increased, the hardness values also increase. Experimental results show that when W content increased, the wear rate、grain size、contact angle and the mechanical properties of the Cr_2WN coating are superior to those in other W contents It will have the minimum flank wear and the tool life is 4 times longer than those of the uncoated samples.

EP-25 Microabrasion Wear Testing of PEO Coatings on 2024 Aluminium Alloy, ***A. Pilkington*** *(t.pilkington@sheffield.ac.uk), H.X. Cheng, A. Yerokhin, A. Matthews*, University of Sheffield, United Kingdom

Plasma electrolytic oxidation (PEO) attracts increasing interest as an environmentally friendly technology to improve the wear resistance of Al alloy surfaces. The conventional PEO process suffers from a low growth rate, microstructural defects such as shrinkage cracking and voids which limits the effectiveness of the oxide ceramic coating on Al alloys. A recently introduced higher frequency pulsed bipolar (PBP) PEO treatment enables faster production of oxide coatings with refined morphology based on gamma alumina. This created a requirement to evaluate the wear resistance of the new PEO coatings with greater sensitivity than conventional wear tests (e.g. Tabor test). It is also useful to discriminate between wear rates of the well known outer porous layer and denser inner regions of PEO coatings. A microabrasion test can discriminate between local wear rate variations due to hardness or morphology within a thin surface coating. It also offers greater freedom from self abrasion. The microabrasion test was used to investigate the wear coefficients of a series of PBP PEO coatings on 2024 aluminium alloy. A 25mm AISI 52100 steel ball was used with 1200 grit SiC slurry. Normal loads in the range 0.1-0.3N were used for between 50-1000 cycles. Wear scars were examined by optical microscopy and surface profilometry. Apparent wear volumes measured by the two techniques were compared. The effective ball diameter was estimated from the wear scar profile data. SEM imaging was used to investigate the wear mechanism. The wear coefficients obtained are discussed in relation to the coating morphology, hardness and phase composition.

EP-26 Influence of Nitrogen Ion Implantation on Hard Coating Layer to Improve Adhesion Strength Using Combined Surface Modification, ***G.C. Jeong*** *(gcjeong@kpu.ac.kr),* Korea Polytechnic University, Korea*, Y.H. Sohn,* University of Central Florida*, S. Kwun*, Korea University, Korea

In order to improve the durability and pefermance of molds and tools, diverse methods of surface modification are used, including PVD. In this method adhesion strength between a substrate and coating plays an important role. To improve adhesion strength, a combined surface modification method is ofen utilized. In this study, on substrate(STS420), a various combination of ion-nitriding, Cr intermediate layer, CrN coating layer and nitrogen ion-implantation was examined. Phase constituents, microstructure, adhesion strength and hardness of coating-substrate system with combined surface modification was examined by using opical microscopy, X-ray diffraction, transmission electron microscopy, scratch test, and nano-indentation. Highest adhesion strength was observed when CrN coating was formed by Nitrogen implantation on ion-nitrided substrate with a Cr-intermediate layer. Influence of processing sequence and combination is related to microstructural observations and adhesi on strength.

EP-27 Ti(C,O,N)-Based Coatings for Biomedical Applications: Influence of Composition and Structure om the Mechanical/Tribological and Biological Behaviour, *N. Jordão, S. Ribeiro,* Universidade do Minho, Portugal*,* ***J.C. Sanchez-Lopez****, M.D. Abad,* Instituto de Ciencia de Materiales de Sevilla, Spain*, F. Vaz, M. Henriques, R. Oliveira,* Universidade do Minho, Portugal*, R. Escobar-Galindo,* Instituto de Ciencia de Materiales de Madrid (ICMM -CSIC), Spain*, S. Carvalho (sandra.carvalho@fisica.uminho.pt)*, Universidade do Minho, Portugal

Application of thin films in the biomedical engineering field represents an attractive challenge due to the multiple situations where they may improve or even functionalize a certain parts of human body. Implants are one of such cases, representing one of the most active fields within the so-called biomaterials R&D. Implant failure is a huge problem for both the patient and governmental agencies, once it involves repeated surgeries and consequently considerable economical resources. This failure can be attributed to excessive wear and wear debris and also to microbial infection. Byway, the use of several kinds of nitride-based this films has been carried out in the group, with some promising results. Thus, the main aim of the present work is to study such nitride-based thin films, namely in what concerns to C and O additions to well-known TiN thin films. The obtained Ti(C,O,N) based coatings where then characterized in terms of surface biofilm formation in order to acqu ire knowledge to use them in several medical devices. The thin films were deposited by DC unbalanced reactive magnetron sputtering. Mechanical/tribological resistance of the films was achieved by hardness testing as well as by friction and wear measurements. The tribological response was studied under biological fluid, using reciprocating and pin-on disk configurations. An improvement of the wear rate is observed by application of a bias voltage during film preparation and also for reduced oxygen contents in the films. Regarding biological properties, it was noticed that all samples showed similar ability for Staphylococcus epidermidis biofilm formation, although it was reduced in comparison to the control (stainless steel without coating). However, concerning with fibroblast viability, it was possible to verify that sample coated with TiON had a lower effect that samples with other coatings.

Characterization: Linking Synthesis Properties and Microstructure
Room: Town & Country - Session FP

Symposium F Poster Session

FP-1 DC Electric Field Induced Second Harmonic Generation Studies of Low Dielectric Constant SiOC(-H) Thin Films, *R. Navamathavan,* Chonbuk National University, Korea, *C. Y. Kim, H. S. Lee, J.-K. Woo,* ***C.K. Choi*** *(cckyu@cheju.ac.kr),* Cheju National University, Korea, *S.H. Seo, H.Y. Chang*, Korea Advanced Institute of Science and Technology, Korea

Second harmonic generation (SHG) optical techniques have been utilized to investigate charge carrier dynamics associated with surfaces and interfaces of semiconductor/dielectric structures as a noninvasive and reliable probe method. The performance and reliability of metal-insulator-semiconductor (MIS) structures depends more and more on the microscopic quality of the dielectrics and their interfaces. In this study, the Al/SiOC(-H)/p-Si(100)/Al interface is probed by the interferometry of direct current (dc) electric field induced second harmonic (EFISH) generation. Carbon doped silicon oxide (SiOCH) film with low dielectric constant are deposited on p-type Si(100) substrates by using plasma enhanced chemical vapor deposition (PECVD) technique. The SiOC(-H) films with different precursor flow rate of ratios varying from 40 to 100 % are deposited. The output of a Q-switched Nd:YAG laser at 1064 nm with a pulse duration of 8 ns, maximum average power of 9 W and energy of approximately 5 mJ/pulse is used as the fundamental radiation. The incoming linearly polarized light is directed onto the Al/SiOC(-H)/p-Si(100)/Al samples at a 45° incident angle and focused to 750 μm diameter spot on the sample. The SHG signal are selected by a polarization analyzer and detected in the photomultiplier tube. The interface state density of the as-deposited Al/SiOC(-H)/p-Si(100)/Al structure prepared with the flow rate ratio of 90% was considerably lower than with the flow rate ratio of 60%.

FP-2 Light Out-Coupling Properties of Organic Light-Emitting Diodes with the Ba-doped Alq3 Layer and Degradation Mechanism of the Devices Based on its Electronic Structure, ***J.T. Lim***, *G.Y. Yeom (gyyeom@skku.edu)*, Sungkyunkwan University, Taiwan

The Ba-doped Alq_3 layer into the organic light-emitting diodes (OLED) was inserted both to reduce barrier height for an electron injection and to improve the electron mobility, from cathode to the electro-transporting layer of tris(8-quinolinolato)aluminum (III) (Alq_3). The light out-coupling property of the OLEDs, which is consisted of glass / ITO / 4,4',4"-tris[2-naphthylphenyl-1-phenylamino]triphenylamine (2-TNATA, 30 nm) / 4,4'-bis[N-(1-napthyl)-N-phenyl-amino]-biphenyl (NPB, 18 nm) / Alq_3 (42 nm)/ Ba-doped Alq_3 (20 nm, x %:x= 0, 25, and 75) / Al (20 nm), was investigated. The OLED with the Ba-doped Alq_3 layer of 25% showed the highest light out-coupling characteristic as the luminance of about 61000 cd/m^2. This device performance could be interpreted on the base of chemical reaction between Ba and Alq_3 as well as electron injection property by analyzing electronic structure of the Ba-doped Alq_3 layer. In ultra violet photoemission spectroscopy, when a doping concentration of Ba was increased from 0 to 75 %, the barrier height for electron injection was decreased in the Ba-doped Alq_3 layer. Meanwhile, in O 1s X-ray photoemission spectroscopy, the Alq_3 radical anion species was formed when Alq_3 is doped with Ba of 25 %. However, In the case of Alq_3 doped with Ba of 75%, Alq_3 molecules were severely decomposed. When a doping concentration of Ba is changed, the light-emitting characteristics of the devices were well coincided with formation mechanism of Alq_3 radical anion species and Alq_3 decomposition species, being originated from chemical reaction between Ba and Alq_3. Near-edge X-ray absorption fine structure (NEXAFS) spectra at N K-edge showed that electron charge injection could be improved by Ba than a low work function metal such as Cs, because Ba than Cs could provide more electron to the orbitals involved with the pyridyl ring co mposing the Alq_3 molecule. Therefore, Ba on the Alq_3 is estimated to show more excellent device performance than that of Cs.

FP-3 Effect of P Additions on the Thermal Stability and Electrical Characteristics of NiSi, ***H.-F. Hsu*** *(hfhsu@dragon.nchu.edu.tw), C.-L. Tsai, H.-Y. Chan, T.-H. Chen*, National Chung Hsing University, Taiwan

The P-inserted Ni films were fabricated by immersion deposition process on Si(100), and then the NiSi films were formed by annealing in furnace. High sheet resistance of the as-deposited Ni(P) thin film was caused by the presence of high-density and P-insert of grain boundaries. The sheet resistance reduced to low level after annealing at 400^0C because polycrystalline NiSi film with Ni_2P capping layer was formed. The addition of P can enhance the thermal stability of NiSi films, which was exhibited by X-ray diffraction data and sheet resistance measurement. The transformation temperature of NiSi to $NiSi_2$ increased from 800 (for pure Ni films) to 900 ^{0}C because the P-substituted layer on the top of the NiSi film was formed when sample annealed at 500 to 800 ^{0}C. The presence of the P-substituted layer on the top of the NiSi layer can decrease the Gibbs free energy and alter the kinetics of the free-surface-related relaxation mechanism of the NiSi film, which affects the nucleation of the NiSi to $NiSi_2$ transformation.

FP-4 Performance of Various Duplex Systems Based on DLC and Salt Bath Nitriding Processes of 316L Stainless Steel, ***L. Gil***, *S. Liscano,* UNEXPO, Venezuela, *C. Gruescu, D. Chicot,* University of Science and Technology of Lille, France, *E.S. Puchi-Cabrera, M.H. Staia (mhstaia@gmail.com)*, Universidad Central de Venezuela

The present investigation was carried out to study the potential use of the cyanide–cyanate salt bath of a 316L stainless steel as an alternative nitriding process able to provide a higher load-carrying capacity for DLC magnetron sputtered thin coatings used as biomaterial. Despite its low cost, the salt bath has not been used as a valid nitriding process for this kind of steel due to its traditional drawbacks, such as poor control of the process, which leads to the formation of a porous compound layer and, hence, to a bad corrosion and tribological performance. However, a careful characterization of the nitrided layer obtained from this process by means of Vickers indentation, which leads to the determination of the relationships between the nitrogen profile and hardness, could be used to obtain an adequate preparation of the nitrided substrate before PVD deposition, by carrying out the right elimination of the compound layer. Finally, the nitrided substrates produce d in different conditions were characterized by employing standard corrosion and tribological tests and a comparison was made in order to obtain the most functional substrate prior PVD deposition.

FP-6 Microstructure and Nanostructure of PVD ZrN-Cu Thin Films, *M. Audronis, O. Jimenez, A. Leyland,* ***A. Matthews*** *(a.matthews@sheffield.ac.uk)*, The University of Sheffield, United Kingdom

ZrN-Cu films containing variable amounts of copper, namely 8, 33 and 58 at. %, were produced by reactive pulsed unbalanced magnetron sputter deposition. Coatings were found to possess hardness values of 22.5, 9.5 and 3.7 GPa, respectively. The morphology of coatings was investigated by field emission scanning electron microscopy and the microstructure and nanostructure were investigated by conventional (bright-field and dark-field imaging) and high resolution transmission electron microscopy. Complementary X-ray diffraction experiments were also performed. ZrN coatings containing 8 at. % of copper were found to possess a nano-columnar structure composed of ZrN columnar grains, the diameter of which was approximately 15 to 30 nm. The majority of the copper content was apparently dissolved within the ZrN grains, rather than existing as a separate phase. Coatings of the two other compositions were found to be composed of a mixture of mostly equiaxed ZrN and Cu grains, the diameters of which were in the approximate range of 10 to 30 nm. None of the coatings investigated in this study were found to possess the so-called 'nanocomposite' structure, which is often pictured as crystalline grains surrounded by an amorphous phase. Instead, coatings were found to be either single-phase ZrN with Cu in substitutional solid solution for Zr or a mixture of ZrN and Cu nano grains.

FP-7 Bias Efects on the Wear Behavior of AISI 304 Stainless Steel Arc-Deposited CrTiAlN Multilayer Coatings, *C.-H. Hsu,* ***K.-L. Chen*** *(explor88@seed.net.tw), Z.H. Lin,* Tatung University, Taiwan, *C.-K. Lin*, Feng Chia University, Taiwan

In this study, CrTiAlN multilayer coatings were deposited on AISI 304 stainless steel using a cathodic arc deposition system controlled at the different biases. Coating morphology and properties such as coating structure, adhesion, hardness/elastic modulus (H/E) ratio, and abrasion behaviors were analyzed to evaluate the bias effects on the coatings for application on AISI 304 stainless steel. The results showed that when bias value was controlled at -150V, not only Cr content in the coatings had an increase, but also the better properties such as lower Ra value, HF1 adhesion strength, and higher H/E ratio were all obtained. Sequentially, the coated specimen produced with the bias value of -150V showed an outstanding wear resistance as compared to the uncoated and the other coated ones. Keywords: AISI 304, Cathodic arc deposition, CrTiAlN, Bias effect, Wear resistance.

FP-8 Microstructure Analysis of TiO_2 Thin Film Modified by Metal Plasma Ion Implantation for Enhanced Photosensitivity, *D.-Y. Wang,* Mingdao University, Taiwan, ***C.-C. Yen,*** National Chung Hsing University, Taiwan, *M.-H. Shih,* Mingdao University, Taiwan, *L.S. Chang, H.-C. Shih (jackal8914@yahoo.com.tw)*, National Chung Hsing University, Taiwan

Photo-catalytic TiO_2 thin films were prepared by using sol-gel process. To improve the photo-sensitivity of TiO_2 at visible light regime, transition metals of Cu, Fe, and Ni were implanted into the TiO_2 matrix at 30 KeV by using the metal plasma ion implantation (MPII) process. The additional band gap energy levels created due to the formation of the implanted metal oxides extended the photosensitivity range of the anatase TiO_2 to cover the visible light spectra, allowing increased photo-catalytic efficiency of TiO_2. Pure anatase TiO_2 thin films posses a transparency better than 80% transmittance between 450 and 800 nm. The irradiation of the medium-energy metal plasma posed significant effects on the lattice distortion as well as the microstructure of TiO_2 thin films. The loss of transmittance of the metal-implanted TiO_2 was resulted from the formation of the photo-catalysis adsorption in addition to the existence of typi cal adsorption centers such as lattice defects, structural defects, composition fluctuation, impurity compounds, and others. In this study, the implantation parameters were optimized to compensate the loss of TiO_2 transparency without significantly affecting the formation mechanism of impurity oxides. The microstructure and morphological analyses will be conducted by using electron microscopy, photo-luminance spectroscopy, and atomic force microscopy.

FP-9 Analysis of ZrN/Zr Thin Film on Flexible Polyethylene Terephthalate (PET) Substrate Deposited by Unbalanced Magnetron Sputtering, ***H.-M. Chu*** *(g9611534@oz.nthu.edu.tw), J.H. Huang, G.P. Yu*, National Tsing Hua University, Taiwan

Recently, polymer-based organ light-emitting diode (OLED) was developed due to the issues of energy and environment. However, the gas impermeability of polymer substrates is very bad. The permeation of water and oxygen vapor can oxidize the cathode of OLED, which seriously decreases its performance and lifetime. To reach the requirement of OLED (water vapor transmission rate, WVTR, $<10^{-6}$ g/m^2/day; oxygen transmission rate, OTR, $<10^{-3}$ cm^3/m^2/day), several barrier coatings like silicon oxide and nitride have been developed. In this study, ZrN was chosen to deposit on flexible polyethylene terephthalate (PET) substrates by unbalanced magnetron sputtering system (UBMS). According to the literatures, the interlayer between the thin film and substrate can improve the adhesion and relax the residual stress. Besides, the pure Zr interlayer can increase the electric conductivity and the packing density of the thin film. The effect of diffe rent deposition time of ZrN and Zr was investigated in terms of adhesion, electrical resistivity, surface roughness, transmittance and WVTR. Scanning Electron Microscopy (SEM) was used to observe micro-structure. The crystal structure was characterized by Glancing Incident X-ray Diffraction (GIXRD). Compositions of the thin film were measured by Rutherford Backscattering Spectroscopy (RBS) and X-ray Photoelectron Spectroscopy (XPS), and the packing factor can be calculated by the results of RBS. The composition depth profile was analyzed by Auger electron spectrometer (AES). Four-Point Probe was utilized to measure the sheet resistance. The adhesion and the wettability of thin films were tested by Cross-Hatch Test and the Contact Angle System, respectively. Atomic Force Microscopy (AFM) was used to measure the surface roughness. Transmittance of the thin film was determined by UV-Visible Spectrophotometer. Besides, the color and the reflectance of the thin film were analyzed by HunterLab MiniScan XE Plus Spectrophotometer Model 4000VSAV. Finally, Water Vapor Permeation Instrument was used to measure the WVTR. The transmittance decreased with increasing film thickness. The WVTR was obviously affected by film thickness and packing factor. Films with higher packing factor and thickness had better gas impermeability. The sheet resistance was decreased with increasing thickness of Zr interlayer. Further correlations between structure and properties are described in this paper.

FP-10 Dynamic Deformation and Durability Evaluation of Various Lubricant Coated Magnetic Disks, ***W. Kurosaka*** *(s1014219@sstu.nit.ac.jp), K. Oshimoto, S. Miyake*, Nippon Institute of Technology, Japan

Nanowear and viscoelastic evaluation tests were performed to study the nanotribological properties of various perfluoropolyether lubricant (thick-Ztetraol, thin-Ztetraol, D-Tetra and A20H-DD) films coated magnetic disks. The magnetic-disk surface protrudes due to friction, partially. This result corresponds to the result of viscoelastic change by nanosliding evaluated by AFM(atomic force microscopy). In the case of A20H-DD and thick Ztetraol, tanδincreases by friction. This shows the increase of lubricant by sliding. On the other hand, in the case of D-Tetra, tanδ decreases by sliding. Tan δ of the protuberance area is the smallest. This result shows the similar tendency of the case that the magnetic disks without lubricant. The friction properties of the various lubricant coated magnetic disks are evaluated by the reciprocating friction tests. The friction coefficients of A20H-DD coated magnetic disks are stable, and friction damages are also less than the others. T hese superior properties of A20H-DD coated magnetic disk correspond to nanotribological results evaluated by AFM.

FP-11 Interface Evolution of Annealed SiO_2 and Ta/SiO_2 Encapsulated Cu Films, ***A.P. Warren***, *T. Sun (ti857626@pegasus.cc.ucf.edu),* University of Central Florida, *K. Barmak,* Carnegie Mellon University, *M.F. Toney,* Stanford Synchrotron Radiation Laboratory, *K.R. Coffey*, University of Central Florida

Surface roughness induced scattering has been considered as a potential contributor to high resistivity in Cu interconnects. To study the evolution of roughness with annealing, a series of Cu thin films ranging in thickness from 28 nm to 158 nm, encapsulated in SiO_2 and Ta/SiO_2 were fabricated. The samples were annealed at 150°C and 600°C following deposition. The root mean square interfacial roughness was characterized by specular x-ray reflectivity for both the upper and lower Cu interfaces. The lateral correlation length of the roughness was studied by diffuse x-ray reflectivity and grazing incidence small angle x-ray scattering. The measured interfacial roughness was in the range of 2 Å to 15 Å. Little correlation was observed between roughness and thickness for the samples. The roughness of the lower SiO_2/Cu interface did not change with annealing temperature. By contrast, the roughness of the upper Cu/SiO_2 interface decr eased by 60% upon annealing at 600°C. Such a reduction in roughness with annealing temperature was not observed in the SiO_2/Ta/Cu/Ta/SiO_2 samples. The lateral correlation length scaled with thickness for all samples, and annealing at 600°C resulted in longer wavelengths.

FP-12 Formation of Magnetron Sputtered α-Ta at Zero Bias at Room Temperature and its Nitridation Behavior, *C.K. Chung (ckchung@mail.ncku.edu.tw),* ***N.W. Chang***, *T.S. Chen, S.C. Chang, S.T. Hung*, National Cheng Kung University, Taiwan

The tantalum (Ta) films by the conventional sputtering method generally contain a metastable tetragonal β-Ta with higher resistivity than a stable cubic α-Ta. The conventional α-Ta film was grown by sputtering at a substrate temperature up to 400°C or applying a bias on substrate. In this paper, we have investigated the formation and resistivity of α-Ta at zero bias and room temperature (RT) as well as its nitridation behavior for Ta-N thin films on Si (100) by reactive magnetron sputtering in the nitrogen flow ratios (FN2%=FN2/(FAr+FN2)) of 0-10%. The microstructure, surface morphology, and resistivity of different Ta and Ta-N thin films were examined by X-ray diffraction, scanning electronic microscopy (SEM) and four-point probe, respectively. The stable α-Ta films with low resistivity of 105.0 μΩ-cm can be obtained at zero substrate bias at RT. The the resistivity of films increases with increasing FN2% for the formation of Ta-N phase. The phase formation se quence of the films at zero bias at RT is from α-Ta to amorphous-like Ta-N to fcc Ta-N as the FN2% increases from 0 to 10%. The maximum resistivity of Ta-N film here is 313.6 μΩ-cm at 10 FN2% from a polycrystalline phase while the Ta-N films at low 3-5 FN2% has lower resistivity from an amorphous-like phase. The Ta-N film at high FN2% has larger granular structure and open boundary than that of the Ta-N film at low FN2% with close boundary arrangement. The relationship between the resistivity and the microstructure of the sputtered α-Ta and Ta-N films is discussed and established.

FP-13 Process Kinetics During RF Sputtering of $LiCoO_2$ Thin Films for Micro Battery Applications, *C.S. Nimisha, M. Ganapathi, M. Nookala,* ***M.R. Gowravaram*** *(gmrao@isu.iisc.ernet.in)*, Indian Institute of Science, India

Sputtering from multielemental targets often results in non stoichiometric films when the target is not conditioned for a stable composition. In this study we present the target conditioning analysis of $LiCoO_2$ sputter cathodes in terms of the chemical composition of the deposited films. During these studies it was also seen that the substrate roughness and the target-substrate distance play a major role in achieving the required composition in the deposited films. Electrochemical data in relation to these process parameters is presented in this article. The optimized conditions for the deposition of good quality $LiCoO_2$ films with a capacity of 50 microAmpHr/cm^2/μm are given. These conditions are different from the data available in the existing literature where it was demonstrated that good quality films could be deposited at larger target-substrate distance, where as, our study shows that due to thermalization distance of different elements, a target-substrate distance of 5 cm is ideal. The effect of the composition of the gasesous ambient during sputtering is also discussed and is shown that a argon:oxygen ratio of 10:1 results in good quality films in terms of electrochemical performance and composition.

Applications, Manufacturing, and Equipment
Room: Town & Country - Session GP

Symposium G Poster Session

GP-1 Nanocomposite Coatings and Triple Coatings on High Performance Tools with Dedicated Edge Preparation, ***A. Lümkemann*** *(a.luemkemann@platit.com), M. Morstein, T. Cselle, O. Coddet,* PLATIT AG, Switzerland*, V. Hajek, M. Jilek,* Pivot a.s., Czech Republic*, P. Karvankova*, PLATIT AG, Switzerland

Optimum edge preparation can increase tool performance and -lifetime, thereby enhancing the machining process enormously. The importance of a well controlled tool micro-geometry has been proven to be nearly as important as the choice of the coating itself.

Tool pretreatment reduces edge surface roughness and decreases friction between tool and workpiece. By means of a suitable edge preparation, a defined edge rounding will be achieved and edge chipping avoided during the cutting operation.

Tool pretreatment must be adapted to the type of tool (inserts, drills, taps, end mills and hobs) and especially to their applications, depending on the work piece materials, cutting and machine conditions.

In this contribution, dedicated edge preparation case studies will be shown for several examples of high-performance machining, such as cast iron drilling, milling of high-strength steels and dry gear cutting with HSS hobs especially for nanocomposite coatings and the new generation of PVD coatings the TripleCoatings®.

The TripleCoatings® combine several advantages of conventional coatings (TiN, CrN, TiAlN, AlCrN) as of nanocomposite coatings (nc-TiAlN/a-SiN, nc-AlCrN/a-SiN). That makes TripleCoatings® applicable for both general purpose and high performance machining.

GP-2 Deposition of SiO_2 Thin Films with Dielectric Barrier Discharge at Atmospheric Pressure PECVD using PDMS/O_2/He/Ar, ***Y.S. Kim****, J.H. Lee, G.Y. Yeom (gyyeom@skku.edu)*, Sungkyunkwan University, Korea

The use of flexible substrates enables new applications, such as electronic devices and next generation display devices. However, one of the limitations of polymeric substrate in these applications is that oxygen and moisture rapidly diffuse through the material and subsequently degrade the electro-optical devices. To protect the flexible displays from the atmosphere, a thin film which is composed of single inorganic layer or a multilayer should be deposited on the flexible substrate of display device. SiO_2 thin films have a wide variety of electronic applications such as gate dielectrics, insulators, waveguides, etc. Atmospheric pressure (AP)-PECVD processes not only useful compared to other deposition processing conducted in vacuum but also to deposit the material uniformly over the large-area substrate by in-line processing. Among the various plasma sources operating at atmospheric pressure, dielectric barrier discharge (DBD) is one of the most promising source s for generating non-equilibrium plasmas for AP-PECVD processes. Pin-to-plate DBD (dielectric barrier discharge) is high pressure widely applied in surface modification, and thin film deposition. In this study, using the polydimethylsiloxane (PDMS) as the precursor of Si, SiO_2 thin films were deposited on plastic substrates using atmospheric plasma enhanced chemical vapor deposition (AP-PECVD) method at low temperature (< 50 ℃) and its properties were investigated. Atmospheric pressure plasma discharges were generated by applying AC voltage. The substrate was fed to the AP-PECVD system at 0.3 m/min though an in-line feeder. The increase of PDMS flow rate in the gas mixture increased deposition rate. However, it also increased impurities such as carbon group in the film and surface roughness. To have SiO_2 film at a high deposition rate with low impurity and low surface roughness, an adequate mixture of oxygen and PDMS flow rate appears to be needed.

GP-3 Cutting Performance of Cr-Al-N and Cr-Al-Mo-N Coated End-Mill Deposited by Hybrid Coating, ***H.S. Tak****,*** Pusan National University, Korea*, S.H. Kwon,* KAIST, Korea*, K.H. Kim, M.C. Kang (kangmc@pusan.ac.kr)*, Pusan National University, Korea

In this paper, comparative studies on the properties and cutting performance between Cr-Al-N and Cr-Al-Mo-N coated micro end-mill for ultra-high speed machining applications were conducted. Quaternary Cr-Al-Mo-N coatings were deposited on STS 304 steel and Si substrates by a hybrid coating method of an arc ion plating (AIP) for Cr target and a dc magnetron sputtering technique for Al and Mo targets under N2/Ar atmosphere. The synthesized Cr-Al-Mo-N coatings were mainly composed of a substitutional solid solution (Cr, Al, Mo)N. The maximum microhardness of Cr-Al-Mo-N coatings of 35 GPa was obtained with the Mo content of 24.2 at.%. Moreover, the friction coefficient of Cr-Al-Mo-N coatings drastically decreased from 0.9 to 0.48 with increasing Mo content from 0 to 33.2 at.% due to the formation of MoO3 which acted as a solid lubricant between the coating surface and steel ball. And then, Cutting tests were carried out to evaluate the characteristics of micro tool in vertical machining center using ultrahigh-speed air turbine spindle. Especially, the reliable evaluation system of coated tools for micro machining, where the cutting force and tool wear were simultaneously measured, was introduced.

GP-4 Effect of Ferromagnetic Module on Microcrystalline Silicon Thin Films Deposited by Internal-ICP, ***H.-C. Lee****, M.H. Jeon, K.N. kim, J.H. Lim, G.Y. Yeom (gyyeom@skku.edu)*, Sungkyunkwan University, Korea

Hydrogenated microcrystalline(uc-Si:H) silicon thin films prepared by plasma enhanced chemical vapor deposition (PECVD) have proven their potential on the device applications such as solar cells, thin film transistors (TFTs), sensors, etc. Especially, these crystalline silicons have great potentials in the application to active matrix organic light emitting diode because this crystalline silicon has some attractive electrical properties. The high mobility of uc-Si:H, in particular, is strongly expected for the realization of high speed devices on glass substrates. Also, due to its more rigid structure, uc-Si:H may be a solution to the increase of lifetime which is one of the significant problems of a-Si:H. In this study, the effects of internal antenna type-ICP with ferromagnetic module were investigated for the crystallization of silicon thin film during the deposition of silicon by SiH_4/H_2 and the structural properties of the thin films such as surface morphology, cry stalline fraction were studied. This uc-Si:H thin film were deposited on corning 1737 glass, silicon dioxide and single-crystal Si. The film properties were characterized using Raman scattering spectroscopy, scanning electron microscopy (SEM), X-ray diffraction (XRD), High resolution transmission electron microscopy, (HRTEM) and I-V measurements to evaluate film crystallinity, structural image, crystal direction, and dark conductivity, respectively.

GP-5 Line-Type Internal Linear Inductively Coupled Plasma Source for Large Area Roll-to-Roll Plasma Processing, ***J.H. Lim****, K.N. Kim, G.H. Gweon, G.Y. Yeom (gyyeom@skku.edu)*, SungKyunKwan University, Korea

Flexible display devices are being investigated by many researchers as a potential next-generation display. Especially, roll-to-roll plasma processing is an important technique for flexible display processing. For the fabrication of flexible display devices by roll-to-roll plasma processing, not only highly uniform plasma processing but also high processing rates are required to increase the throughput of the processing. In this work, the characteristics of a line-type, internal antenna for an inductively coupled plasma (ICP) source installed with a ferromagnetic module were investigated for possible application to roll-to-roll processing of next-generation display devices. The use of 2MHz instead of 13.56MHz for the 2300mm-long ICP source improved the plasma uniformity to less than 11% along the antenna line due to the lack of the standing wave. In addition, the use of Ni-Zn ferromagnetic material in the line-type antenna improved the plasma density to about $3.1 \times 10^{11}/cm^3$ at 3500W of 2MHz radio frequency (rf) power by confining the induced, time-varying magnetic field between the antenna line and the substrate. When the photoresist-covered glass substrate was etched at 4000W using 40mTorr and Ar/O_2 (7:3), an etch uniformity of about 5~6% was obtained along the antenna line.

GP-6 Through-Thickness Microstructural Characterization of the Plasma Electrolytic Oxidized Titanium Oxide Fabricated on Metal Titanium, ***P.-J. Chu*** *(P.Chu@sheffield.ac.uk), A. Yerokhin, A. Leyland, A. Matthews,* University of Sheffield, United Kingdom*, J.-L. He*, Feng Chia University, Taiwan

Titanium dioxide (TiO_2) films prepared by plasma electrolytic oxidation (PEO) on titanium have been considered for a variety of applications such as mechanical wear resistance, antimicrobial protection, photocatalytic surface functionality, bone implant biocompatibility and osteoinduction, as well as for electrode materials in dye-sensitized solar cell (DSSC) devices. PEO layers are, known to be porous, typically exhibiting both compositional and structural inhomogeneity through the layer thickness. Little attention has previously been paid to PEO layer structure at a scale less than its film thickness - particularly for the PEO-TiO_2/Ti system (for which the crystal structure over the entire layer thickness is in need of more precise determination). This study aims to explore the abovementioned knowledge gap, using several material characterization techniques. The cross-sectional morphology of the PEO layer is observed by scanning electron microscopy (SEM). Overall crys tal structural distribution through the layer thickness is examined by glancing angle X-ray diffraction (GA-XRD). Discrete crystallites in the PEO layer are characterized by transmission electron microscopy (TEM) to determine morphology and crystal structure. The results show that micrometer-scale porous anatase TiO_2 films can be synthesized, composed primarily of the anatase phase, with crystallites mostly distributed close to the substrate interface, while minor amounts of the rutile phase can be found towards the top of the layer.

Microstructural and compositional evolution through the layer thickness is explained in terms of the observed process effects.

GP-7 Electron Temperature Cooling Down with Multi-Step Ionizations in an Electron Beam Generated Plasma, *H.Y. Chang,* ***S.H. Chae*** *(sh_chae@kaist.ac.kr)*, Korea Advanced Institute of Science and Technology (KAIST), Korea

In general, the plasma generated by an electron beam is known to have the low electron temperature. Because high energy electrons frequently collide with background gases, electrons loss their energy easily. We also consider the effect of multi-step ionizations as a reason of the low electron temperature in an electron beam generated plasma. The excited state neutrals are important in multi-step ionizations. In the experiment, the electron temperature is measured by the Langmuir probe in an Ar electron beam generated plasma. EEDF (electron energy distribution function) is obtained. The relative excited state Ar density is measured by the spectrometer. As the pressure increases, the electron temperature decreases as well as the relative excited state Ar density increases. We confirm the effect of multi-step ionizations in an electron beam generated plasma.

GP-8 Structure and Properties of Low Temperature Plasma Carburized Austenitic Stainless Steels, *R.M. Souza,* University of São Paulo, Brazil, *M. Ignat,* SIMAP Grenoble INP, France, *C.E. Pinedo,* ***A.P. Tschiptschin*** *(antschip@usp.br)*, University of São Paulo, Brazil

Austenitic stainless steels cannot be conventionally surface treated at temperatures close to 550°C due to intense precipitation of nitrides or carbides. Plasma carburizing allows introducing carbon in the steel at temperatures bellow 500°C without carbide precipitation. Plasma carburizing of AISI 316L and 304L was carried out at 480°C, during 20 hours, using CH_4 as carbon carrier gas. The results show that a 20 microns carbon expanded austenite layer was formed on the surface. DRX results showed that the austenitic FCC lattice parameter increases from 0.358nm to 0.369nm, giving an estimation of circa 10 at. % carbon content. Lattice distortion, resulting from the expansion and the associated compressive residual stresses increase the surface hardness to 1040 HV0.025. Micro-scale tensile tests were conducted on specimens prepared with the conditions selected above, which has indicated that the damage imposed to the expanded austenite layer was more easily related to eac h separated grain than to the overall macro-scale stresses imposed by the tensile test.

GP-9 Conversion Coatings Developed in Ionic Liquid and Their Anti-Corrosion Capability for a Magnesium Substrate, ***J.K. Chang*** *(catalyst@mail.mse.ncku.edu.tw), M.H. Chuang, W.T. Tsai,* National Cheng Kung University, Taiwan, *M.-J. Deng,* National Chung Hsing University, Taiwan, *I.W. Sun*, National Cheng Kung University, Taiwan

Fabrication of a protective conversion coating on a magnesium (Mg) substrate was successfully conducted in butylmethylpyrrolidinium bis(trifluoromethylsulfony)imide (BMP–NTf2) ionic liquid. Effects of the reaction temperature and applied potential on material characteristics of the produced layer were investigated using a scanning electron microscope, an X-ray diffractometer, and also an X-ray photoelectron spectroscope. In addition, corrosion resistances of the coated Mg sample and a bare Mg plate (as a counterpart) were evaluated by electrochemical measurements in 3.5 wt% NaCl aqueous solution. The analytical data indicated that a fluorine-containing surface layer was uniformly developed in the BMP–NTf2 ionic liquid, and thus provided a satisfactory protection for the Mg substrate against the hostile environment.

GP-10 Manganese Oxide Thin Films Electrodeposited Using a Potentiodynamic Method– Effects of Potential Sweep Rates on the Material Characteristics and the Pseudocapacitive Performance, ***M.T. Lee*** *(n5891119@ccmail.ncku.edu.tw), J.K. Chang, Y.T. Hsieh, W.T. Tsai,* National Cheng Kung University, Taiwan, *C.-K. Lin*, Feng Chia University, Taiwan

Manganese (Mn) oxide films were prepared by potentiodynamic electrodeposition in manganese acetate aqueous solution. Effects of the potential sweep rate on physicochemical characteristics of the deposited oxides were investigated. Surface morphologies and crystal structures of the oxides were examined using a scanning electron microscope and an X-ray diffractometer, respectively. X-ray photoelectron spectroscopic analyses were also performed to probe the chemical states. Moreover, electrochemical properties of the oxides were evaluated by cyclic voltammetry (CV). The analytical results suggested that the chemical state could be the most crucial factor that governed the pseudocapacitive performance of Mn oxide. A higher potential sweep rate led to a lower Mn valence and also to a higher hydrous state of the deposited Mn oxide. Accordingly, the oxide specific capacitance improved from 262 F/g to 337 F/g when the sweep rate of deposition potential increased from 100 mV/s t o 400 mV/s.

GP-11 SiO_x Protective and Barrier Coatings by Atmospheric Pressure Plasma Jet Deposition, ***P. Scopece*** *(scopece@civen.org), I. Kulyk, R. Sulcis, F. Marinello, A. Patelli*, CIVEN Association Venice, Italy

Atmospheric plasma superficial treatments represent an interesting alternative to vacuum systems in order to increase the on-line processing capability and to reduce costs. Industrial interests have already led to some applications in this field and in other cases results are very promising for future applications@super 1,2@@. In addition Atmospheric Pressure Plasma Jet (APPJ) can selectively treat specific part of a sample and can be used in the coating of 3D substrates. In this work we present the results on thin layers deposition of silica obtained by APPJ. By varying the deposition parameters silica coatings were studied both as anti-corrosive layers on iron samples and as gas-barrier layers for polypropylene substrates. Coating thickness have been evaluated by means of stylus profiler measurements and a good control at the nanometer scale has been obtained by varying the plasma jet speeds. Composition of the coatings was analyzed by mean of FT-IR and ATR spectroscopy, while coating morphology and modifications have been characterized by Scanning Electron Microscope (SEM). Corrosion behavior was characterized by mean of Electrochemical Impedance Spectroscopy (EIS) and a comparison with a salt spray test is presented. Finally, studies on gas permeation behavior of coated samples were performed by measuring the CO2 and O2 transmission rate and results related to the influence of the number of coating layers and to the precursor amount are also presented.

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GP-12 Multi-Layer Coatings of Aluminum for Corrosion Resistance and Electromagnetic Shielding, *J.-H. Chen, Y.-J. Fang, H.-T. Hsu,* ***T.-J. Yang*** *(yangtj@fcu.edu.tw)*, Feng Chia University, Taiwan

Since aluminum and its alloys are, by far, the most common of the light metals, naturally most of the information deals with aluminum finishing. Anodizing of aluminum provides better corrosion resistance, but causes electromagnetic interference (EMI) problem. An attempt has been made in this study to relate the corrosion resistance and EMI shielding of aluminum by multi-layer coatings. Non-porous barrier film and porous film of aluminum oxide can be produced by anodizing aluminum in 5 wt% oxalic acid solution at 15°C for 4 hours. Nickel ions were absorbed in porous film and reduced subsequently by hydrogen gas at 500°C for two hours. Nickel atoms distributed inside porous film were acted as active sites for electroless nickel plating with dimethyl amine borane (DMAB) reducing agent. As the surface of porous film was covered by Ni-B coating, multi-layer of Ni-B/Cu/Ni-P coatings was prepared by electroless plating method and responsible for EMI shielding. The measured decibels attenuation (dB) is greater than 30 in the frequency range 900-1800 MHz. The corrosion potential (Ecorr) and corrosion current (Icorr) are – 726.5 mV (vs. Ag/AgCl) and 0.3134 μmA/cm^2, respectively, based upon the electrochemical analysis of anodized aluminum. Microstructures of multi-layer coatings were examined by FE-SEM and EDS analyses.

GP-13 Characterization and Photocatalytic Activity of Composite V-TiO_2/ITO Thin-Film Electrode, *C.Y. Chang,* Mingdao University, Taiwan, ***M.Y. Chang*** *(mingyi@ms4.kntech.com.tw),* National Chung-Hsing University, Taiwan, *Y.H. Hsieh,* Mingdao University, Taiwan, *Y.S. Wang*, National Dong Hwa University, Taiwan

The object of this study is to modify the TiO2/ITO photocatalytic electrode with vanadium. The V-doped TiO_2/ITO photocatalytic electrodes were synthesized by sol-gel method and dip-coating method. Both TiO_2/ITO and V-TiO_2/ITO photocatalytic electrodes were characterized by Field emission scanning electron microscope（FE-SEM）, Electron spectroscopy for chemical analysis system（ESCA）, X-ray diffractometer（XRD）and Isoelectric point（pHIEP）. The photocatalytic and photoelectrocatalytic activities were evaluated by the degradation of methylene blue under different parameters. The results of the external analysis of SEM, ESCA and XRD, the particle size of the TiO_2/ITO photocatalytic electrode is about 30 nm and mainly anatase structure. The particle size of V-TiO_2/ITO photocatalytic electrodes is not uniform with the size range, from 10 nm to 100 nm. The 0.30VT-I photocatalytic electrode has the best ability both on the adsorpt ion and photocatalytsis. Even in the visible light system, the 0.30VT-I photocatalytic electrode has great photoactivity as same as in the UV light system. The V-doped TiO_2/ITO photocatalytic electrodes may convert N-type semiconductor into P-type semiconductor. Applied potential 0.300V would effectively increase the photoelectrocatalytic activity.

GP-14 Plasma Processing Efficiency in Pulse Plasma System, ***M. Zlatanovic*** *(jzlatanovic@beotel.net), I. Popovic*, Faculty of Electrical Engineering, Serbia

A significant advance in plasma surface processing of materials due to application of pulse power supply was introduced such as enhanced discharge stability, independent control of old and new process parameters, deposition and synthesis of new materials, enhanced reproducibility, better control of structure and morphology of deposited coatings and development of new surface treatment processes. Pulse plasma provides reliable operation but a new type of unhomogeneity due to periodic transition to the stationary state may result in non homogenous surface properties caused by "plasma spreading" effect. Number of adjustable system parameters is quite large and their influence on process efficiency is often opposite. It was found that the increase of pulse frequency results in decreased process efficiency, but on the other hand it leads to decreased gas discharge instabilities occurrences and provides more stable operation. From detailed analyses of plasma nitriding pro cess it was concluded that the compromise between the process efficiency and process stability is necessary. Based on the measurements, a variable voltage controlled impedance gas discharge electrical model was introduced. The model include different discharge electrical behavior during the process of the gas discharge spreading over the cathode surface and the process of gas discharge transition to the stationary state. The simulation results have shown that the dynamic as well as static gas discharge properties influence the shape of the pulse plasma electrical system response. The overall process efficiency was estimated through the optical emission signal analysis, since the data on the active species generation responsible for physical and chemical processes in the discharge are stored in optical emission signals. The stationary state concentration of the excited active species for nitriding process cannot be attained in the case of high frequency and low duty cycle puls ed plasma, which resulted in less efficient chemical processes on the cathode surface. Charge particles generation is related to pulse power characteristics and is strongly influenced by generator properties, interconnection line and the most significantly, on the vacuum chamber properties including workpiece shape and physical properties. The results of thermochemical plasma processing of two steel grade samples in nitrogen containing discharge were presented.

GP-15 Effects of Pressure on Physical Properties of Amorphous Carbon Film, ***K. Kayama*** *(hokuto.sultan04@gmail.com), M. Watanabe,* Keio University, Japan*, H. Kodama,* Kanagawa Academy of Science and Technology, Japan*, T. Suzuki*, Keio University, Japan

It is expected that amorphous carbon films will be used for mechanical components especially to sliding parts because of their excellent properties such as high hardness, high wear resistance and low friction. In general, amorphous carbon films are synthesized under low pressure (under 10 Pa), but this technique costs much for vacuum devices etc. Therefore, it is necessary to realize low-cost and high-speed synthesis in order to expand application of the film. In this study, we designed and set up plasma CVD equipment where carbon films are synthesized under sub-atmospheric pressure (1 kPa -101 kPa). In order to synthesize the films, plasma was generated by dielectric-barrier discharge method and acetylene and nitrogen mixture gas were introduced under nitrogen environment. The surface of the films was observed by SEM and AFM, the hardness of the films was measured by nano-indentation test.

GP-16 Control of Plasma Non-Uniformity in Large Area / Very High Frequency Capacitive Discharges, ***S.K. Ahn*** *(notask@kaist.ac.kr), B.K. Na, H.Y. Chang*, Korea Advanced Institute of Science and Technology, Korea

To control the plasma non-uniformity induced by the electro-magnetic effects in large area / very high frequency capacitive discharges, various power feeding methods were tested in a rectangular capacitive discharge driven at 90 MHz (20 x 30 cm electrodes). Spatial electron density distributions for the power feeding methods were measured using spatially resolved rf compensation Langmuir probe. From the measurement, we found that plasma uniformity can be controlled by the number of power feeding points and spatial position of each feeding points. In addition, we also found that phase differences between each feeding points significantly affect the plasma uniformity. Variation of the plasma uniformity depending on the power feeding methods can be understood as a result of changes of electromagnetic field distribution between electrodes.

GP-17 Photoelectrocatalytic Degradation of Sodium Oxalate by TiO_2/Ti Thin-Film Electrode, *T.C. Cheng,* Mingdao University, Taiwan*, Y.H. Hsien,* National Chung-Hsing University, Taiwan*, K.S. Yao, Y.Y. Chen,* ***Y.C. Yen****, C.Y. Chang (cychang@mdu.edu.tw)*, Mingdao University, Taiwan

The TiO_2 thin film was deposited on the titanium plate by chemical vapor deposition (CVD) method. The photoelectrocatalytic degradation of sodium oxalate was investigated by prepared TiO_2 thin film reactor with additional electric potential at 365 nm radiation. The batch system was chosen in this experiment and the controlled parameters were pH, different supporting electrolytes, applied potential and initial $Na_2C_2O_4$ concentration were examined and discussed. The results revealed that the additional applied potential in photocatalytic reaction could prohibit recombination of electron/hole pairs but the photoelectrocatalytic effect was decrease when the applied potential was over 0.25 V. In additional, the various electrolytes increased the removal of sodium oxalate while the sodium sulfate (Na_2SO_4) had the most significant effect on photoelectrocatalysis. The better photoelectrocatalytic degradation of sodium oxalate occurred at pH3 when comparing the pH influence.

GP-18 Effect of Additives and pH in H_2O_2-Based Slurry on Cu-CMP Corrosion Behavior, ***C.-C. Hung****, W.-H. Lee, S.-C. Chang,* National Cheng Kung University, Taiwan*, Y.-D. Juang, Y.-L. Wang (ylwang@tsmc.com)*, National University of Tainan, Taiwan

Copper metallization is achieved by combining dual damascene process with tantalum base diffusion barrier layer with chemical mechanical polishing (CMP) to remove excess metals. For an effective application of CMP to 65-nm device fabrication, a complete investigation of the polishing behavior of copper continues to be important because CMP is a highly complicated process. The fundamental mechanism of chemical and mechanical phenomena between the wafer and the slurry is still not fully understood. In this study, effect of additives and pH of H_2O_2-based slurry on copper corrosion behavior was investigated by dc polarization techniques. The results reveal that different additives and pH value had a strong influence on the electrochemical behavior of copper corrosion.

GP-19 Effect of Plasma Nitriding Pre-Treatment on D2 Steel Surface Deformation Under Nitride Based Multilayer AIP Coating, ***T. Okude*** *(okude.toru@kobelco.com), D. Yasunaga, K. Yamamoto,* Kobe Steel Ltd., Japan*, T. Kashi, K. Imai,* Koshuha All Metal Service CO., Ltd., Japan*, T. Takazawa, J. Yoshida*, Nippon Koshuha Co,. Ltd., Japan

Nitride based AIP coatings are used as a surface treatment of stamping molds for ultra high-tensile steel sheet. Comparing with the conventional VC, TiC forming thermal diffusion processes, thermal distortion of the molds is significantly minimized during the lower temperature AIP processes. In some cases, however, deformation of the mold surface under the AIP coating occurs during the steel sheet stamping that causes film cracking resulting in insufficient life time. Plasma nitriding processes were applied to D2 steel samples as a pre-treatment of nitride based multilayer AIP coating (BELCOAT-SS[1]) in order to develop duplex technology that prevents the deformation of the D2 steel mold surface under the AIP coating. Scratch test was used to investigate the deformation of the substrate surface that causes AIP film cracking. Temperature, bias voltage and treatment time of the plasma nitriding processes were controlled to optimize the surface property. Maximizing nitroge n penetration depth and minimizing iron nitride formation are found to be essential to increase film cracking critical load in the scratch test that was improved up to 160 %.

@suiper 1@] http://www.kobac-us.com/belcoat.html

GP-20 Hydrophobic Film Deposition from Remote HMDSN Atmospheric Pressure Plasma Jet, ***C. Huang*** *(chunhuang@saturn.yzu.edu.tw), C.-H. Liu, S.-Y. Wu*, Yuan Ze University, Taiwan

Hydrophobic films were deposited at room temperature by remote atmospheric pressure plasma jet (APPJ), using hexamethyldisilazane (HMDSN) as monomer and argon (Ar) as carrier gas, have been investigated for the surface properties as a function of plasma operational parameters including in RF plasma power, nozzle distance, and monomer flow rate. The remote APPJ deposited films have been analyzed by static contact angle measurement, fourier transform infrared spectroscopy (FTIR), UV–vis spectrometer, and atomic force microscopy (AFM). The hydrophobicity of remote APPJ deposited films have been studied and related to the chemical composition and to the surface morphology of the plasma coated SiOx layers. It was detected that remote APPJ deposited films obtained water-repellent properties by static contact angle measurement. From FT-IR analysis, it resulted that remote APPJ deposited films become more inorganic as the monomer flow rate increased. UV–vis spectra, detected in the range of 300–800 nm, examined the improved transparency in the visible and increased absorption in UV region. The AFM analysis confirmed the hydrophobic features of the increased surface roughness of remote APPJ deposited films.

GP-21 Characterization of Cuprous Oxide Films by Using Atmospheric Pressure Nitrogen Plasma Torch, *H.-Y. Chen (hychen@cc.kuas.edu.tw),* ***M.-W. Tsai****, C.-H. Tsai*, National Kaohsiung University of Applied Sciences, Taiwan

The Cu films were deposited onto glass by magnetron sputtering, after that the films were treated by using atmospheric pressure plasma torch with different nitrogen/oxygen ratios at 500@supper o@C for 10 min. X-ray diffraction patterns showed the as-deposited Cu films were (111) and (200) orientations. The cuprous oxide peaks appeared below nitrogen plasma torch containing 100 ppm oxygen, while additional cupric oxide peaks showed up above 1% oxygen. The as-deposited Cu films were nano-crystallites feature and the large grains were found after plasma treatment. The optical bandgap of cuprous oxide phase was 2.3 eV and 2.1 eV for cupric oxide phase. The resistivity of cuprous oxide was 2.9 Ω-cm and cupric oxide phase was 0.9 Ω-cm, which are consistent with the literature reports.

GP-22 Mechanical Properties Evaluation of Chromized Tungsten Carbide-Cobalt Hardmetals, ***J.-L. Li****, J.-W. Lee (cwlee@mail.tnu.edu.tw), Y.-T. Lin, C.-J. Wong*, Tungnan University, Taiwan

Chromizing treatment has shown promising potential applied in mechanical industry due to the excellent corrosion and mechanical properties provided by this process. However, the application of chromizing process on the tungsten carbide-cobalt hardmetals has never been reported elsewhere. In this work, the pack chromization process has been adopted on tungsten carbide-cobalt hardmetals to produce a chromium contained hard surface layer. The SEM and XRD were employed to analysis the surface and cross-sectional morphologies and crystalline phases of chromized materials, respectively. The mechanical properties of the chromized tungsten carbide-cobalt hardmetal were evaluated by a nanoindenter, Rockwell C hardness tester and a scratch tester. It was observed that the chromizing layer was around 2 to 10 μm in thickness, which consisted of the $(Cr,Fe)_2N_{1-x}$ and $(Cr,Fe)_{23}C_6$ phases in the outer layer and an $(Cr,Fe)_{23}C_6$ phase in the inn er layer after the chromization process held at 950 20°C for 1 to 9 hrs. The hardness of the chromized hardmetals was improved effectively. The adhesion quality of the chromized tungsten carbide-cobalt hardmetal was influenced by the thickness of the surface layer.

GP-23 First Results of the New Digital (Pulsed) Electron Beam - Physical Vapor Deposition Process with Digital Coating Capabilities, ***R. Edinger*** *(edinger@pavac.com)*, PAVAC Industries Inc., Canada

In 2006 a new Pulsed Electron Beam – Physical Vapor Deposition process was presented at COM2006 in Montreal that would allow users to digital program the chemistry of coatings. In September 2006 the production of the coating machine was started. In 2008 the machine was commissioned and the testing started. The pulsed LASTRON electron beam system is configured to program an almost unlimited number of combinations for elements and therefore create new coatings. The technology is targeted for researchers and companies who are in need to develop new coatings by applying almost unlimited coating combinations. This process is based on the ability to pulse the electron beam synchronized to a number of input variables. The electron beam system can pulse at frequencies up to 50kHz with a minimum pulse width of 500ns. The electron beam generator works at voltages up to 100kV; first tests where conducted at 70 and 80kV.

GP-24 Synthesis of SiO_x Films on Polycarbonate Substrates under Atmospheric Pressure, ***M. Noborisaka*** *(okura0306@gmail.com),* Keio University, Japan*, H. Kodama,* Kanagawa Academy of Science and Technology, Japan*, T. Suzuki*, Keio University, Japan

In order to use polymer materials for vehicular windows, it is necessary to improve hardness, scratch resistance and weatherability. In general, hard thin films are coated on top of the surface such as SiO_x films which is highly transparent and have scratch resistance. Needless to say, for the low cost, it is necessary to realize SiO_x synthesis at high-speed and low-cost to large area. The atmospheric pressure plasma CVD technique using dielectric barrier discharge (DBD) is an ideal and suitable method for this purpose. In this study, we synthesized SiO_x films on PC substrate using trimethylsilane and oxygen mixture gas for process gas. Hardness of the films was measured by nano indenter and structure was analyzed by X-ray photoelectron spectroscopy and Fourier transform infrared spectroscopy.

GP-25 Effect of Gas Mixture of Plasma Post-Oxidation on Corrosion Properties of Plasma Nitrocarburised AISI 4130 Steel, ***N. Mandkarian****, F. Mahboubi (mahboubi@aut.ac.ir)*, Amirkabir University of Technology, Iran

Plasma post-oxidizing is a process to improve the corrosion resistance of nitrocarburised parts. In this study, the effect of gas mixture of post-oxidation process on corrosion resistance of AISI 4130 plasma nitrocarburised steel has been studied. Plasma nitocarburizing was carried out at 520ºC for 5 h in an atmosphere containing 49 vol% nitrogen, 49 vol% hydrogen and 2 vol% carbon dioxide. The nitrocarburised samples were post-oxidized at 450ºC for 1 h under different O_2:H_2 ratios (5:1, 1:1,1:3, 1:5 and 1:7). The treated samples were studied using XRD, SEM, surface roughness measurement, micro-hardness and potentiodynamic methods. X-ray diffraction patterns revealed that with decreasing the O_2:H_2 ratio from 5:1 to 1:5, the amount of magnetite phase and the corrosion resistance of the samples were increased. It was seen that with increasing the O_2:H_2 ratio, the thickness of oxide layer was increased and the surface roughness was decreased. Furthermore, a model for oxide formation during plasma post-oxidation is proposed.

GP-26 Low Temperature Treatments for Improving Sputtered ZrO_x Dielectric by High Pressure O_2 and O_3 Passivation, ***S.-C. Chen*** *(scchen0921@gmail.com), T.-C. Chang, H.-H. Su, C.-C. Huang, P.-C. Yang, H.-C. Huang, D.-S. Gan, N.-J. Ho*, National Sun Yat-Sen University, Taiwan

In this study, we proposed the low temperature (150°C) oxygen (O_2) and ozone (O_3) treatments with high-pressure of 1000 psi to improve electrical properties of sputtered zirconium-oxide (ZrO_x) thin-film dielectric. Based on X-ray photoelectron spectroscopy (XPS) analyses, the increase of bonding energy of Zr-O and after O_2 and O_3 treatments was attributed to the reduction of traps in dielectric film. In addition, the leakage currents after treatments were also can be suppressed because the leakage mechanism was transferred from trap-assisted tunneling to Schottky-Richardson emission. From the experimental results, O_3 produced by UV light illumination in O_2 ambient has the superior passivation ability than O_2 resulting in the better improvements for electrical characteristics.

GP-27 Improvement of Properties of Indium Tin Oxide Thin Film using Supercritical Carbon Dioxide Treatment, ***S.-W. Tsao*** *(d943050011@student.nsysu.edu.tw), T.-C. Chang, C.-C. Huang, H.-H. Wu, S.-C. Chen, Jin Lu, H.-C. Huang, D.-S. Gan, N.-J. Ho*, National Sun Yat-Sen University, Taiwan

Indium tin oxide (ITO) thin films, for the top electrode of solar cells, must be with low resistivity and high transparency. In the study, we proposed a new method to improve the properties of ITO using supercritical carbon dioxide. The structure, morphology and electro-optical characteristics have been analyzed by X-ray diffraction, scanning electron microscopy, four-point electrical measurements and spectrophotometry, respectively. Then, the chemical composition of the ITO was measured by using x-ray photoelectron spectroscopy (XPS). After supercritical carbon dioxide treatment, the conductivity was improved and the transmittance was also increased in the visible region. In addition, the peak energy values of the XPS spectra were shifted after treatment.

GP-28 Effect of Low-Temperature Supercritical Fluid Technology Treatment on the Performance of ZnO TFTs, ***M.C. Chen*** *(d962030002@student.nsysu.edu.tw), T.-C. Chang,* National Sun Yat-Sen University, Taiwan*, G.-W. Jhang, Y.-H. Tai*, National Chiao Tung University, Taiwan

In this paper, the method of the low temperature supercritical fluid technology treatment was successfully applied to improve the electrical characteristics of sputtered ZnO TFT. The experimental results indicated that on/off current and threshold voltage of the device were greatly improved. According to X-ray photoelectron spectroscopy (XPS) analyses, the improvements were attributed to the increase of binding energies of Zn-O bonds and the reduction of traps at the grain boundary in ZnO thin films. In addition, the structural, optical properties and surface topography of ZnO thin films using X-ray diffraction (XRD), Photoluminescence (PL) and atomic force microscopy (AFM) were also investigated.

GP-29 Surface Modification with Borane Clusters: Superior Protection of Metal Surfaces Against Corrosion, ***T. Base*** *(tbase@iic.cas.cz),* Institute of Inorganic Chemistry of the Academy of Sciences of the Czech Republic, v.v.i., Czech Republic*, M.G.S. Londesborough,* Institute of Inorganic Chemistry of the Academy of Sciences of the Czech Republic, v.v.i.*, J. Bould*, Institute of Inorganic Chemistry of the Academy of Sciences of the Czech Republic, v.v.i., Czech Republic

The tarnishing of silver causes technical problems for various hi-tech applications such as, for example, silver telescopic mirrors. Recently, we have reported on the interactions between thiolated carborane clusters with high dipole moments and gold surfaces.[1] In this contribution particular attention will be paid to the following carboranethiol species: 1,2-$(HS)_2$-1,2-$C_2B_{10}H_{10}$ (compound 1) and 9,12-$(HS)_2$-1,2-$C_2B_{10}H_{10}$ (compound 2), and their use as modifiers of silver coatings. In a recent study of ours, modified silver surfaces were exposed to H_2S and their corrosion was monitored using several techniques including UV-Vis and X-ray photoelectron

spectroscopies, and Rutherford Back Scattering. We have shown that compound 2 can be effectively used as a protective mono-molecular layer to provide silver surfaces with enhanced stability against corrosion.[2] Both derivatives when immobiliz ed on silver surfaces are very stable towards heating and remain unchanged up to 400°C. Additionally they exhibit remarkable stability towards X-ray radiation. The stability of these species on the silver surface is the result of their unique electronic structure and molecular geometries. Compounds 1 and 2 have dipole moments of 4.1 D and 5.9 D respectively, and this character will be discussed in association with their behavior during the corrosion process. We will also provide a basis for the comparison of compounds 1 and 2 with organic thiols.

Aside to the chemical protection of surfaces, modification with boron clusters can also add various other advantageous features to surfaces. We have shown recently that several metallaborane clusters can selectively and reversibly uptake and release small molecules of gases such as O_2, CO, SO_2,[3] and others, and can potentially be used as colorimetric sensors. Additionally, larger boron clust e rs, such as syn and anti isomers of $B_{18}H_{22}$,[4] can be used as fluorescent labels. Results describing our effort to immobilize these functional molecular clusters will also be presented.

New Horizons in Coatings and Thin Films
Room: Town & Country - Session HP

Symposium H Poster Session

HP-1 Impact of Strain Engineering on Nanoscale Strained Si NMOSFETs with a Silicon-Carbon Alloy Stressor, ***W.-C. Wang***, National Chung Hsing University, Taiwan, *C.-C. Lee,* Taiwan Semiconductor Manufacturing Company, Taiwan, *J. Huang, S.-T. Chang (stchang@dragon.nchu.edu.tw)*, National Chung Hsing University, Taiwan

The stress distribution in the Si channel regions of a Silicon-Carbon source/drain NMOSFETs with various widths were studied using 3D ANSYS simulations. The mobility enhancement was found to be dominated by the tensile stress along the transport direction and compressive stress along the growth direction in wide width devices. Stress along the width direction was found to have the least effect on the drain current in wide width cases. Stress along the width direction slightly degraded the mobility gain in the narrow width regime, contributing a slight degradation of the total drive current gain in the smaller width region. The compressive stress along the vertical direction perpendicular to the gate oxide contributes significantly to the mobility enhancement and cannot be neglected in nanoscale NMOSFETs. The impact of width on performance improvements such as the drive current gain was also analyzed using 3D TCAD simulations.

HP-2 Growth and Characterization of ZnO Nanoflowers, ***J.H. Park***, *S. Prikhodko, M. Pozuelo,* University of California - Los Angeles, *S.D. Sitzman,* Oxford Instruments America, *S. Kodambaka (kodambaka@ucla.edu)*, University of California - Los Angeles

ZnO is a direct wide band gap (3.37 eV) semiconducting piezoelectric material with potential applications in a wide variety of areas including radiation-hardened electronics, optoelectronics, spintronics, piezotronics, and catalysis. Here, we report the formation of nanoscale hexagonal dendrites ("flowers") during chemical vapor deposition of ZnO on Au-coated Si(100) and SiO_2/Si(100) substrates. All our growth experiments are carried out in a three-zone 2" tube furnace using high-purity (99.9 %) metallic Zn powder (average particle size of ~ 15 μm) and purified air as Zn and O sources, respectively. We observe highly regular ~1-μm-sized flower structures on 100-nm-thick Au-coated substrates maintained at 340°C while flowing 200 Torr of argon/air gas mixture at 200/2 sccm. We characterized the as-grown structures in situ using scanning electron microscopy coupled with electron backscattered diffraction (SEM-EBSD) and energy dispersive X-ray spe ctroscopy (EDS), and ex situ using transmission electron microscopy (TEM). From SEM imaging, we estimate the thickness of these flowers to be ~ 52 ± 5 nm. EDS data suggests that the flowers are primarily composed of Zn and O. Selected area electron diffraction and EBSD patterns acquired from individual ZnO flowers indicate that they are single crystals with a wurtzite (hexagonal) structure. We find that ZnO flower growth depends sensitively on the growth temperature, gas environment, and Au film thickness. Based upon these results, we suggest a tentative mechanism for the growth of these flower structures.

HP-3 Cryogenic Temperature Characteristic of 65nm n-MOSFET's under Uniaxial Tensile Stress, ***Y.-J. Kuo*** *(d943050018@student.nsysu.edu.tw), T.-C. Chang*, National Sun Yat-Sen University, Taiwan

Strained silicon is a novel technique to enhance carrier mobility and drain current in nanometer MOSFET's. In this experiment, the electrical characteristic of strained MOSFET's was measured at widely temperature. The device under test was used gate length 65 nanometer n-MOSFET's. In order to get uniaxial tensile stress from the channel, the device was bent along channel length. By this method, 19% increase in drain current was observed. The drain current of strained device is higher than unstrained device between 300K-150K, this phenomenon was attributed to reduce phonon scattering. When the temperature keeps dropping from 150K to 77K, drain current represented a reverse characteristic. A detailed investigation in two different behavior has been fully discussed in this paper.

HP-4 Reliability Study of Through-Silicon via (TSV) Copper Filled Interconnects, ***A. Kamto*** *(akamtotegueu@bama.ua.edu),* The University of Alabama, *Y. Liu, L. Schaper,* University of Arkansas, *S.L. Burkett*, The University of Alabama

Through-silicon vias (TSVs) have been extensively studied because of their ability to achieve chip stacking for enhanced system performance. The fabrication process is becoming somewhat mature, however, reliability issues need to be addressed in order for an eventual transition from laboratory to production. In our laboratory, vias with tapered sidewalls are formed through a modified Bosch process using deep reactive ion etching (DRIE). Vias are; lined with silicon dioxide using plasma enhanced chemical vapor deposition (PECVD); followed by sputter deposited titanium barrier and copper seed layers before filling with a reverse pulse copper electroplating process. Following attachment of the process wafer to a carrier wafer, the process wafer is thinned from the backside by a combination of mechanical methods and reactive ion etching (RIE). Fabricated vias are subjected to thermal cycling, using a Physical Property Measurement System (PPMS), with temperature ranging from -25°C to 125°C; and the resistance is measured as a function of temperature. For long via chains, resistance changes somewhat linearly upon cycling for temperatures above room temperature; and values change erratically at lower temperatures. This test method forms the basis of reliability studies, to be presented in this paper, in which via chain size, temperature ramp rates, temperature regions, and number of cycles are varied.

HP-5 Properties of Pure and Silver Doped Ti_3SiC_2 Films Deposited by HiPIMS, *R. Bandorf (ralf.bandorf@ist.fraunhofer.de), M. Schmidt, H. Gerdes,* Fraunhofer Institute for Surface Engineering and Thin Films IST, Germany, ***G. Mark***, MELEC GmbH, Germany

$M_{n+1}AX_n$-phase materials are used as electrical conductive ceramic. Especially Ti_3SiC_2 is synthesized as wear resistant contact material. High Power Impulse Magnetron Sputtering (HiPIMS) opens new horizons tailoring the resulting film properties. In contrast to the usually applied DC sputtering for HiPIMS films an improved wear resistance, a reduced resistivity, and a modified structure were observed. For further improvement of the conductivity the films were doped with silver. Since the HiPIMS process realizes a glassy structure besides the reduction of resistivity also an improved corrosion resistance is expected.

HP-6 Effects of Alloying Elements(Zr,Hf) on the Nanopore and Nanotube Formation of Ti-30(Nb,Ta) Alloys, ***H.-C. Choe*** *(hcchoe@chosun.ac.kr), Y.-M. Ko,* Chosun University, Korea, *W.A. Brantley*, Ohio State University

The two-step anodization techniques have been used to improve the bone tissue integration. The electrochemical formation of ordered nanopore and nanotube has been reported for Ti anodization in fluoride-containing acid electrolytes at moderate voltage. Nanopore and nanotube formation on the Ti oxide is important to improve the cell adhesion and proliferation in clinical use. In this study, nanopore and nanotube formation of Ti-30Nb-xZr(Hf) and Ti-30Ta-xZr(Hf) alloy have been investigated using various condition of , nanopore and nanotube formation methods. Ternary Ti-30Nb-xZr(Hf)(x=3, 10, 15wt%) and Ti-30Ta-xZr(Hf) (x=3, 10, 15wt%) alloys were prepared by using high purity sponge Ti (G&S TITANIUM, Grade. 4, USA), Ta, Zr, Hf and Nb sphere (Kurt J. Lesker Company, 99.95% wt.% in purity). Two kinds of Ti alloys prepared using the vacuum arc melting furnace. In order to homogenize, solution treatment was carried out for 1hr at 1050°C in an argon atmosphere, followed by wat er quenching to stabilize the β phase. Microstructures of the alloys were examined by optical microscopy (OM, OLYMPUS BM60M, JAPAN) and scanning electron microscopy (SEM, HITACHI-3000, JAPAN). Two-step anodizing was used for surface modification; nanopore formation was performed by potentiostatic experiment using a conventional two electrode configuration at 180V in 1 M H3PO4 electrolyte at room temperature. Nanotube

formation were carried out with a conventional three-electrode configuration with a platinum counter electrode and a saturated calomel(SCE) reference electrode. Experiments were performed in 1M H3PO4 with small additions of NaF(0.1-0.8wt%). All experiments were conducted at room temperature. Electrochemical treatments were performed by using potentiostat (EG&G Co, 362, U.S.A). The electrochemical treatment consisted of a potential ramp from the open-circuit potential to an end potential at 10 V with a scan rate of 500mV/s followed by holding the sample at 5-10V for 30min-120min. The nanopores and nanotubes could be controlled by two-step anodizing. It was depended on the composition and surface oxide films.

HP-7 Nanostructual Conductivity and Structural Information of Ruthenium Dioxide Films, *Y.C. Liang (yuanvictory@gmail.com),* Chienkuo Technology University, Taiwan, ***Y.C. Liang***, University of Southern California

The effects of oxygen partial pressure on the crystallographic and opto-electronic properties of Zr-doped In_2O_3 (Zr-In_2O_3) films by rf magnetron sputtering were initially studied. The Zr-In2O3 film grown in an Ar atmosphere has a preferred orientation of (222). The results of X-ray diffraction show that as the oxygen content of the sputtering gas increases, the crystallographic structure in the Zr-In_2O_3 film becomes more random. The surface of Zr-n_2O_3 film becomes rougher as the oxygen partial pressure decreases. The film grown at a pure Ar atmosphere has a smaller O/(Zr+In) atomic ratio compared with the values of the films grown with an addition of oxygen gas. The resistivity is minimized to $3.51\times10_4$ Ωcm when the Zr-n_2O_3 film is grown in an Ar atmosphere and the average transmittance in the visible light region is ~85%. The optical band gap decreases as the oxygen partial pressure increases.

HP-8 Nano-Structural Titanium Dental Implants Synthesized by Sand-blasting Etching and Plasma Etching Techniques, *Y.-Y. Chang (yinyu@mail2000.com.tw),* ***Ya-Ting Hsu***, *Y.-C. Yang, H.-I. Kao, C.-P. Lai*, Mingdao University, Taiwan

In this study, nano-structured titanium dioxide films (TiO_2) were deposited on dental biomedical implant titanium materials by using Sand-blasting etching (SLA) and plasma etching/deposition techniques. A Ti interlayer coating was deposited on the sand-blasted titanium by using cathodic arc evaporation. Following anodization and plasma etching were conducted to make nano-structured TiO_2 on the Ti samples. Different nano-structures, such as nono-webs and nano-rods, were formed on the titanium substrates. The crystallographic texture of the deposited coatings was characterized using glancing incidence X-ray diffraction (GIXRD), while the surface morphorlogy was studied using field emission scanning electron microscopy (FESEM) equipped with EDS. XPS was used to characterize the bonding structure of the deposited coatings. Cell compatibility test was conducted to explore cells affinity after the surface modification of titanium. Osteoblast-like OS cells were cul tured on the nano-structured TiO_2 coated titanium samples after 1 day of incubation. An ELISA instrument was used to conduct the OS cell adhesion assay, proliferation assay and alkaline phosphatase (ALP) activity analyses. The OS cell surface morphology was obtained by FESEM observation. The nano-structured titanium dioxide films (TiO_2) can improve the osseointegration of the titanium dental implants.

HP-9 Gas Sensing Properties of Hollow tin (IV) Oxide Nanostrucutred Films by Electrophoretic Deposition, ***S.-C. Wang*** *(scwang@mail.stut.edu.tw), P.-J. Hu,* Southern Taiwan University, Taiwan, *R.-K. Chiang,* Far East University, Taiwan, *C.-Y. Chen, C.-K. Lin*, Feng Chia University, Taiwan

In this study, the hollow SnO_2 nanostructured powder was synthesized by thermal decomposition method and electrophoretic deposited on a patterned Al_2O_3 substrate for the gas sensing properties measurement. The phases and microstructure of the powders were characterized by the XRD, SEM, TEM and BET. Tin oleate complex and 1-octadecanol were used as the precursors, which are thermal decomposed and assembled as octahedron hollow $Sn_6O_4(OH)_4$ agglomerate powder at the temperature of 200°C in N_2 atmosphere. After heat treatment at 700°C for 1h in air, the powder was transformed to crystalline SnO_2 nanopartilce in size of 50 nm and the hollow octahedron agglomerate still remains. The hollow SnO_2 nanostructured electrodes with promising gas sensing properties were prepared by electrophoretic deposition using an isopropyl alcohol solution. The nitrogen-adsorption and desorption isotherms and the relatively gas se nsing properties of CO gas are characterized and discussed in the text.

HP-10 Effect of Seed-Layer Films on the Growth of ZnO Nanowires via Hydrothermal Technique, ***S.-N. Bai*** *(snbai@ctu.edu.tw)*, Chienkuo Technology University, Taiwan

ZnO nanowires have been prepared on the ZnO film-coated silicon substrates (100) by a hydrothermal method. The ZnO films were deposited by spin-coating polymeric precursors. Then, the ZnO thin films were annealed in air at various temperatures ranging from 350 to 850°C for 1h. On the ZnO thin films, the well-oriented nanowires were synthesized by a solution growth process using zinc nitrate and hexamethylenetetramine in aqueous solution. The ZnO nanowires were characterized by different structural and optical techniques, including the X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and cathodoluminescence (CL). The XRD analysis with grazing incidence and SEM results indicate that the ZnO nanowires are single crystalline structure with preferential orientation along the c-axis direction with a full-width at half-maximum (FWHM) less than 0.5°. The TEM analysis further verifies that the ZnO nanowires are highly preferr ed grown along the (002) crystal plane. The spacing between adjacent (002) lattice planes is estimated as 0.52 nm. The optical properties of the nanowires were measured using CL for the ZnO seed-layer films annealed in air from 350 to 850°C for 1h. The CL spectra in the visible spectrum exhibit one strong broad deep-level emission band that may be due to the intrinsic or extrinsic defects. It can be observed that the ZnO nanowires synthesized on the seed-layer films, which annealed at different temperatures, show different optical behaviors. The dependence of the optical characteristics on the ZnO nanowires with various substrate properties is also discussed.

HP-12 Study of Relationship Between DC Power and DLC HCPIIP Process Gases as it Affects the Initiation and Sustaining of Plasma, ***G. Saha*** *(gsaha@hyperiontechnologies.com), L. Glenesk*, Hyperion Technologies Inc., Canada

A major problem of operating machinery and equipment in the oil sands is the unpredictable failure from operating in this highly aggressive environment. One of the significant causes of that problem is premature material wear. The primary objective of the study is to substantially increase the wear resistance and hence, wear life of these components, thereby increasing process efficiency by reducing maintenance and wear damage downtime and costs. An approach to minimize this wear is the use of protective coatings. Hollow cathode plasma ion immersion processing (HCPIIP) diamond-like carbon (DLC) coating technique is used for the project. The HCPIIP technology is particularly well-suited to the application of protective coatings to the internal surfaces, long and tubular components and components with complex internal geometries where a line-of-sight (LOS) application of coating materials is impossible. As part of the HCPIIP DLC process parameter optimization, this research reports on the experimental tests determined to find out a minimum power setting for the initiation and maintenance of a sustained plasma in the HCPIIP process of DLC coating. As the relationship affects the creation and sustaining of HCPIIP plasma by ensuring the maximum current flow and optimum pressure setting inside a workpiece, it is considered to be a major development towards finding an optimum solution for wear resistant coating properties. The study will lead to advance the knowledge by determining the importance, statistical range, and interplaying capabilities of the HCPIIP process parameters and, ultimately, the writing of the recipe.

HP-13 Resistive Switching Characteristics and Mechanism of $SrZrO_3$ Thin Films, ***M.-H. Lin***, *M.-C. Wu, S.-W. Jan, Y.-H. Huang,* National Chiao Tung University, Taiwan, *C.-H. Lin,* Winbond Electronics Corporation, Taiwan, *T.-Y. Tseng (tseng@cc.nctu.edu.tw)*, National Chiao Tung University, Taiwan

The resistive switching behaviors of rf-sputtered $SrZrO_3$ (SZO) thin films were investigated in this paper. The memory states of Al/$SrZrO_3$/$LaNiO_3$/Pt structure can be switched between high resistance state (HRS) and low resistance state (LRS) by applying voltage signal, showing reversible and bistabe resistive switching characteristics. The SZO resistive thin film memory device exhibits lower switching voltages than doped SZO thin film device, reducing the power consumption during device operation. The retention characteristics of both HRS and LRS are stable up to 10^6 s at room temperature (RT) and 85°C. After applying the successive read voltage stress (-0.3 V), the resistive ratio of HRS and LRS remains nearly 10^3 over 12000 s at RT and 85°C. The conduction mechanisms of HRS and LRS are dominated by Frenkel-Poole emission and Ohmic conduction, respectively. As a result, the resistive switching from HRS to LRS might be ex plained due to the formation of conducting filaments constituted by the arrangement of the defects in the bulk SZO thin film. On the contrary, conducting filaments are ruptured by Joule heating effect, leading to the memory state switching back to HRS. The undoped SZO memory device with its superior memory characteristics is a promising candidate for next generation nonvolatile memory application.

HP-14 Surface Morphology Changes of Anodized Ti-x(Nb,Ta) Binary Alloys for Dental Implant, ***K. Lee***, *H.-C. Choe (hcchoe@chosun.ac.kr), Y.-M. Ko,* Chosun University, Korea, *W.A. Brantley*, Ohio State University

Commercial pure titanium(Cp-Ti) and Ti-6Al-4V alloy have been widely used for orthopedic implant materials and dental implant materials because of its excellent combination of biocompatibility, corrosion resistance and mechanical properties. However, the Ti-6Al-4V alloy is currently utilized and should be replaced, since the release of Al and V ions causes long-term health problems. And it can also lead to resorption of adjacent bone tissue due to the great elastic modulus difference between the implant and bone. Thus, there are efforts for developing new titanium alloys with non-toxic elements. Nb and Ta are found to reduce the elastic modulus when alloyed with titanium in certain preferred quantities. Recently, titanium oxide layer has been used for improving the biocompatibility of implants. The advantage of using titanium oxide layer is that it can be grown directly on the Ti and Ti alloys surfaces, by cost-effective techniques such as anodic oxidation. In this stud y, electrochemical characteristics of anodic oxide layer formed on titanium binary alloy surface have been investigated. Titanium oxide layers were grown on Ti-XTa and Ti-XNb(X=10, 20, 30 and 40 wt%) alloy substrates using phosphoric acid electrolytes. For this study, the Ti-Ta and Ti-Nb alloys were manufactured by arc melting on a water-sealed copper hearth under an argon gas atmosphere with a non-consumable tungsten electrode. These specimens were melted six times by inverting the metal for homogeneous structure. Ti-Ta and Ti-Nb alloys were homogenized in argon atmosphere at 1000℃ for 24h followed by a rapid quenching in ice water. The samples were incrementally polished by utilizing 120 grit emery paper down to 2000 grit emery paper. The polished and cleaned binary alloy disks were anodized in solution containing typically 1 M H_3PO_4 at room temperature. A direct current(D.C) power source was used for the process of anodization. For electrochemical measurement s, the cell consisted of conventional three-electrode configuration with Pt rod and a saturated calomel electrode (SCE) as the counter and reference electrode, respectively. All experiments were carried out in 0.9 % NaCl solution at 36.5±1℃. The corrosion resistance of anodized binary Ti-Ta and Ti-Nb alloys were higher than those of the non-anodized Ti alloys in 0.9% NaCl solution. Surface morphology changes of anodized Ti-x(Nb,Ta) binary alloys depend on content of Nb and Ta.

HP-15 Nonvolatile Memory Effect of W Nanocrystals Thin Film Under Various Nitride-Base Plasma Treatments, ***S.-C. Chen*** *(scchen0213@gmail.com), T.-C. Chang,* National Sun Yat-Sen University, Taiwan, *W.-R. Chen,* National Chiao Tung University, Taiwan, *Y.-C. Lo, K.-T. Wu,* National Tsing Hua University, Taiwan, *S.M. Sze,* National Chiao Tung University, Taiwan, *J. Chen, I.H. Liao,* ProMOS Technologies, *F.-S. Huang*, National Chiao Tung University, Taiwan

In this study, a Tungsten nanocrystal thin film as charge storage center of nonvolatile memory was prepared and we focused on the electrical influence of nonvolatile memory effect for using the nitride-base various plasma treatments on our prepared sample. Transmission electron microscopy analyses revealed the microstructure in the thin film and X-ray photon-emission spectra indicated the variation of chemical composition after the nitride-base various plasma treatments. Electrical measurement analyses show the improvement of charge storage effect because the nitride-base various plasma treatments can enhance the surrounding silicon oxide quality of W nanocrystals. Moreover, the data retention and endurance characteristics of the W nanocrystals thin film nonvolatile memory were compared their performances under the different plasma treatment with passivated effect. In the previous research, this plasma treatment technique is first proposed to use on the metal nanocrysta ls nonvolatile memory application and to study the electrical characteristics. In addition, the process is compatible with the current flash memory fabrication technology.

HP-16 Influence of Annealing Temperature on Formation of Mo Nanocrystal Memory in Oxygen Incorporated Mo and Si Thin Film, ***C.-C. Lin***, National Chiao Tung University, Hsin-Chu, 300, Taiwan, *T.-C. Chang (tcchang@mail.phys.nsysu.edu.tw),* National Sun Yat-Sen University, Taiwan, *C.-H. Tu,* National Chiao Tung University, Taiwan, *S.-C. Chen, J.-Y. Lin,* National Yunlin University of Science and Technology, Taiwan, *S.M. Sze, C.-W. Hu, T.-Y. Tseng*, National Chiao Tung University, Taiwan

In this study, we investigated the influence of rapid thermal annealing temperature on formation of Mo nanocrystal memory in oxygen incorporated Mo and Si thin film. X-ray photon-emission spectra and transmission electron microscopy analyses reveal a change of chemical bonds and microstructure in the thin film at the various annealing temperatures. Electric measurement analyses indicate the charge storage effect of the thin film at the higher annealing temperatures due to formation of Mo nanocrystals embedded in nonstoichiometry silicon oxide. The difference in electric characteristics for the thin film at the various annealing temperatures was correlated to the change of the chemical bonds and the microstructure after the annealing.

HP-17 Charge Stored Effects of Ni-O-Si and Ni-Si-N Nanocrystals Thin Film Using a Low Temperature Fabrication, ***W.-R. Chen*** *(weiren93@gmail.com),* National Chiao Tung University, Taiwan, *T.-C. Chang,* National Sun Yat-Sen University, Taiwan, *C.-Y. Chang*, National Chiao Tung University, Taiwan

In recent years, most methods of nanocrystals thin film fabrication generally need the thermal treatment with high temperature and long duration. This procedure will influence thermal budget and throughput for the current manufacture technology of semiconductor industries. Hence, an ease and low temperature fabrication technique of Ni-O-Si and Ni-Si-N nanocrystals was demonstrated for nonvolatile memory application in this study. The memory structure of Ni-O-Si nanocrystals embedded in the SiO_x layer was fabricated by sputtering a commixed target ($Ni_{0.3}Si_{0.7}$) in an Ar/O_2 environment at room temperature. It can be considered that the oxygen plays a critical role during sputter process for the nanocrystal formation. In addition, a high density (~10^{12} cm^{-2}) nanocrystal also can be simple and uniform to be fabricated in our study. We also proposed a formation of Ni-Si-N nanocrystals by replacing O_2 by N_2 environment during the sputtering process. It was also found that a high density Ni-Si-N nanocrystal was embedded in the silicon nitride (SiN_x) which presented larger memory effect. Therefore, by using this internal competition mechanism of charge trapping layer for these elements (Ni, Si, and O/N), we can obtain a metallic nanocrystals nonvolatile memory with a low temperature process.

HP-18 Correlations Between Microstructure and Properties of High Purity Electrodeposited Nickel Coatings, *A. Godon,* ***J. Creus*** *(jcreus@univ-lr.fr), X. Feaugas, E. Conforto, P. Girault,* Université de la Rochelle, France, *L. Pichon,* Université de Poitiers, France, *C. Savall*, Université de la Rochelle, France

Protection of metallic surfaces against aqueous aggressive environments is a matter of great concern because of economical reasons and environmental issues. Protective coatings are commonly used to improve corrosion resistance. However, mechanical and/or tribological resistance may also be required to ensure the durability of structures, depending on the field of application. Recent studies have shown that polycrystalline metals with a grain size in the nanometer range seem to exhibit improved mechanical properties@super 1,2@. Moreover corrosion resistance is also strongly influenced by grain size@super 3,4@. The mechanisms by which these properties are linked to grain size have not been explained yet. In the case of nickel, some limitations of these studies arise from difficulties in controlling the microstructure and the purity of the coatings. The aim of this research is to acquire a better knowledge of the interplay between structure, composition, corrosion resistance and m echanical properties. In order to carry through these objectives, nanocrystalline nickel has been chosen as a model material. The coatings are obtained by electrodeposition in different baths (Sulfamate bath and Watts Bath) without additives, in order to avoid the incorporation of impurities@super 5@. The composition of the coatings is analyzed by Glow Discharge Optical Emission Spectrometry (GDOES). The microstructure is characterized using different techniques (XRD, SEM, TEM and AFM) in order to study the influence of deposition parameters on microstructure at different scales. Polarization curves (in H2SO4 1M) and micro-hardness measurements are used to characterize the properties of the coatings comparatively to several forms of nickel: microcrystalline bulk nickel with different grain sizes, single crystals of different crystallographic orientations and commercial nanocrystalline nickel. Our experimental approach includes a careful preparation of the surface in order to separ ate the effects due to several factors which can modify the surface reactivity: roughness, contamination, deformation, crystallographic orientation. (paragraph@@super 1@T.H. Yim, S.C. Yoon and H.S. Kim, Mater. Sci.Eng. A 449-451 (2007) 836-840. @paragraph2@super 2@Y.J. Li et al., Acta Mater. 55 (2007) 5708-5717. @paragraph2@super 3@R. Mishra, R. Balasubramaniam, Corros. Sci. 46 (2004) 3019-3029. @paragraph2@super 4@L.Wang et al., Scripta Mater. 55 (2006) 657-660.

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HP-19 Biocompatibility and Anti-Microbial Properties of TiO_x Thin Films, *S.E. Rodil (ser42@iim.unam.mx), H. Arzate,* Universidad Nacional Autónoma de México, ***A Almaguer-Flores***, Universidad Nacional Autónoma de México, Mexico

Titanium oxide thin films were deposited by rf-magnetron sputtering using a pure titanium target and reactive plasma conditions (oxygen + argon). Neither substrate bias nor temperature were used, therefore the films were amorphous and substoichiometric. Nevertheless, the biological response of the oxide films in comparison to medical grade stainless steel (SS) and to Ti6Al4V samples was particularly good. As biocompatibility tests, we

studied the attachment and proliferation/viability of human osteoblast cells on the TiO_x films. The number of attached cells on the TiO_x surfaces after 24 hours was twice the number on the SS substrate. While the proliferation after three days of incubation was one order of magnitude higher on TiO_x than on SS. Similarly results were obtained for the expression of Alkaline Phosphatase after 5 and 15 days of incubation, which is considered a marker of osteoblast differentation and new bone formation. These results suggest that these easily deposited TiO_x films could be used as a surface modification for orthopaedical implants and since microbial infection is still one of the main causes of implant failure, we decided to study the adhesion of bacteria in comparison to the standard Ti6Al4V material. The bacterial adhesion studies were done using nine different strains from the oral microbiota. The results showed that the number of colony forming units counted after 24 hours, 3 and 7 days was extremely low on the TiO_x films in comparison to SS and Ti6Al4V surfaces.

HP-20 Field Emission Properties of Zinc Oxide Nanorods Grown on ZnO/Si Substrates by Using Plasma Treatment, ***I.-C. Yao***, *T.-Y. Tseng (tseng@cc.nctu.edu.tw), P. Lin*, National Chiao Tung University, Taiwan

The ZnO nanorods on ZnO/Si substrates were synthesized by using the low temperature growth aqueous solution method. The different Argon/Oxygen ratio plasma treatments were carried on the as-grown ZnO nanorods to provide the nanorods with various tip angles. The morphology and crystal structure of the ZnO nanorods were examined by scanning electron microscopy, transmission electron microscopy and X-ray diffraction, respectively. The field emission properties of the ZnO nanorods with different tip angle ($95^o$and 110^o) are: the turn-on electric fields (at the current density of $10\mu Acm^{-2}$) are about 2.3 and 3.0 $V\mu m^{-1}$, respectively, while the threshold electric fields (at the current density of $1mAcm^{-2}$) are 6.0 and 6.6 $V\mu m^{-1}$, respectively. The improved field emission properties are believed to benefit from decreased emitter tip angle.

HP-21 Comparison of the Ti-Si-O Composites Nanocrystals Synthesized via Different Methods for Nonvolatile Memory Applications, ***L.-W. Feng*** *(b8823048@student.nsysu.edu.tw),* National Chiao Tung University, Taiwan*, T.-C. Chang,* National Sun Yat-Sen University, Taiwan*, P.-S. Wang,* National Chiao Tung University, Taiwan*, C.-F. Weng, M.-C. Chen, D.-S. Gan, N.-J. Ho, H.-J. Huang,* National Sun Yat-Sen University, Taiwan*, C.-Y. Chang*, National Chiao Tung University, Taiwan

The purpose of this study was to compare the Ti-Si-O nanocrystals properties and electrical behavior prepared from two methods between co-sputtering $TiSi_2$-Si with post-annealing in oxygen ambient and co-sputtering Ti-SiO_2. The characteristics of Ti-Si-O composition were analyzed by X-ray photon-emission spectra and the formations of nanocrystals were observed by high resolution transmission electron microscopy under the various annealing temperatures. The charge storage properties of Ti-Si-O nanocrystals fabricated by different methods on the metal oxide semiconductor (MOS) structure has been investigated. capacitance–voltage (C–V) measurements for memory window and the endurance and retention characteristics are exhibited for nonvolatile memory application.

HP-22 Synthesis and Electrical Characterizing of p-Type ZnO:Sb Nanostructures, ***J.M. Wu*** *(jmwu@fcu.edu.tw),* Feng Chia University, Taiwan*, C.-W. Fang, L.-T. Lee,* National Chung-Hsing University, Taiwan*, Y.-H. Lin*, National Tsing Hua University, Taiwan

P-type zinc oxide (ZnO) doped antinomy (Sb) nanostructures have been synthesized. The different weight percentage of Sb powder (for example: 1/150, 1/70, 1/30, and 1/10 wt.%) was mixed in Zn powder to act as source materials. The Sb doped ZnO nanostructures were therefore synthesized. These nanostructures were grown on alumina substrate at 600°C by vapor transport method that employed the vapor-solid (VS) growth mechanism. Upon illumination by UV light (365 nm), the photoelectric current of the ZnO:Sb nanowires exhibited a rapid photo-response as a UV lamp was switched on and off. The photocurrent is strongly depending on the dopant concentrations, which leads a different electron-hole pair separation rate. Field emission from as-synthesized ZnO:Sb nanostructures shows large current densities (1 mA/cm^2) and low turn-on field (2-3 V/μm), which are comparable to those of undoped ZnO nanostructures. Ethanol gas sensors were also fabricated on alumina substrate us ing these ZnO:Sb nanostructures.

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HP-23 Effect of the Third Elements Implantation in Titanium Dioxide Thin Films to Improve the Photocatalytic Properties, ***K.-W. Weng*** *(wl2426@ms26.hinet.net),* Mingdao University, Taiwan*, T.-N. Lin,* Institute of Nuclear Energy Research, Taiwan*, C.-H. Chao, S.-Y. Lien,* Mingdao University, Taiwan*, Y.-C. Chen*, National Chung Hsing University, Taiwan

Titanium dioxide (TiO_2) films are stable and inexpensive materials which have the photocatalytic characteristics and have been extensively studied. The band gap of TiO_2 is 3.2eV with the absorption wavelength locating at UV region. Therefore, the sunlight absorption rate of TiO_2 films is relatively low. Recently, many researchers pay more attention to improve the activation and visible light absorption rate of TiO_2 films. In this study, the effect of adding third elements in TiO_2 films to improve the photocatalytic properties is investigated. The samples were prepared by a hybrid system containing physical vapor deposition (PVD) and metal plasma ion implantation (MPII). High quality TiO_2 films were first fabricated by r.f. magnetron sputtering system, followed by metal plasma ion implantation of third elements, such as Cr, V and Fe. The results show that the band gap of TiO_2 with third elements implant is less than 3eV, resulting in the absorption wavelength range shift ing to 400-550nm. As a result, the activation and sunlight absorption rate of TiO_2 thin films with the addition of third elements is greatly increased by the hybrid processing method.

HP-24 Fabrication of Dye-Sensitized Solar Cells Based on ZnO Nanowires, *M.F. Hossain, S. Biswas, M. Shahjahan,* ***T. Arakawa****, T. Takahashi (takahash@eng.u-toyama.ac.jp)*, University of Toyama, Japan

Recently, great attention has been paid to dye-sensitized solar cells (DSCs) due to their low fabrication cost. The high light-to-energy conversion efficiencies achieved with DSCs may be attributed to the nanoporous TiO_2 electrode. Zinc oxide (ZnO) is a wide band gap semiconducting material with a similar band gap and electron affinity to those of TiO_2 and has been considered as an alternative material in DSCs applications. One-dimensional (1D) nanowires have been extensively studied in recent years. ZnO nanowires have attracted great interest for promising applications in optoelectronics devices. Several methods have been demonstrated to fabricate 1D nanostructures, such as vapor liquid-solid epitaxy, chemical vapor deposition, and pulse laser deposition, but these gas phase techniques still have some limitations for substrate size and the need for high temperature operation (above 800°C). Among the various techniques for the preparation of ZnO nanowires, the relatively simple sol-gel method is the most widely used because of its ability to obtain films with tailored properties on large, curved substrates. In our present study, we systematically study the feature-controlled ZnO nanowires arrays via the hydrothermal method and ZnO sol-gel thin films were used as the seed layers on SnO_2 :F coated glass with different pretreatment conditions. The ZnO nanowires have been characterized by the TG-DTA, XRD, SEM AFM, FTIR and UV-VIS system. The surface morphology of the nanoporous ZnO nanowires strongly depends on the annealing temperature of the seeding ZnO layers. It was revealed from optical study that the dye absorption increases with the increase of annealing temperature. Incident photon-to-current efficiency is calculated for all the solar cells with different ZnO thin films. The variation of photoelectric conversion efficiency of the DSCs, deposited with various annealing temperature is discussed with the analysis of diffe rent structure of ZnO nanowires and the corresponding dye-incorporations.

HP-25 Comparative Study of Dye and CdS Sensitized Grätzel Solar Cells, ***M.F. Hossain****, S. Biswas, M. Shahjahan, T. Takahashi (takahash@eng.u-toyama.ac.jp)*, University of Toyama, Japan

Dye-sensitized solar cells (DSCs) have been widely investigated as a next generation solar cells because of their simple structure and low manufacturing cost. One of the key factors for enhancing the efficiency of DSCs is the light harvesting properties of dye, attached on the surface of titanium oxide (TiO_2). Ruthenium has been mostly used as sensitizer in DSCs, which is very expensive. An alternative method is to couple the TiO_2 electrode with narrow band gap semiconductor which enhances the light absorbing property as well as the overall efficiency of DSCs. In this study, cadmium sulfide (CdS) and ruthenium based dye were used as sensitizers. The photovoltaic performances of the solar cells, consisting of these two sensitizers, were compared. TiO_2 photoelectrodes were prepared on SnO_2:F coated glass by facing target reactive sputtering technique. CdS was deposited on TiO_2 by inexpensive chemical bath deposition technique and was annealed at 400^oC in high vacuum of $5x10^{-5}$ Pa for improving crystallinity. The dye and CdS sensitized Grätzel solar cells consist of iodide and polysulfide-based electrolytes respectively. Mildly coated platinum counter electrode was used in both solar cells. The structural, optical and surface morphological properties of TiO_2 photoelectrodes were characterized by x-ray diffraction analysis, energy dispersive x-ray analysis, ultraviolet-visible spectroscopy, atomic force microscopy and field emission scanning electron microscopy. Interestingly, all the photovoltaics parameters of CdS sensitized Grätzel solar cells are higher for back side

illumination than the front side illumination. The CdS sensitized solar cells shows higher photoelectric-conversion efficiency with back side illumination than the ruthenium based DSCs in both side illuminations. It is may be due to the fact that nanoporous TiO_2 electrode incorporates both the dye and CdS in a same manner, how ever thin polycrystalline layer of CdS remains as a separate layer on the TiO_2-surface, which enhances the photoelectric-conversion efficiency.

HP-26 Electron Field Emission of Single Wall Carbon Nanotube by Catalytic CVD, ***T.-Y. Lu***, *Y.-H. Yang, C.-K. Liu, H.-C. Shih (hcshih@mx.nthu.edu.tw)*, National Tsing Hua University, Taiwan

Since their discovery in 1993, single wall carbon nanotubes (SWCNTs) have been studied because of their unique chemical and physical properties. Many important applications of SWCNTs such as sensors, field emitters, transistors, and supercapacitors have been demonstrated.

In this work, high quality and large quantity single wall carbon SWCNTs were fabricated by ethanol at 900°C in a tubular furnace. The Fe and Mo metals were used as catalysts to deposit SWCNTs on the surface of the MgO sol-gel nanopowders[1]. SWCNTs have diameters in the range of 0.6-1.6 for SWCNTs in bundles, and diameters in the range of 2-6 nm for isolated SWCNTs were identified by the high-resolution transmission electron microscope and the radial breathing mode (RBM) of the Raman spectrum. A high yield of the as-synthesized SWCNTs is over 500 wt % relative to the weight of Fe and Mo metal in the catalyst[2]. The extremely low turn-on field Eto = 0.008 V/@,icron@ and threshold field Eth = 0.07 V/μm show that the SWCNTs have much better performance than the multi wall carbon nanotubes[3].

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HP-27 The Role of Metal Plasma Ion Implantation in Anatase-Titanium Dioxide: Correlation Between Photoreactivity and Implantation Mechanism, ***M.-H. Shih***, Mingdao University, Taiwan, *C.-C. Yen,* National Chung Hsing University, Taiwan, *D.-Y. Wang,* Mingdao University, Taiwan, *L.S. Chang, H.-C. Shih (jackal8914@yahoo.com.tw)*, National Chung Hsing University, Taiwan

The pure titanium dioxide thin films were prepared on glass substrates by the sol-gel spin coating method. The as-deposited TiO_2 thin films were subjected to metal plasma ion implantation at 30 KeV to incorporate transition metals to investigate the effect of impurity species and ion dosage. We theoretically investigated the photoreactivity of the metal ions as implants modify the band gap properties of anatase TiO_2. According to the results of X-ray photoelectron spectroscopy (XPS) analysis, the Me-implanted (Me= Cu, Fe, Ni, ...) TiO_2 leads to the formation of implanted metal oxide. The implanted metal oxides extended the photosensitivity range of the anatase TiO_2 to cover the visible light spectra. Compared to the pure TiO_2, a photo-catalysis was observed in the visible light regime. The implantation mechanism will be conducted by using UV-Vis spectrophotometer, photo-luminance spectroscopy and cathode-luminescence.

HP-29 The Properties of Transparent Semiconductor $Zn_{1-x}Ti_xO$ Thin Films Prepared by Sol-Gel Process, ***C.-Y. Tsay*** *(cytsay@fcu.edu.tw),* Feng Chia University, Taiwan, *H.-C. Cheng,* Industrial Technology Research Institute, Taiwan, *C.-Y. Chen, K.-J. Yang, C.-K. Lin*, Feng Chia University, Taiwan

Zinc oxide based transparent semiconductor films have attracted considerable interest for AMLCD, AMOLED display and E-paper applications. This work investigated the effect of various Ti addition (0, 1, 3, 5, and 10 at.%) on the microstructure, surface morphology, transparency, and resistivity of ZnO thin films prepared by sol-gel process. The as-prepared films were annealed in air at 500 °C for 1 hr. Experimental results showed that additions of Ti into ZnO thin films ($Zn_{1-x}Ti_xO$) not only refined the grain size but also increased the transmittance and resistivity of thin films. In the present study, the $Zn_{0.9}Ti_{0.1}O$ thin films exhibited the best performance with an average transmittance of 91.0% (an increase of ~12% over a pure ZnO thin film), a resistivity of 1.1×10^5 ω-cm, and a RMS roughness value of 3.0 nm.

HP-30 Silver Phthalocyanine Films for Photovoltaic Applications, ***A. Mahajan*** *(dramanmahajan@yahoo.co.in),* DAV College, India, *H. Gupta, R.K. Bedi*, Guru Nanak Dev University, India

Silver phthalocyanine (AgPc) has attracted considerable interest in organic photovoltaic devices because of its outstanding optical and electrical properties. To improve performance of devices based on AgPc, a series of silver phthalocyanine (AgPc) films has been prepared under different experimental conditions. These samples have been studied for their structural, optical and electrical properties. The X-ray diffraction and SEM pattern of these films show crystalline behaviour of films. The electrical conductivity and optical band gap of the films increases with increase in substrate temperature, whereas activation energy decreases. The activation energy of the films found to lie in 0.41-0.83 eV. Analysis of optical absorption measurements on the films indicates that the interband transitions energies lie in 3.9-4.1 eV. Keeping in view, the electrical and optical properties of AgPc films single layer (Fluorine doped tin oxide / AgPc /Aluminium) and double layer (Fluori ne doped tin oxide / Crystal violet / AgPc /Aluminium) junctions have been fabricated under different experimental conditions. The J-V relationship for the single and double layer devices are found to be in good agreement with standard diode equation.

HP-31 Synthesis and Magnetic Properties of Self-Assembled Manganese-Iron Spinel Nanocrystal Films, ***C.-R. Lin*** *(crlin@mail.stut.edu.tw), S.-Z. Lu*, Southern Taiwan University, Taiwan

Manganese-iron oxide Mn@sub3-x@Fe@subx@O@sub4@ (x=1.25, 1.50, 1.75) nanocrystals were prepared through polyol reduction of manganese chloride tetrahydrate (MnCl@sub2@-4H@sub2@O) and ferric chloride hexahydrate (FeCl@sub3@-6H@sub2@O) in the presence of oleic acid, oleylamine, and sodium hydroxide (NaOH). The as-synthesized nanocrystals have the cubic spinel structure and mean crystallite size of 4.8-5.3 nm. Their monodispersities were characterized by dynamic light scattering (DLS) and transmission electron microscopy (TEM). Magnetic measurements show that the as-synthesized magnetic nanocrystals display a superparamagnetic behavior with zero coercivity and remanence. The saturation magnetization of Mn@sub3-x@Fe@subx@O@sub4@ obtained by the plots of M against 1/H curve were 1.8, 5.1, and 6.8 emu/g for x = 1.25, 1.50, and 1.75, respectively. Thermal annealing induces the change of crystallite size and thus the magnetic properties of the nanocrystal assembled films. Both m icrostructure and magnetic behavior of the nanocrystal assembled films are very sensitive to the film composition. In general, the coercivity of film first increases as the crystallite size increases reaches a maximum and then decreases for any further increase in crystallite size. Very high coercivity (3200 Oe) was observed for composition with Mn@sub1.75@Fe@sub1.25@O@sub4@. Magnetic interaction between nanocrystals of these self-assembled nanocrystal films was analyzed by examining the remanent magnetization curves via the Henkel-plots and delta M-plots@super 1,2@. This controlled synthesis and assembly can be used to fabricate Mn@sub3-x@Fe@subx@O@sub4@ nanocrystal films for future nanomagnetic applications in various technological fields, such as ferrofluids, sensing elements, and recording media.

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HP-32 Effect of Nitridation on Silicon Nanocrystals for Nonvolatile Memory Application, ***F.-Y. Jian***, *T.-C. Chang (tcchang@mail.phys.nsysu.edu.tw), A.-K. Chu,* National Sun Yat-Sen University, Taiwan, *R.-Y. Wang,* UMC (United Microelectronic Corporation), Taiwan, *S.-C. Chen,* National Sun Yat-Sen University, Taiwan, *C.-K. Kao*, ProMOS Technologies, Taiwan

Nonvolatile silicon nanocrystals memory using an in-suite nitride passivation has been studied in this work. Program properties are found superior to that without nitridation, while erase properties are almost the same for both samples. The program characteristic could be improved due to the lower the electrical field across the nitride layer and reducing leakage current through the blocking layer. On the other hand, the erase characteristic is mainly dominated by the electrical field in tunnel oxide under smaller gate voltage. As the erasing bias increasing, the silicon-nitride layer can not suppress the gate injection because the conduction band of silicon-nitride layer is lower than that of oxide layer under the Fowler-Nordheim tunneling. In addition, the retention characteristics have been found almost the same with and without nitridation in this experiment. The trap level in silicon-nitride layer (about 1.3eV below conduction band) is higher the conduction band in silicon, therefore, electron can not be stored in those traps in the silicon-nitride layer effectively. The result could be used to clear the electric properties for silicon nanocrystals based nonvolatile memory.

HP-33 Growth of ZnO Nanorods and Nanowires by Supercritical CO2 Fluid Treatment, ***K.-C. Chang*** *(doubleccc@yahoo.com.tw), T.-F. Young, T.-C. Chang,* National Sun Yat-Sen University, Taiwan*, C.-T. Tsai,* National Tsing Hua University, Taiwan*, S.-C. Chen, H.-C. Huang, D.-S. Gan, N.-J. Ho, Yong-En Syu*, National Sun Yat-Sen University, Taiwan

A low-temperature method, supercritical CO2 fluid (SCCF) technology, was applied for oxidation of metal Zn film on glass substrate at 150 XC. In this study, Zn film was deposited by DC sputtering at room temperature and post-treated by SCCF, which is mixed with 0.15 vol % H2O and 0.15 vol % ethanol (sample A), and with only 0.15 vol % H2O (sample B). The scanning electron microscopy (SEM) images indicate that high density ZnO nanorods for sample A and nanowires for sample B were formed on the glass substrate. SCCF technology has shown successful oxidation the Zinc at low temperature for the first time. The mechanism of SCCF oxidation has also been discussed in this paper.

HP-34 Effects of the Anodizing Processon the Corrosion Behaviors of Ti-xHf Alloys, ***Y.-H. Jeong****, H.-C. Choe (hcchoe@chosun.ac.kr), Y.-M. Ko, H. Ahn*, Chosun University, Korea

Titanium and its alloys, such as Cp-Ti and Ti-6Al-4V alloy have been extensively studied for the applications of orthopedic and dental implant materials because of their excellent mechanical properties, corrosion resistance, and outstanding biocompatibility. However, a native TiO2 has not enough bioactivity to osseointegrate with bone and the Ti-6Al-4V alloy in an acceptable prosthetic biomaterial, recent studies indicated that the release and accumulation of Al and V ions could have harmful effects on the human body. Hafnium belongs to the value metal group and hafnium oxide has numerous properties. These properties make hafnium oxide a valuable material to be used for various applications such as optical coatings, gas sensors of capacitor and as protective coatings for biomedical applications. Anodizing method of Ti-based alloys has attracted great attentions due to interfacial properties. In this study, Ti-xHf binary alloys containing 10, 20, 30 and 40 wt% Hf contents were manufactured by the vacuum furnace system. These Ti-xHf alloys were anodized in solution containing typically 1M H3PO4. A direct current power source was used for the process of anodization. Firstly, anodization power was controlled with 120, 170, and 220V, respectively, at room temperature. Secondly, the anodized surfaces of Ti-xHf alloys were crystallized by heat treatment at 300℃, 500℃, and 600℃ for 6 hours in Ar atmosphere. The microstructure was characterized by XRD and SEM. The corrosion behaviors of the anodized samples at different experimental conditions were studied using potentiodynamic test and A. C. impedance test in 0.9% NaCl solution at 36±1℃. The result shows that anodized Ti-xHf alloys showed that pore size increased as applied voltage increased, whereas, pore size decreased as Hf content increased. The anodized Ti-xHf alloys were exhibited more good corrosion resistance than non treated Ti-xHf alloys.

HP-35 The Studies of Flexible a-Si:H, *Y.-T. Chou,* ***C.-Y. Su****, P.-T. Liu (ptliu@mail.nctu.edu.tw), S.-Y. Tsai,* National Chiao Tung University, Taiwan*, I.-H. Peng*, National Tsing-Hua University, Taiwan

We investigated the effects of the uneasily mechanical strain stress on flexible hydrogenated amorphous silicon (a-Si:H) thin film transistors (TFTs) in this work. The proposed a-Si TFTs were fabricated on thin steel foil and all process temperature was well-controlled below 200℃. The reliability of TFTs was discussed by applying DC bias stress on gate electrode up to 104 seconds and the strain was imposed on the device parallel to the source-drain current path. Our results indicated both outward and inward strain stress can lead to an un-recoverable destruction on a-Si:H TFTs at the first time bending behavior and derived the device in different performance from initial case permanently. We provided a model and used activation energy to explain the bending behavior. The passivation effect was also discussed in these studies. The device with passivation layer not only increased the performance, but also enhanced the reliability of devices. By adding one more thermal t reatment on the device with passivation layer, the threshold voltage was 1.3V closed to 0V, and electrical performance was obvious enhanced.

HP-36 Characterization of Electrically Conducting Polyaniline Nanofiber/polyimide Nanocomposites, ***A. Hopkins*** *(alan.r.hopkins@aero.org)*, The Aerospace Corporation

Nanocomposites of polyaniline nanofibers and polyimide were fabricated and studied using small angle neutron scattering (SANS). The immiscible nature of the conformationally dissimilar polyaniline nanofiber and polyimide host is established by a series of experiments involving neutron scattering. Based on these techniques, we conclude that the crystal structure of the polyimides is not disrupted, and that there is no mixing between the two components on a molecular level. The morphology of the conducting salt component was analyzed by SANS data and was treated by two common models: Debye-Bueche (D-B) and inverse power law (IPL). Due to deviations in the linear curve fitting over a large scattering range, neither the D-B nor the IPL model could be used to characterize the size and shape of all PANI-0.5-CSA/polyimide blend systems. At 1 and 2% concentration, the D-B model suggested salt domains between 20 and 70 Å with fractal geometries implied by the IPL model. As salt concentrations are increased to 5%, the structures are observed to change, but there is no simple structural model that provides a suitable basis for comparison.

Surface Engineering for Thermal Management
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TSP Poster Session

TSP-1 Thermal Conductivity of Polymer-Ceramic-Metallic Nanolaminates, ***A.R. Waite*** *(adam.waite@wpafb.af.mil),* Air Force Research Lab/UTC, Inc./University of Dayton*, J.G. Jones,* Air Force Research Lab*, A.A. Voevodin,* Air Force Research Lab/University of Dayton*, C. Muratore,* Air Force Research Lab/UTC, Inc.*, A.M. Urbas,* Air Force Research Lab*, J.O. Enlow,* Air Force Research Lab/UES*, T.J. Bunning*, Air Force Research Lab

A novel multilayer polymer-metal-ceramic nanolaminate coating was synthesized by room temperature Plasma Enhanced Chemical Vapor Deposition (PECVD) and Magnetron Sputtering (MS) processes. The nanolaminate was deposited in a custom built, two-chamber CVD-PVD system. The polymer deposited was a low refractive index, highly cross-linked fluoropolymer deposited by PECVD from an octafluorocyclobutane gas precursor. The high refractive index ceramic layers were deposited by MS of a TiO_2 target. High and low thermal conductivity metal layers were also deposited by MS. This nanolaminate structure realizes the idea of creating an optical coating from a stack of high and low refractive index dielectrics with the additional functionality of an enhanced thermal conductivity in the coating plane. Thermal conductivity of the optical coating was tuned by inserting very thin metallic layers of varying composition (Ti and Cu) and thicknesses into the nanolaminate structure. The fluoropolymer and TiO_2 layers were approximately 100 nm and 60 nm, while the metal layers varied from 1 to 10 nm. The refractive indices of the fluoropolymer and TiO_2 were 1.38 and 2.31, respectively, at a wavelength of 532 nm. The nanolaminate thermal conductivities were measured by Time Domain Thermoreflectance (TDTR) using a laser pump probe technique.

TSP-2 Surfaces Incorporating Phase Change Materials for Controlled Storage and Release of Thermal Energy, ***C. Muratore*** *(chris.muratore@wpafb.af.mil),* Air Force Research Laboratory*, S. Aouadi,* Southern Illinois University Carbondale*, A.A. Voevodin*, Air Force Research Lab/University of Dayton

Applications on multiple size scales require maintenance of a constant temperature for optimal performance. The utility of phase change materials (PCMs) where thermal energy can be stored and released without changes in temperature through the PCM latent heat of fusion and/or vaporization, has been demonstrated in macroscale applications such as plasterboard for building interiors or textiles for winter clothing which both employ embedded volumes of encapsulated wax tuned to melt at a temperature slightly above the desired operating temperature. Energy is stored through the melting process. If the temperature drops below the melting point, energy is released as the wax solidifies. Phase change materials would also be useful for temperature maintenance at the micro- and nanoscale. For example, optical links between microprocessors and other chips where changes in temperature-dependent characteristics of optical sources and receivers (i.e., wavelength and current) can ren der the device inoperable unless the intended temperature of operation is maintained. One means of passive temperature maintenance on this size scale is through composite surfaces incorporating phase change materials. Laser patterning and reactive ion etching of oxidized silicon substrates with 20 micron wide by 10 micron deep holes were investigated initially. The holes were filled with materials known to undergo phase changes at target operating temperatures. The initial reservoir geometry was 20 microns wide by 10 microns deep. Surfaces with 40 percent coverage of reservoirs with these dimensions were expected to store up to 1 joule of thermal energy when filled with bismuth. This energy could be stored and released as the ambient temperature deviated from the PCM melting point to modulate the surface temperature as the ambient temperature fluctuated. Metallized samples of silicon containing PCM-filled reservoirs were studied with an infrared microscope to identify the te m perature profile on the surface as the material was heated and cooled from behind to observe temperature discontinuities induced by phase changes. Equivalent total reservoir volumes with different dimensions and fractional surface coverage were produced using both techniques to investigate the dependence surface temperature and phase change temperature on reservoir geometry.

TSP-3 Anti-Bacterial TiN_x-Ag Coatings on Titanium Dental Implants, *Y.-Y. Chang (yinyu@mail2000.com.tw),* ***C.-C. Hsuer****, P.-J. Lin, H.-T. Hsu, D.-Y. Wang*, Mingdao University, Taiwan

Titanium-based materials have been used for dental implants due to their excellent biological compatibility, superior mechanical strength and high corrosion resistance. The osseointegration rate of titanium dental implants is related to their composition and surface treatment. A better anti-bacterial performance of the abutment seated in the prosthetic crown is beneficial for the osseointegration rate. In this study, Ti_2N-Ag coatings with different titanium and Ag content were deposited on bio-grade pure Titanium dental implant materials. A twin-gun magnetron sputtering system was used for the deposition of the Ti_2N-Ag coating. The Ag content in the deposited coatings was controlled by the magnetron power ratio of Ag/Ti targets. WDS was used to characterize the composition of the deposited Ti_2N-Ag coatings. The crystalline structure and bonding states of the coatings were analyzed by XRD and XPS. The bacterial adhesion and bactericidal effects of the Ti_2N-Ag coated Titanium was assessed using E-coli cell tests. Growth and killing kinetics on Ti_2N-Ag coatings were determined by an optical density (OD) analysis at 600 nm. It is proposed that the nanostructure and Ag content of the Ti_2N-Ag coatings were correlated with the biocidal property.

TSP-4 A Study of the Anti-Microbial Properties of TiN/Ag Nanocomposite Coatings, ***P.J. Kelly*** *(peter.kelly@mmu.ac.uk), H. Li, K.A. Whitehead, J. Verran,* Manchester Metropolitan University, United Kingdom*, R.D. Arnell*, University of Central Lancashire, United Kingdom

Titanium nitride (TiN) is a hard, wear resistant coating material, which is widely applied to components operating in an abrasive wear environment. When co-deposited with silver, the coatings form a nanocomposite structure consisting of nanoparticles of silver embedded in a TiN matrix. Silver is lubricious, and the 'self-lubricating' nature of these coatings, combined with their high hardness and scratch resistance makes them attractive for tribological applications. However, combining these properties with the inherent anti-microbial nature of silver also opens up novel applications for TiN/Ag nanocomposite films in, for example, the bio-medical or food processing industries, where surfaces that are durable, safe, readily cleanable and resistant to microbial contamination are required. In this study, TiN/Ag coatings have been deposited by co-sputtering onto tool steel and stainless steel substrates. By control of the target powers, the silver content of the films w as varied in the range 0-25 atomic percent and the films have been characterised using SEM, XRD, EDX and AFM to determine the size, shape and distribution of the silver nanoparticles. The initial interaction, i.e., attachment and retention, of specific microorganisms on the coated substrates has also been assessed. The microorganisms tested included Escherichia coli and Staphylococcus aureus. The antimicrobial activity of diffusible components of the surfaces was tested using zones of inhibition assays, whilst antimicrobial activity by contact was assessed using live/dead staining (and microscopic analysis) and agar overlay methods. The strength of attachment of retained cells was assessed using AFM.

TSP-5 Carbon Nanotube - MoS2 Nanocomposites as Self-Lubricating Coatings, ***B. Sirota****, X. Zhang, B. Luster, A. Church,* Southern Illinois University Carbondale*, C. Muratore,* Air Force Research Lab/UTC, Inc.*, A.A. Voevodin,* Air Force Research Lab/University of Dayton*, P. Kohli, S. Talapatra, S. Aouadi (saouadi@physics.siu.edu)*, Southern Illinois University Carbondale

Solid Lubricants (SLs) characterized by low friction coefficient and wear rates drastically improve the life span of instruments which undergo extreme frictional wear. However, the performance of SLs such as sputtered or nanoparticulate molybdenum disulfide (MoS2), tungsten disulphide (WS2), or graphite deteriorates heavily under extreme operational conditions (for example elevated temperatures3). Here, we show that composites of carbon nanotubes (CNT)-MoS2, produced by electrodeposition of MoS2 on vertically aligned CNT films have low coefficients of friction (~0.03) and wear rates (~ 10-13 mm3/N.mm) even at 300°C (two orders of magnitude better than nanoparticulate MoS2). The high load bearing capacity of CNTs provides a strong enduring support to MoS2 nanoclusters and is responsible for their ultra low wear rates. The technique described here to produce SLs with extremely appealing frictional properties will provide valuable solutions for a variety of tribological ap plications.

TSP-6 Visible Light Activated Bactericidal Effect of V-TiO_2 Thin Film on Fish Pathogens, ***T.C. Cheng****, C.Y. Chang,* Mingdao University, Taiwan*, C.I. Chang,* Fisheries Research Institute, Taiwan*, H.C. Hsu, C.J. Hwang, D.Y. Wang, K.S. Yao (alesksyao@gmail.com)*, Mingdao University, Taiwan

Bacterial infection is always a problem for fish farming. To maintain hygienic aquatic environments become an important strategy to prevent bacterial infection in hatchery and pond using re-circulated water systems which will accumulate high concentration pathogen easily. Using environmental friendly strategy such as TiO_2 to inhibit the growth of fish pathogens become an alternative. However, requirement of using ultraviolet (UV) light to activated TiO_2 is a major limitation for bactericidal application on fish farming because fishes are vulnerable to intensive UV light. Therefore, vanadium (V) ion was added to TiO_2 thin film to increase photosensitivity under visible light irradiation and its bactericidal effect activated by visible light on fish pathogens is evaluated. V- TiO_2 are synthesized using sol-gel method and spin-coated on glass slide followed by 400 °C calcinations. Particles size are characterized using scanning electron micr oscope (SEM) and crystal structures are examined using X-ray diffractometry (XRD). The fish bacteria pathogens of Aeromonas hydrophila, Edwardsiella tarda and Streptococcus iniae causing great fish farming loss are deposited on the glass and radiated with visible light for various time intervals. The bacteria mortality is estimated using 2, 3, 5-triphenyl tetrazolium chloride (TTC). The results demonstrate that mortalities of fish bacteria pathogens are significantly higher in visible light radiated V-TiO_2 thin film groups than those of visible light radiated TiO_2 only thin film groups. Regardless of fish pathogen species, more than 50% mortalities are found in groups of 90 minutes visible light radiated V-TiO_2 thin film. In conclusion, visible light, instead of UV light, can be used to activate V-TiO_2 thin film to disinfect fish pathogens.

TSP-7 Molecular Dynamics Atom-by-Atom Simulations of Chemical Vapor Deposition of SiNH, *J. Houska (jiri.houska@polymtl.ca),* ***J.E. Klemberg-Sapieha****, L. Martinu*, Ecole Polytechnique de Montreal, Canada

In this contribution, we report molecular dynamics (MD) simulations of the atom-by-atom chemical vapor deposition of SiNH materials from N_2-SiH_4 plasma. The interatomic interactions are described by empirical potentials of Tersoff type. Each step of the deposition process consists of impacts of SiH_x and N radicals (both energetic ions and slow neutrals) onto material surface, a constant-energy MD run, a thermalization run, and removal of desorbed (resputtered) particles.

We calculate sticking coefficient of individual elements at various ion energies (20-300 eV) and SiH_x radicals compositions (x=1-3), and show formation of (a) interface (mixing) layers due to damaging of the Si substrate and (b) hydrogenated SiN networks. We investigate how the particle flux composition, ions-to-neutral ratio, energy of ions and deposition temperature affect the material's characteristics such as density or hydrogen content. We find that a higher SiH_x-to-N ratio leads to low-density, voids-containing networks, and calculate size of the voids. Using both classical MD simulations and ab-initio liquid-quench simulations, we investigate bonding preferences in SiNH networks, including formation of unbonded H_2 molecules.

The calculated results, compared with an experiment, allow one to predict relationships between particle fluxes, compositions and structures of SiNH materials for optical or electronic devices.

TSP-8 Corrosion Resistance of Stainless Steel Bipolar Plates for Fuel Cell by High Temperature Nitrogen Implantation, ***D.H. Kwon****, K.K. Kim, J.S. Kim,* Pusan National University, Korea*, S.M. Moon,* Korea Institute of Machinery & Materials, Korea*, J.S. Lee,* Korea Atomic Energy Research Institute, Korea*, M.C. Kang (kangmc@pusan.ac.kr)*, Pusan National University

Ion implantation is a novel surface modification to enhance the mechanical, chemical and electrical properties of substrate surface using high energy ions. Metallic bipolar plates for the proton exchange membrane fuel cell offer many advantages over conventional graphitic materials. These include relative low cost, high strength, and ease of manufacture. As a metallic bipolar plate can be easily shaped from thin sheet with complex shape, significant improvement in the fuel cell power/volume ratio can be achieved.

However, corrosion of the metallic bipolar plates is a severe problem. Corrosion affects the performance and lifetime of a fuel cell. The research on surface modification has been advanced to improve the properties of engineering materials. This experiment was performed in the high temperature in order to increase the implanted depth. The samples are implanted with 50～120 keV N-ion at substrate temperature ranging from 50～400℃. Nano-hardness and AES(Auger electrons spectroscopy) were measured from nitrogen ion implanted layer. The sliding wear and impact wear properties of ion implanted samples depend strongly on the ion doses and implantation temperature.

TSP-9 Antibacterial Properties and Tribology of a-C:Ag Coatings Deposited by Pulsed Cathodic Filter Arc, ***J. Endrino*** *(jlendrino@icmm.csic.es),* Instituto de Ciencia de Materiales de Madrid, Spain, *M. Allen,* Ohio State University, *J.C. Sanchez-Lopez,* Instituto de Ciencia de Materiales de Sevilla, Spain, *R. Escobar Galindo,* Instituto de Ciencia de Materiales de Madrid, Spain, *A. Anders,* Lawrence Berkeley National Laboratory, *J.H. Horton, T.M. Horton,* SUNY Upstate Medical University, *J.M. Albella*, Instituto de Ciencia de Materiales de Madrid, Spain

Amorphous carbon (a-C) is known to be a biocompatible material with good chemical inertness, this makes it a strong candidate to be used as a matrix that embeds metallic elements with an antimicrobial effect. We have deposited a set of a-C:Ag films using a dual-cathode pulsed filter cathodic arc source, the arc pulse frequency of the silver and graphite cathodes was controlled in order to obtain samples with various silver contents. In this study, we analyze the advantages of incorporating silver into a-C by studying the antimicrobial properties and tribology of the deposited films. The silver atomic content of the deposited samples was analyzed using glow discharge optical spectroscopy (GDOES). The deposited films were characterized by X-ray diffraction (XRD) and Raman spectroscopy. The bactericidal efficacy against staphylococcus of samples deposited on 24-well tissue culture plates was evaluated.

TSP-10 Enhancement of Electrochemical Activity of Olivine-Type Cathode Materials by Carbon Nano Tube, ***Y.-C. Chen*** *(d9531903@oz.nthu.edu.tw), J.-M. Chen, C.-H. Hsu, J.-W. Yeh, H.C. Shih*, National Tsing Hua University, Taiwan

$LiFePO_4$ is an attractive cathode material due to its low cost, good cyclability and safety. However, the Fe^{2+}/Fe^{3+} redox potential in $LiFePO_4$ is about 3.4V, setting a lower working voltage compared with other cathode materials. The natural drawback limits its development and application. Recently, other olivine-type cathode materials have been reported, such as $LiMnPO_4$, $LiCoPO_4$ and $LiNiPO_4$ with the potential energy of Mn^{2+}/Mn^{3+}, Co^{2+}/Co^{3+} and Ni^{2+}/Ni^{3+} redox systems about 4.1V, 4.9V and 5.1V, respectively. Because of the lower conductivity of the olivine-type materials, they are unable to be applied in the commercial products. In order to enhance the conductivity, some carbon materials are used as such as carbon black and KS4. In this work, carbon nano tube was used to substitute the traditional carbon materials due to its unique chemical and physical properties. The superior c haracter of carbon nano tube is not only applied as the anode materials but also as the conductive materials in the cathode materials. The surface of the olivine-type cathode we prepared was modified by the carbon nano tube and the electrical performance thus was enhanced.

TSP-11 Bactericidal Effect of TiO_2 Particle with Magnetic Core on Fish Pathogens, *T.C. Cheng, K.S. Yao,* Mingdao University, Taiwan, *C.I. Chang,* Fisheries Research Institute, Taiwan, ***H.C. Hsu***, *C.J. Hwang, D.Y. Wang, C.Y. Chang (cychang@mdu.edu.tw)*, Mingdao University, Taiwan

The UV irradiated TiO_2 has been used to disinfect bacteria in water. It is usually used either in powder form or coating on supporting materials such as glass or active carbon. Although the efficiency of bactericidal effect of TiO_2 powder is higher than those of coated TiO_2 due to large reaction surface, the recovery of TiO_2 powder from water to be reused is difficult. Therefore, we develop a simple method to coat TiO_2 on magnetic particles, evaluate its bactericidal effect on fish pathogens, and recollect form water with magnet. Nano-iron-particles are synthesized and added to TiO_2 sol-gel followed by 400 ^{o}C calcinations. Thereafter, TiO_2 are grinded into powder. Particles containing iron are collected with magnet and then their photocatalytic activity is confirmed by the degradation of indigo carmine dye under UV-A irradiation. The particles size, crystal and surface structure are characterized using scanning electron microscope (SEM) and X-ray diffractometry (XRD) before and after evaluation of bactericidal effect on fish pathogens (Edwardsiella tarda and Streptococcus iniae). Highly bactericidal effects are found while no significant change on TiO_2 particles properties after they were recollected easily from water using magnet. This result strongly suggests that TiO_2 particle with iron core can be applied widely in other solution without the TiO_2 recovery problems.

TSP-12 Simultaneous Oxidation and Hydroxyapatite Coating on Titanium and Enhancement of Bioactivity of Osteoblast-Like Cells, *S.K. Moon,* Yonsei University College of Dentistry, Korea, *B.Y. Kim,* Incheon National University, Korea, *K.Y. Kim,* MST Technology Company, Korea, *K. Kim, D.H. Lee,* ***M.H. Hong***, *Y.K. Lee (leeyk@yuhs.ac)*, Yonsei University College of Dentistry, Korea

Titanium alloys have been proved to be very suitable materials for load bearing bioimplant application and have successfully been used in biomedical and dental implants. Unfortunately, titanium exhibits poor osteoinductive properties like most metals. This drawback has recently been addressed by coating the metal with a layer of the hydroxyapatite(HA). Titanium oxide(TiO_2) coatings on titanium alloys have demonstrated acting as a chemical barrier against release of metal ions from the implant. However, the chemical bond with the living bone in the body is not very strong, therefore a double layer of HA/TiO_2 coatings on titanium alloys should possess a very good combination of biochemical stability and mechanical properties. Many techniques have been investigated for deposition of HA onto titanium alloys. Among theses techniques, plasma spraying is the most popular method, but it is difficult to apply uniform coating on implants with complex geometries. Micro-arc oxidati on represents a relatively new surface modification technique where thick, hard and anticorrosive oxide coatings can be easily and cost-effectively fabricated. In this study, a phosphate salt solution was used as the electrolyte for micro-arc oxidation. HA powder and ethylenediol were added to the distilled water for the preparation of the HA suspensions. pH value of the solution was adjusted in the range of 4-11 using ethylenediamine and malonic. For micro-arc oxidation, the specimens were immersed in a phosphate salt with containing various amount of hydroxyapatite. A d.c. of 400 V was applied, giving a current density of 20-40 mA/cm^2 and raising the bath temperature around 75^{o}C. After characterization of the coating film using XRD, SEM and EPMA, osteoblast-like MG 63 cells were cultured onto the hybrid coating film of titanium up to 4 weeks. Culture onto the hybrid coating film exhibited significant higher attachment as well as proliferation of osteoblast-like MC 63 cells than those of titanium alloys ($p<0.05$). This result indicates that a hybrid combination of micro-arc oxidation and electrophoretic deposition could be expected to be a promising coating technique onto titanium alloys.

TSP-13 Preparation and Properties of Branched Polymers as Postoperative Tissue Adhesion Barriers, *S.-R. Hsieh,* Taichung Veterans General Hospital, Taiwan, ***C.-J. Chang*** *(changcj@fcu.edu.tw),* Feng Chia University, Taiwan, *P.-C. Kwan*, Taichung Veterans General Hospital, Taiwan

Undesirable tissue-adhesions after surgical treatment often induce severe problems. Biocompatible polymers and oligomers with pendent polyethylene glycol-polycaprolactone (PEG/PC) diblock or polyethylene glycol-polylactide (PEG/PL) diblock side chains were synthesized as postoperative tissue adhesion barriers. The chemical structure of the side chain and the relative length of each block changed the flexibility of the films. The degradation properties of the polymer/oligomer composite films can be tuned by introducing oligomers with smaller molecular weight. The viability of the fibroblast NIH3T3 cells in the presence of the polymers were assessed using the MTT (3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide) assay. It confirms the low toxicity of the polymers to fibroblast NIH3T3 cells. The animals received surgical defects to their pericardium and left parietal pleura of the chest wall. The polymeric films were applied to and cover the pleural defects o f chest wall. After 28 days, the pericardial and pleural space of the animal was examined by direct observation of adhesion and histological examination. The surgical surfaces covered with the films presented no tissues-adhesion. The histological examination of the tissues confirms that these films are very effective in preventing post-surgical tissue adhesion.

TSP-14 Coating of Electrically Conductive CNT/PTFE Composite Film on the Metal Bipolar Plate for PEMFC, ***Y. Show*** *(show@keyaki.cc.u-tokai.ac.jp), K. Takahashi*, Tokai University, Japan

Metal bipolar plate for fuel cell (FC) has advantages of high manufacturability and mechanical strength. The fuel cell using metal bipolar plates generally shows lower output power than that of carbon bipolar plates, because its surface is corroded and increases contact resistance between bipolar plate and membrane electrode assembly (MEA) in fuel cell. In this study, electrically conductive film, consisting of carbon nanotube (CNT) and Polytetrafluoroethylene (PTFE), was coated on the metal bipolar plate for PEMFC. The composite film coating increased the output power of the Fuel Cell. The CNT/PTFE film was formed from dispersion fluids of the CNT and the PTFE. CNT dispersion was made from multi-wall type CNT. Cellulose derivatives were added into water to disperse the CNT. Water based commercial PTFE dispersion was used in this study. The dispersion fluids of the CNT and the PTFE were mixed and stirred by applying the ultrasonic wave. The CNT/PTFE dispersion was appli ed to stainless steel bipolar plate at the thickness 50mm. The bipolar plates were dried under the atmosphere of 40^{o}C for 30 min, and then were heated at 350^{o}C for 10min. The fuel cell using the bare stainless steel bipolar plates showed maximum output power of 1.7W. The bipolar plate coated with the composite film showed the maximum output power of 2.7W. Impedance analyzer measurement for these FCs indicated that the composite film coating decreased the contact resistance between the bipolar plate and the MEA. Therefore, the FC fabricated with the metal bipolar plate, which is coated with the CNT/PTFE composite film, shows high output power.

TSP-15 Electrical Enhancement of DMFC by Hybrid PVD Pt-M/C Alloy Catalyst, *T.-N. Lin (tnl@mail.mse.ncku.edu.tw),* ***K.-W. Weng****, W.-F. Wang, Y.-L. Chen*, Mingdao University, Taiwan

Direct methanol fuel cells (DMFC) are studied extensively owing to the simple cell configuration, high volume energy density, short start-up time, and operation reliability. However, major drawbacks include high production cost, poisoning of catalyst and methanol crossover. In this study, a simple method in the preparation of Pt-M/C catalysts, using a magnetron sputtering (MS) and a metal-plasma ion implantation (MPII) has been utilized. The Pt catalysts were sputtered onto the gas diffusion layer (GDL), followed by implanting Cr、Fe、Ni、Mo catalysts by MPII (accelerating voltage is 20 kV and implantation does is 1x1016 ions/cm2). The catalyst film crystallinity and microstructure were analyzed by x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), scanning electronic microscopy (SEM), and Transmission Electron Microscope (TEM), respectively. The cell performance was tested by potential stat/galvano stat. The results indicated that the membrane electro de assembly (MEA) of Pt-Cr/C structures can enhance the cell performance of DMFC. The measured power density for methanol concentration at 2M is 0.45 mW/cm^2 and the open circuit voltage (OCV) of 0.334 V.

TSP-16 High Temperature Oxidation of Mild Steel With and Without Al Coating in the Atmosphere Containing Ethanol, *D.H. Chen,* ***M. Badaruddin*** *(D9603801@mail.ntust.edu.tw),* National Taiwan University of Science and Technology (NTUST), Taiwan*, C.J. Wang*, National Taiwan University of Science and Technology (NTUST)

A mild steel of SAPH440 used for motorcycle and automobile exhaust pipe was oxidized by a simulation of high temperature oxidation at 500℃, 600℃, and 700℃. The oxidation behavior influenced by the composition of ethanol and sulfur in the atmosphere was studied by mass change, metal loss, and morphology. The experimental results showed that sulfur, hydrogen and oxygen comprising of the oxidation and sulfidation aggressively attacked the metal. After exposing for 24 h at 600 ℃, the oxide scale creates Fe_2O_3, Fe_3O_4, and FeO from the outer to the substrate, while the hollow whisker of Fe_2O_3 is formed on the surface. In addition, FeS, $Fe_{1-x}S$, MnO, and MnS scatter over the scale. In the outer and inner the oxide scale composes of Fe_2O_3 and Fe_3O_4 under 570℃. Moreover, the scale provides a good adhesion with substrate during high-temperature oxidation. The scale thickens gradually on the surface with the increase of ethanol content. However, the lost metal decreases. For the constant composition of ethanol, the lost metal proportionally reduces against sulfur content.

TSP-20 Modeling Film Growth in Reactive Sputtering Process, ***S. Faddeeva****, J. Oseguera (joseguer@itesm.mx), S. Martínez, F. Castillo*, ITESM-CEM, Mexico

The film thickness and the rate of film growth in reactive sputtering processes are of great practical interest. Based on Berg's model and associated with mass balance during deposition rate, we introduce film thickness and the rate thin layer growth. For these approach different temperatures for substrates, walls and target were considered; chemical reactions for compound formation were assumed. Predicted results are compared with experimental ones.

TSP-21 Influence of a Lateral Electric Field on Nucleation, Growth, and Conductivity of Gold Films on Sapphire, ***M.S. Byrne****, R.J. Lad (rjlad@maine.edu)*, University of Maine

While electric fields directed normal to a substrate are routinely used to modify the energetics of arriving species during film growth, very few studies have investigated the influence of lateral electric fields on the surface transport and resulting morphology of thin films. We have used a Pt electrode structure on r-sapphire substrates to apply a large lateral electric field (up to 10^5 V/m) during the initial nucleation and growth of Au films. The current through a load resistor was measured during film deposition to monitor the film conductivity and to determine the onset of percolation conductivity between the Au nuclei as they begin to coalesce. In addition, atomic force microscopy and high resolution scanning electron microscopy observations were made of film morphology at various Au coverages. Because Au does not wet the sapphire substrate, three-dimensional Au islands form and measurable electrical conductivity does not occur until the equivalent Au film thickness is greater than approximately 8 nm. The film morphology was found to be influenced by the electric field, but the exact mechanism for morphological changes is unclear. The resulting film structures formed in the presence of the lateral electrical field may be attributable to a number of factors including enhanced surface adatom transport, local heating effects, and electromigration.

TSP-22 A Theoretical Model for the Influence of Surface Morphology of the Template Side and Barrier Layer Sides of AAO on the Growth Kinetics of ZnO Nanowires, ***S.H Deulkar****, J.L Huang (jlh888@mail.ncku.edu.tw), H.J. Yo*, National Cheng Kung University, Taiwan

ZnO nanowires are grown inside AAO pores existant on the template side while simultaneously ZnO nanowires are grown on the barrier layer side of the AAO substrate by using CVD deposition method. The deposition temperature is 903 K while the chamber pressure is 4.45 torr. The Oxygen flow rate and the carrier gas (Ar) flow rates is respectively 0.45 and 23 sccm.

The kinetics of the growth of nanowires inside the AAO pores is found to be dependent on the vertical velocity gradient of the gas phase existant inside the CVD tube as well as the ambient temperature,while the kinetics of growth of ZnO nanowires on the barrier layer side of the substrate is found to be influenced by the hexagonal network of cells with convex surfaces with narrow grain boundaries. ZnO nucleation takes place at the grain boundary via VLS mechanism, as grain boundaries provide low surface energy for nucleation.

EDS scan during TEM analysis of the FIB samples revealed the variation in Atomic percentage of ZnO ranging from the tip of the nanowires to the interior of the pores. TEM studies revealed the single crystal structure of the nanowires grown within the pores as well as those grown on barrier layer sides,while micro PL studies revealed the existence of a distinct excitonic bandgap for the nanowires grown on either surface of AAO.

Numerical computation based on the characterization data has been utilized to develop a simplistic growth model that verifies albeit qualitatively the subtleties of growth kinetics of ZnO nanowires, prevalent on the two morphologically different surfaces of AAO.

TSP-23 A Study About the Biocompatibility of Titanium Oxide Nanotubes Prepared by Anodic Oxidation, ***H. Ahn*** *(henrie90@gmail.com), D.K. Kim, B.-O. Kim, D.-K. Kim, C.S. Kim, S. Lee,* Chosun University, Korea*, B. Lee,* Gallions Reach Health Centre, United Kingdom*, J.B. An, H.-C. Choe*, Chosun University, Korea

We have examined and discovered the effect of heat treatment on the crystalline of titanium nanotubes which were prepared by anodic oxidation in an electrolytic solution in 1M phosphoric acid and 1.5 wt% fluoride acid at constant voltage of 20 for 10 minutes. Then, the formed nanotubes were heat treated at 200 – 800℃ in the air, oxygen, and vacuum, respectively. Cytotoxicity tests were performed in MTT assay which treated L929 fibroblast cell culture using indirect method. The microstructure changes of the activated surfaces were observed by XPS, FE-SEM, XRD and TEM. We found that the titanium nanotubes had the diameter of 90~95nm, the length of 400~500nm, and the thickness of 11nm. After heat treatment at the high temperatures, the diameter and length of nanotubes became smaller and shorter, respectively, and the thickness became thicker. The phases of titanium nanotubes are amorphous at 200℃, amorphous + anatase at 300℃, amorphous + anatase + rutile at 500℃, and rutile (almost 95%) at 800℃. Under three different conditions heat treated nanotubes showed no significant difference in the crystalline.

TSP-24 Growth Characteristics and Sintering Behavior of YSZ Thin Film Prepared by E-Beam Evaporation at Various Pressures, ***H.-H. Huang*** *(funs@csu.edu.tw),* Cheng Shiu University, Taiwan*, M.-C. Huang, M.-H. Chen, C.-F. Yang,* National Kaohsiung First University of Science and Technology, Taiwan*, C.-Y. Hsu*, Cheng Shiu University, Taiwan

Deposited at working pressure ranging from $1*10^{-5}$ to $1*10^{-3}$ Torr, the yttria-stabilized zirconia, YSZ, thin film had been prepared by E-beam evaporation at substrate temperature of 200°C. The XRD, TEM, SEM and UV-Vis were respectively used to determine the structure, microstructure, morphology and transmittance of YSZ thin films. Results show that the YSZ thin film with fluorite phase was obtained and XRD reflection peaks of (111), (200), (220), (311), (222) and (400) were found. The preferred orientation of [200] was found when thin films prepared at low working pressure, however, the [111] was at high pressure. The grain size and internal strain of YSZ films both decrease with working pressure increasing. The transmittance of YSZ films deposited at various working pressures is similar which is in the range of 70-90%, meanwhile, the energy gaps of YSZ thin film were similar in the range of 3.73-3.78 eV. After 1200°C sintering, the nano-grain ed morphology was obtained.

Coatings for Use at High Temperature
Room: Royal Palm 1-3 - Session A3-2

Thermal Barrier Coatings
Moderator: A. Bolcavage, Rolls-Royce Corporation, R. Mevrel, ONERA, K. Murphy, Howmet Castings

8:00am **A3-2-3 Moisture-Induced Spallation of Thermal Barrier Coatings**, ***M. Rudolphi*** *(rudolphi@dechema.de), D. Renusch, M. Schütze*, DECHEMA e.V., Germany

Spallation of thermal barrier coatings (TBCs) usually occurs during cool down when thermal expansion mismatch stresses are maximal. However, sometimes a delayed failure of the coating is observed at the end of their life-time, which is the spallation of the coating after the sample is cold. This so called Desktop Effect is believed to be strongly influenced by the presence of water (i.e. water vapor) in the ambient environment. While the influence of water / water vapor on corrosion rates at high temperatures has been under investigation for several decades, the detrimental effect of water on oxide scale adhesion has received only little attention or even remained unnoticed. In order to develop a deeper understanding of the underlying mechanisms of moisture induced spallation a series of experiments was designed that incorporates elemental analysis as well as crack detection. Hydrogen concentration depth profiles derived from nuclear reaction analysis were measured on A PS TBCs after pre-oxidation in dry and humid environments to clarify the role of hydrogen and possible transport mechanisms. Acoustic emission measurements and metallographic investigations were employed to investigate the micro-crack evolution.

8:20am **A3-2-4 Thermal Barrier Coatings Adherence and Spallation : Interfacial Indentation Resistance and Cyclic Oxidation Behaviour Under Thermal Gradient**, ***J. Sniezewski*** *(sniezews@enstimac.fr), V. Vidal, Y. Le Maoult, P. Lours*, Université de Toulouse, France

Thermal barrier coatings adherence and spallation : interfacial indentation resistance and cyclic oxidation behaviour under thermal gradient Thermal barrier coatings (TBC) are complex multi-materials systems used in hot parts of gas turbines. In service, one of the most critical damage results from the spallation of the yttria stabilized zirconia (YSZ) layer, leading to a detrimental temperature increase of the underlying superalloy. The toughness of the interface between the bond coat and the top coat is the main parameter that controls the adherence of the zirconia layer. This parameter can be straightforwardly approached by loading the interface using an appropriate indentation technique and determining the critical force required to cause the delamination of the TBC. The resulting interfacial toughness is calculated for as-deposited TBC and cyclically oxidized TBC. The cyclic oxidation of TBC is performed using a dedicated equipment, recently developed in order to r eproduce as close as possible the real in-service thermal conditions of gas turbines. Specifically, the cyclic oxidation test permits to generate and control a thermal gradient through the TBC system. In addition, the equipment is instrumented with a video camera to monitor in situ the interfacial crack propagation and the resulting spallation of the TBC. An analysis of the interfacial toughness evolution as the function of the type of cyclic oxidation is proposed. Namely, the number of cycles, the oxidation temperature and the holding time at high temperature as well as the magnitude of the thermal gradient are considered and thoroughly studied. Concomitantly, the mechanisms of crack initiation and growth and the modes of spallation are investigated and critically discussed.

8:40am **A3-2-5 Correlation of Mechanical Properties and Electrochemical Impedance Spectroscopy Analysis of Thermal Barrier Coatings**, *J. Gómez-García, A. Rico, C.J. Múnez,* ***P. Poza*** *(pedro.poza@urjc.es), V. Utrilla*, Universidad Rey Juan Carlos, Spain

Thermal barrier coatings (TBC) are widely used in turbine engines for propulsion and power generation where materials to withstand increasing operating temperatures, mechanical loads and chemical degradation are required. The aim of these coatings is to insulate the metallic components from the aggressive environment at high temperature increasing turbine entry gas temperature, which promotes overall engine efficiency. TBCs comprise at least two layers: a ceramic top coating and a metallic bond coat. This metallic layer increases the adhesion of the ceramic coating and provides enhanced oxidation and corrosion resistance. To this end, these coatings should be capable to develop a surface oxide layer thermodynamically stable, slow growing and adherent. During service at high temperature a thermally grown oxide layer (TGO) is formed between the metallic overlay coating and the ceramic top coat. TGO growing is the most important phenomenon controlling TBC durability. Out of plane stresses are developed at the TGO - bond coat and top coat interfaces. These stresses increase as TGO thickens promoting fracture at the interfaces and cracking within the brittle ceramic coating, modifying the top coat apparent mechanical properties. Finally, the system fails by spalling. For these reasons, it should be interesting to analyse the degradation status during service by a non destructive method. Electrochemical impedance spectroscopy (EIS) analyse the system impedance which will be modified by TGO thickening and top coat cracking. The aim of this work is to analyse by EIS two TBCs after isothermal oxidation in air and correlate the electrical parameters, which characterize the system status, with the apparent mechanical properties. The TBCs analysed were a $CaZrO_3$ top coat, sprayed onto an AISI 304 substrate using NiAlMo as overlay coating, and a $ZrO_2(Y_2O_3)$ ceramic coating, plasma sprayed onto an Inconel 600 substrate using NiCrAlY as bond coat.

9:00am **A3-2-6 The Response of Zirconia Layers under External Thermal and Mechanical Solicitations: Microstructural Evolution and Mechanical Stability**, *B. Benali, A.M. Huntz,* ***M. Andrieux*** *(michel.andrieux@u-psud.fr),* University Paris Sud 11, LEMHE-ICMMO, France, *F. Jomard,* Groupe d'Etude de la Matière Condensée (GEMaC), France, *M. Ignat*, SIMAP Grenoble INP, France

The microstructural evolution and the mechanical response of zirconia coatings deposited by OMCVD on steel substrates, were analysed before and after being submitted to thermal and/or mechanical solicitations. The coatings were deposited at two different temperatures and with different partial pressures of oxygen. The as deposited zirconia films presented two phases: the monoclinic one, as the metastable tetragonal phase. With these obtained films on substrate systems, two kinds of in-situ experiments were performed: deflection under controlled atmosphere and temperature gradients and tension with and without heating. The experimental results showed a strong dependence of the mechanical stability of the films to their deposition conditions. Their microstructural evolution in terms of phase transformation, grain sizes, internal stresses was then analysed from XRD experiments. These analyses allowed defining the limits of the domains of the phases, and their amount of transformation.

9:20am **A3-2-7 Understanding and Modeling of Multicomponent Interdiffusion for Life Prediction and Life Extension of Thermal Barrier Coatings**, ***Y. Sohn*** *(ysohn@mail.ucf.edu)*, University of Central Florida **INVITED**

Solid-state multicomponent interdiffusion is a subject of great interest for its intellectual merit and practical applications in materials and coatings for high temperature applications. Along with a brief review of framework for phenomenological descriptions, this talk will survey the importance of multicomponent-multiphase interdiffusion with specific examples from superalloys and thermal barrier coatings (TBCs) used in gas turbine engines. Results and analysis from laboratory experiments, field applications and microstructural modeling are presented to highlight the cross-fertilization of science and applications. Experimental concentration profiles of individual components in experimental diffusion couples using NiAl, Ni3Al and various Fe- and Ni-base austenitic alloys and superalloys are examined to determine interdiffusion fluxes, which are then directly integrated to determine multicomonent interidiffusion coefficient. Understanding of diffusional interactions v ia quantified interdiffusion coefficients then can be employed to design and select alloys and coatings that will provide microstructural stability for durable applications at high temperature. Phase field modeling can also provide mechanistic understanding of multicomponent interdiffusion and microstructural development that can lead to life prediction models and life extension methodologies. Using available thermodynamics and mobility data, a phase field model has been utilized to simulate and predict interdiffusion behavior and microstructural evolution in multi-phase diffusion couples of Ni-Al and Ni-Cr-Al systems. Based on existing theories and observations of failure mechanisms, approaches to model critical phenomena associated with thermal barrier coating (TBC) failure, namely sintering of ytrria-stabilized zirconia (YSZ) topcoat, (t'→f+m) phase transformations in YSZ topcoat, high temperature oxidation (i.e., growth of thermally grown oxide, TGO) of bond coats, mu lticomponent-multiphase interdiffusion between bond coat and superalloy substrate, and mode-III fracture at the YSZ/TGO and TGO/bond coat interfaces are presented.

10:00am **A3-2-9 Comparison of the Oxidation Behavior of Beta and Gamma - Gamma Prime NiPtAl Coatings**, *J.A. Haynes (z15@ornl.gov),* ***B.A. Pint,*** Oak Ridge National Laboratory, *Y. Zhang,* Tennessee Technological University, *I.G. Wright*, Oak Ridge National Laboratory

A new class of gamma-gamma prime platinum diffusion coatings are receiving increasing attention for use as a bond coat in thermal barrier coating applications. This study investigated and compared the oxidation behavior of gamma-gamma prime NiPtAl coatings to the more traditional beta-phase NiPtAl bond coatings formed by chemical vapor deposition. Both types of NiPtAl coatings were fabricated on the same Ni-base superalloys (single crystal Rene N5 and CMSX-4) using the same electroplated Pt source. Coatings were exposed to identical cyclic oxidation tests at 1100 and 1150C. Compositions of selected coatings were characterized by electron microprobe analysis to evaluate distribution of Pt and Al after 1,000 oxidation cycles. Coatings and oxidation products were characterized by scanning electron microscopy. The thickness, integrity and microstructure of the alumina scales were compared as a function of temperature, coating type and superalloy composition.

10:20am **A3-2-10 Effect of Exposure Conditions on the Oxidation of MCrAlY-Bondcoats and Lifetime of Thermal Barrier Coatings**, ***J. Toscano*** *(j.toscano@fz-juelich.de), M. Subanovic, E. Wessel, D. Naumenko, L. Singheiser, J. Quadakkers*, Research Center Juelich, Germany

The high temperature oxidation and corrosion resistance of TBC-coated blades in gas turbines must be guaranteed under varying conditions in power generation. The introduction of new CO_2-capture technologies implies the operation of these materials in combustion gases with higher $CO_2$and water vapor concentrations and possibly lower oxygen levels than in the conventional power generation technologies.

Aiming to assess the oxidation behavior of the typically employed MCrAlYs under such varying conditions, different experiments were carried out in air and H_2O-containing gases at temperatures between 1000-1100°C. The long term behavior of overlay coatings and free-standing MCrAlYs with chemical compositions similar to those commonly used in gas turbine blades was investigated. The oxidation kinetics was evaluated through mass gain measurements on the latter after predetermined intervals. Additionally, the effect of MCrAlY oxidation behavior on th e lifetime of ceramic TBC coatings in the mentioned atmospheres was estimated. Subsequently, extensive characterization of the oxide scales formed and the internal oxidation encountered was performed using light microscopy, Laser Raman Spectroscopy and SEM/EDX. The results of the microstructural studies were correlated with the scale growth kinetics and the long-term TBC performance.

Hard Coatings and Vapor Deposition Technology
Room: Golden West - Session B6-3

Hard and Multifunctional Nano-Structured Coatings
Moderator: M. Stueber, Forschungszentrum Karlsruhe, C.P. Mulligan, Benet Laboratories, U.S. Army ARDEC, R. Sanjines, EPFL

8:00am **B6-3-1 Progress in the Development of Adaptive Nitride-Based Coatings for High Temperature Tribological Coatings**, ***S. Aouadi*** *(saouadi@physics.siu.edu), B. Luster, P. Kohli,* Southern Illinois University Carbondale, *C. Muratore,* Air Force Research Laboratory/UTC, Inc., *A.A. Voevodin*, Air Force Research Laboratory **INVITED**

Adaptive tribological coatings were recently developed as a new class of smart materials that were designed to adjust their surface chemical composition and structure as a function of changes in the working environment to minimize friction coefficient and wear between contact surfaces. This presentation provides an overview of the current research developments in this field, including: (1) Chameleon nanocomposite coatings which are produced by depositing a multi-phase structure whereby some of the phases provide mechanical strength and others are lubricious; (2) Micro- and nano-textured coatings which consist of ultra-hard nitride films with highly ordered micropores and nanopores that are subsequently filled with solid lubricants using various techniques such as lithography, reactive ion etching, laser texturing, pulsed air arc treatment, and ceramic beads as placeholders for sputter deposition; and, (3) Carbon and nitride nanotubes that are filled electrochemically with solid lubricants. The frictional and wear properties of the above three classes of newly developed adaptive structures, tested in various controlled environmental conditions (temperature, humidity), will be discussed in detail.

8:40am **B6-3-3 Influence of the Chemical Composition on Tribological Properties of Nitride-Based Nanocomposite Coatings**, *P. Dessarzin,* ETH Zurich and PLATIT AG, Switzerland, *P. Karvankova,* ***M. Morstein*** *(m.morstein@platit.com),* PLATIT AG, Switzerland, *N.M. Renevier,* University of Central Lancashire, United Kingdom, *N.D. Spencer*, ETH Zurich, Switzerland

As a consequence of the increased use of dry machining strategies such as high-speed cutting (HSC), high-performance cutting (HPC) and hard turning, modern tool coatings are constantly challenged by severe environments, combining oxidative, abrasive and adhesive wear.

In order to evaluate the role of coating chemical composition in related high-temperature situations, this study compares friction, wear and oxidation behaviour of different conventional and nanocomposite PVD coatings, deposited using the rotating arc cathodes technology.

At room temperature, dry-sliding experiments were carried out in dry air on coated HSS plates against metalloid (WC-Co) and ceramic (Si_3N_4) counterparts. For both pin materials, a distinct decrease of the coating wear coefficient was found upon addition of carbon, silicon or chromium to the $Ti_{1-x}Al_xN$ base system. In contrast, the coefficient of friction remained essentially unchanged except for the case of carbon. SEM investigations showed that the wear was dominated by an abrasive mechanism, with only few cases of ball material transfer to the friction track. Neither fracture nor delamination of the coatings were observed.

High-temperature pin-on-disk tests of coatings on cemented carbide plates against alumina balls revealed early failure of carbon-containing coatings due to their low oxidation resistance. Most nitride and oxynitride coatings were able to withstand the conditions with only minimum oxide scale formation, yet the beneficial effect of Cr addition on the wear resistance was found to be less pronounced. The wear volume at 600°C correlated well with dry milling tests in steel, however not strongly with room-temperature nanomechanical properties of the coatings. Therefore, the hot hardness and -modulus of selected coatings were measured in situ, using a special indenter setup.

9:00am **B6-3-4 Development and Properties of Advanced Nitride Coatings for Tooling Applications**, ***M. Lechthaler*** *(markus.lechthaler@oerlikon.com), F. Neff, E. Plesiutsching,* OC Oerlikon Balzers AG, Liechtenstein, *R. Franz*, University of Leoben, Austria

Advanced manufacturing technologies such as high performance cutting augment the requirements of wear protective coatings for tooling applications. Within this work, recent investigations in industrial coating development of arc evaporated coating applications are presented. In order to achieve superior coating properties, additional alloying elements including Si and B as well as transition metals were added to the highly investigated Al-Cr-N coating system. Moreover, the improvement of the coating properties due to fine tuning of deposition parameters is discussed. This allows the design of new coatings with tailored coating properties leading to superior performance in machining applications. Furthermore, investigations are highlighted on innovative promising material systems for new wear resistance coatings such as AlNbN. X-ray diffraction and scanning electron microscopy as well as transmission electron microscopy analyses were carried out to investigate the microstructure and morphology of the coatings. The mechanical properties are characterized by nanoindentation measurements, whereas annealing tests in ambient atmosphere served to determine the oxidation resistance. Finally, the correlation between mechanical properties and the application performance of these coatings is evaluated in cutting tests. The comparison to state-of-the-art coatings allows the estimation of the potential of these coatings for future industrial use.

9:20am **B6-3-5 Influence of B on Structural, Mechanical and Tribological Properties of Arc Evaporated Al-Cr-N Thin Films**, ***C. Tritremmel*** *(christian.tritremmel@unileoben.ac.at), P.H. Mayrhofer,* University of Leoben, Austria, *M. Lechthaler,* OC oerlikon Balzers AG, Liechtenstein, *C. Polzer,* PLANSEE Composite Materials GmbH, Austria, *C. Mitterer*, University of Leoben, Austria

Al-Cr-N is a well-established hard coating system with excellent mechanical and tribological properties. Structure and mechanical properties of transition metal nitrides like TiN or CrN can be further improved by boron addition; thus we systematically investigated its effect on Al-Cr-N. Coatings were synthesized by cathodic arc evaporation in pure N_2 atmosphere at 3.5 Pa and 500°C in an industrial-scale Oerlikon Balzers Innova deposition system. The B content in the targets was varied between 10 and 20 at% at constant Al/Cr atomic ratio of 1.8. X-ray diffraction revealed that all coatings exhibit a face-centered cubic structure in the as-deposited state. Texture and mechanical properties of the coatings are, however, slightly changed by varying substrate bias and nitrogen pressure. Residual stress measurements indicated a low compressive stress level (-1.2 GPa) even at high substrate bias. Nanoindentation experiments revealed a

significant hardness enhancem ent in comparison with B-free Al-Cr-N coatings, where a hardness maximum of 43 GPa was found for Al-Cr-B-N synthesized using the target with 10 at% B. Tribological ball-on-disc tests confirmed that B addition is a promising attempt for further improvement of Al-Cr-N coatings, yielding wear coefficients in the range of $1x10^{-17}$ m^3/Nm at room temperature.

9:40am **B6-3-6 From Understanding the Growth Mechanism to the Design and Fabrication of High-Performance Functional Coating Architectures**, ***L. Martinu*** *(ludvik.martinu@polymtl.ca),* Ecole Polytechnique de Montreal, Canada*, A. Amassian,* Cornell University*, E. Bousser, S. Hassani, J. Houska, R. Vernhes, J.E. Klemberg-Sapieha*, Ecole Polytechnique de Montreal, Canada **INVITED**

Recent advances in the technological sectors of aerospace, automotive, biomedical and pharmaceutical applications as well as in energy and environment control stimulate research on high performance functional coatings. In many cases, the ever increasing requirements involve an "ideal" combination of the mechanical, tribological, corrosion, thermal and other characteristics that can only be satisfied by using specifically tailored film architectures including nanocomposite, nanolaminate, multilayer and graded layer systems.

In this presentation, we will outline approaches that allow one to design and fabricate high-performance protective coatings based on understanding the film growth mechanisms, on the lessons learned in the area of optical coatings such as optical interference filters, and on the use simulation techniques. We will briefly describe our recent studies of the ion-surface interactions in plasma environments (bias- and pulse-controlled PECVD and PVD techniques) using a methodology combining in situ real-time spectroscopic ellipsometry and different complementary microstructural and chemical analysis methods, while specifically addressing selected nanostructured systems such as Ti-Si-N, Ti-Si-C-N and Cr-Si-N coatings.

We will show examples in which the modeling approach helped us to enhance our understanding of the growth-structure-property relationships, and that allowed us to predict functional behavior of different coating combinations; this includes: (i) Dynamic Monte-Carlo simulation of the ion bombardment effects on subplantation and interfacial mixing; (ii) Molecular dynamic simulations of the nanocomposite structures capable of predicting the experimentally observed superhardness; and (iii) Finite element design of film architectures for the prediction of enhanced resistance to solid particle erosion. Finally, we will discuss new opportunities and surface engineering strategies leading to attractive technological solutions in the areas of protective coatings for aerospace, biomedical and optical applications.

10:20am **B6-3-8 Synthesis of the Al-Cr-O-N Coatings by Cathodic Arc Evaporation**, ***D. Kurapov*** *(denis.kurapov@oerlikon.com), T. Bachmann, H. Rudigier,* OC Oerlikon Balzers AG, Liechtenstein*, M. Doebeli*, Ion Beam Physics, Paul Scherrer Institut and ETH Zuerich, Switzerland

In the present work we report on the chemical composition, structure, mechanical properties, and cutting performance of the Al-Cr-O-N coatings deposited by cathodic arc evaporation. The coatings have been synthesized using Al_xCr_{1-x} alloyed targets where x varies from 0 to 0.85. The reactive gas mixture was adjusted in a way to obtain coatings with composition variying from AlCrN to $(Al,Cr)_2O_3$. The chemical composition of the coatings was investigated by means of Rutherford backscattering spectroscopy (RBS). The evolution of the crystallographic structure as a function of chemical composition was studied by X-ray diffraction (XRD). The morphology of the coatings was analyzed by scanning electron microscopy (SEM). Mechanical properties of the coatings were measured by nanoindentation. Cutting tests were performed in order to correlate properties of the coatings with their cutting performance.

10:40am **B6-3-9 Combinatorial Approach to the Growth of α-$(Al,Cr)_2O_3$ Solid Solution Strengthened Thin Films by Reactive r.f. Magnetron Sputtering**, ***M. Stueber****, D. Diechle (dominic.diechle@imf.fzk.de), H. Leiste, S. Ulrich,* Forschungszentrum Karlsruhe, Germany*, V. Schier*, Walter AG, Germany

The development of superior coatings for high performance cutting tools is a key factor for significant advances in metal working. Aluminum oxide thin films deposited by chemical vapor deposition (CVD) methods are industrially well-established since years. Recently, the physical vapor (PVD) synthesis of aluminum oxide and derivative coatings is attracting large scientific and technical interest. Especially aluminum-chromium oxide coatings are promising candidates for offering simultaneously thermal stability, chemical inertness, excellent high temperature toughness and hardness with regard to their mixed ionic and covalent bonds. A combinatorial approach to the growth and microstructure evolution of Al-Cr-O thin films by means of reactive r.f. magnetron sputtering is presented. A segmented target consisting of two half plates of Al and Cr was used for the deposition experiments carried out under stationary conditions in a laboratory scale PVD coater (Leybold Z 550). Opposite to the cathode five substrate samples (commercial cemented carbides, silicon wafers) were placed in equidistant positions. The r.f. cathode power was set to 500 W and the total gas pressure was kept constant at 0.65 Pa for all experiments with a fixed oxygen to argon gas flow ratio. The substrate temperature was varied between 180°C and 600°C, while the r.f. substrate bias voltage was varied systematically between 0 V and -400 V. Detailed results on the coatings composition, constitution, microstructure and properties will be presented. XRD and TEM studies clearly show that the growth of well adherent, nanocrystalline, stoichiometric, metastable corundum-like solid-solution strengthened $(Al_{1-x},Cr_x)_2O_3$ thin films with a high degree of crystallinity and Vickers hardness up to 2600 HV0.05 is possible at non-equilibrium conditions already at low substrate temperatures of 500°C.

11:00am **B6-3-10 Dense Nanostructured Oxide Coatings Made By Plasma Spray and Conventional Consolidation Processes**, ***M. Gell*** *(mgell@mail.ims.uconn.edu),* University of Connecticut, United States*, E.H. Jordan, J. Wang, C. Muoto*, University of Connecticut

Dense oxide coatings are traditionally used in wear, environmental and functional applications. There is an increasing body of research that demonstrates that superior properties for these applications can be obtained if the grain size is reduced into the nano-scale range (<100 nm). In this study, magnesis-yttria nanoscale powder, with a grain size of about 50 nm, is produced by a combustion synthesis process. This powder is then consolidated using a variety of methods, including vacuum hot pressing, hot isostatic pressing, spark plasma sintering, and suspension plasma spraying. The powder is also agglomerated so that it can be directly plasma sprayed. The microstructure and hardness of the resultant coatings made from the various processes will be described.

Optical Thin Films
Room: Royal Palm 4-6 - Session C3

Optical Characterization of Thin Films
Moderator: U. Beck, BAM Berlin, T. Tiwald, J.A. Woollam Co., Inc.

8:00am **C3-1 Spectroscopic Ellipsometry for Characterization of Thin Films and Surfaces: Harnessing Materials for Energy**, ***M. Schubert*** *(schubert@engr.unl.edu)*, University of Nebraska-Lincoln **INVITED**

Greenhouse gas emission, global climate concerns and modern lifestyles transform energy production, consumption and efficiency to be the most critical problem of our society. Harnessing materials for energy is seen momentarily as the only key to generating our future energy infrastructure in balance with rising global economic and ecologic imbalance. In this presentation we critically review our current perspective on spectroscopic ellipsometry – a nondestructive optical characterization technique – in energy materials research. Specifically, we address most recent progress towards boosting knowledge for thin-film solid state lightning, solar cell and battery storage applications: The optical Hall effect. In this newly developed approach, thin film heterostructures are studied under strong external magnetic fields, and unbound charge excitations are monitored which thereby reveal signatures of their density, effective mass and mobility, very much in similarity to the electrical Hall effect. However, unlike the electrical Hall effect, the optical Hall effect does not require electrical contacts, and can provide increased information if multiple layered structures are investigated. Examples include micro and nanostructured silicon solar cells – today's photovoltaic conversion work horse, surface depletion and accumulation layer properties in InGaN – a novel candidate for multiple-junction solar cells, terahertz resonances in chiral metal nanowires –candidates for harvesting electromagnetic radiation energy, a study of the origin of the intercalated charge induced polaron formation in tungsten oxide – an energy storing material candidate, and magnetization properties in antiferromagnetically ordered ZnMnSe – a potential spintronic element.

8:40am **C3-3 Effect of Laser Fluence on ZnCdS Thin Films**, *S. Singhal,* ***A.K. Chawla****, R. Chandra (ramesfic@iitr.ernet.in), H.O. Gupta*, Indian Institute of Technology Roorkee, India

II–VI Zn based ternary alloys are of great interest because of their applications in the production of devices such as short wavelength emitting laser diodes and light emitting diodes. Their electronic band structure and the wide range of band gap together with a low refractive index allow intense light transmission and also effective electron transport at high electric field. The interest in Zn based ternary alloys is also particularly concerned with the increasing demand for materials useful for the

production of flat panel displays, high efficiency electroluminescent and field emission devices. Among the new and up to date techniques used for thin film deposition, pulsed laser ablation (PLA) is one of the most versatile methods to obtain layers of several materials that can be processed into a pellet target. One of the important features of this method is based on the possibility of maintaining the stoichiometry of the ablated target in the deposited layer. We have deposited the nanocrystalline thin films of $Zn_{1-x}Cd_xS$ (x = 0.4) by pulsed laser ablation on corning glass substrates. We have varied the laser fluences during deposition keeping all other parameters fixed. The deposited samples were characterized using X-ray diffraction for the phase purity and crystal structure. The stoichiometric composition of these alloys was estimated using lattice constant calculated from the XRD data. Surface morphology of the samples was examined using AFM and FE-SEM. Optical properties were studied at room temperature by transmittance, reflectance, and Photoluminescence measurements. It was observed from the PL data that with increase in laser fluence there is a decrease in the band gap. Transmission data shows a transmittance of more than 70% in the visible region recorded by UV-Vis-NIR spectrophotometer. TEM investigation of the samples reveals that the particles are spherical in shape with average diameter of 15-20 nm.

9:00am **C3-4 Effect of Rapid Thermal Annealing on the Electrical and Optical Behaviors of Cu2O-Ag Nanocomposite Thin Films**, ***C.-C. Tseng***, National Chung Hsing University, Taiwan, *J.H. Hsieh,* Mingchi University of Technology, Taiwan, *W. Wu (wwu@dragon.nchu.edu.tw)*, National Chung Hsing University, Taiwan

Cu2O–Ag nanocomposite thin films were deposited by reactive co-sputtering on glass substrates. After deposition, some of these films were annealed using a rapid thermal annealing (RTA) system, with the variation of temperature and time. A UV-VIS-NIR photometer and a Hall measurement system were used to characterize the optical and electrical properties of these films with and without RTA. The results reveal that annealing by RTA can cause Ag nano-particles to emerge in the Cu2O matrix. Consequently, the optical properties of these films will change, as well as the electrical behavior. The effects of embedded Ag particles on photo-induced conductivity are discussed.

9:20am **C3-5 Laser Damage Thresholds of Optical Coatings**, ***D. Ristau*** *(d.ristau@lzh.de)*, Laser Zentrum Hannover e.V., Germany **INVITED**

Since the very beginning of laser technology, Laser Induced Damage Thresholds (LIDT) of optical components were always an obstacle for the application of laser systems operating at high power levels. Also, further progresses in the development of new high power laser concepts are often directly limited by the availability of advanced optical components with high quality and LIDT-values. Nowadays, in the course of the development of optical materials with excellent quality and power handling capability, the problem of laser induced damage has shifted from the bulk to the surface of the optical component. The optical surface is objected to various production steps and environmental influences, which modify its structure and composition. Especially, the thin film coating, which is deposited on the optical surface to adapt its reflectance and transmittance to the application, contributes predominantly to the reduction of the LIDT-values. As a consequence, the measurement and optimization of the power handling capability of thin films is considered as one of the primary research areas in modern optics technology and is supported by an extensive scientific community.

In the present paper, a brief review will be given on selected fundamental damage mechanisms in thin films considering different operation conditions of modern laser systems. Also, the current standards for the measurement of LIDT will be described, and examples illustrating some practical aspects of high power optical coatings will be presented. Finally, a summary of the present state of the art will be given, and recent trends in laser technology will be discussed in respect to research in laser induced damage.

10:00am **C3-7 Numerical Ellipsometry: Analysis of Thin Metal Layers Using n-k Plane Methods with Multiple Incidence Angles**, ***F.K. Urban*** *(fk_urban@yahoo.com), D. Barton,* Florida International University, *T. Tiwald*, J.A. Woollam Co., Inc.

Ellipsometry is an optical analytical method based on measuring the change in polarization state of reflected or transmitted polarized light. A major challenge for those utilizing this method has been the computation of reflecting surface physical parameters of interest from the raw measured data. These methods in common use are plagued by local minima and algorithm performance. A recent study examined existing methods for modeling thin metal layers on various substrates. Previously we have applied Complex Analysis in the n-k plane to improve ellipsometry modeling for growing films on substrates. The work presented here applies this advanced methodology to multiple angle measurements over a wavelength range spanning visible for thin absorbing metal films deposited on various substrates. Results show that the new methods work well across light incidence angle and across wavelength. Relative advantages for this kind of film-substrate combination will be presented.

10:20am **C3-8 Evaluation of Optical and Surface Properties of Coated and Painted Materials by Multispectral Imaging**, ***J.A. Toque***, Kyoto University, Japan, *A. Ide-Ektessabi (h51167@sakura.kudpc.kyoto-u.ac.jp)*, Advanced Imaging Technology Laboratory, Japan

Techniques based on the principle that materials emit unique spectral features when subjected to incident radiation have been applied in the study of painted and coated materials, especially those intended for decorative purposes as well as coatings where optical properties take precedence. Among these techniques, spectroscopic ellipsometry and goniospectroscopy are commonly used to verify color and spectral information and measure material constants. However, the data they yield are limited to the spectral response and derived material constants. In this study, an imaging technique, which can give both spectral and spatial information were explored to investigate painted and coated materials. The set-up composed of: a monochromatic CCD; a mixture of halogen and metal halide light source; and spectrally selective filters, was used to capture multispectral images within the visible to near-infrared spectrum (380-850 nm wavelengths). This spectrum range was shown to be im portant in studying optical and surface properties of coatings. Color information, spectral characteristics and surface features were derived from the images using RGB reconstruction, pseudoinverse method and stereo photometric approach respectively. Results of the analysis were used to predict chemical composition and correlate fine structural change brought by degradation from spectroscopic data. By varying the illumination conditions (e.g. angle, intensity, etc.), surface characteristics were also modeled, visualized and quantified. The results of the modeling and reconstruction were verified by established analytical techniques such as synchrotron radiation x-ray fluorescence and x-ray absorption fine structure (SRXRF and SRXAFS) and commercially available spectrometers. Preliminary results were found to be promising especially since the technique does not require complex tooling and data can be acquired almost instantly. It is also non-destructive and non-invasive with a lmost no limitation on the sample size. Further studies on the application of multispectral imaging to coatings may lead to developments of new analytical techniques for investigating painted and coated materials.

10:40am **C3-9 Investigation of Scattering Mechanisms in Transparent Conductive Ga-doped ZnO Films with Thicknesses of Less than 100 nm**, ***T. Yamada*** *(yamada.takahiro@kochi-tech.ac.jp), A. Miyake, H. Makino, N. Yamamoto, T. Yamamoto*, Kochi University of Technology, Japan

Low resistivity and highly transparent Ga-doped ZnO (GZO) films with different thicknesses of less than 100 nm were deposited on alkali-free glass substrate by an ion-plating method using a direct-current arc discharge, where the deposition conditions are optimized in terms of a reduction in resistivity[1]. Complex dielectric functions of the GZO films in a photon energy range from 0.73 to 3.8 eV were characterized by an ellipsometric model analysis constructed from the Drude and Tauc-Lorentz functions, assuming that the GZO films are a homogeneous. From the Drude model analysis for free-carrier response, optical carrier concentration, N_{opt}, and mobility, μ_{opt}, of the GZO films were obtained, and they were compared with carrier concentration, N_{Hall}, and Hall mobility, μ_{Hall}, determined by Hall effect measurements. While the N_{opt} was in near agreement with the N_{Hall}, the μ_{opt} exhibited the values higher than $\mu_{Ha\ ll}$ in all the films. The difference in the mobility is attributed to grain boundary scattering of carriers. From relationship between the electrical and optical properties of the GZO films with increasing film thickness of less than 100 nm, scattering mechanisms of carrier were discussed. [1] [1]T. Yamada, A. Miyake, S. Kishimoto, H. Makino, N. Yamamoto and T. Yamamoto, Appl. Phys. Lett., 91, 051915 (2007).

[211:00am] **C3-10 Optical and Photoluminescence Studies of Gold Nanoparticles - Embedded ZnO Thin Films**, *A. Patra,* ***V. Damodara Das***, *S. Kasiviswanathan (kasi@iitm.ac.in)*, Indian Institute of Technology Madras, India

Metal oxides find application in variety of fields like photo-catalysis, gas sensing and corrosion protection. Some of the metal oxides like ZnO exhibit two diametrically opposite characteristics viz., high optical transparency in the visible region and high conductivity. On the other hand, exotic properties exhibited by physical systems of small sizes and dimension have attracted much attention. Au nanoparticles (AuNP) for instance exhibit surface plasmon resonance enhanced light absorption, which leads to local heating. Thus AuNP can act as potential nanoheaters in photothermal therapy and it is of interest tune the resonant frequency at around to that of commercial He-Ne lasers. We report herein, the synthesis of AuNP embedded ZnO films by sandwiching a thermally evaporated Au film

between sputtered two ZnO layers The films have been characterized using high resolution transmission electron microscopy (HRTEM), glancing angle X-ray diffraction (GXRD), optical absorpti on and photoluminescence (PL). The peaks in GXRD patterns were identified to be due to the reflections from various planes of ZnO and elemental Au. Formation of AuNP within ZnO matrix has been confirmed by HRTEM images. Pure ZnO film have exhibited a prominent PL peak in the UV region, whereas AuNP embedded ZnO film showed a peak of reduced intensity. This has been attributed to the absorption of the emitted radiation by the AuNP. Further, the size dependence of the plasmon absorption has been studied by forming nanoparticles of various sizes. The resonant frequency reported so far in sandwiched films is low. The UV-VIS-NIR spectrum of the samples has exhibited strong peaks at 608, 638, and 676 nm for AuNP of average sizes 27, 40 and 67 nm respectively. Thus, tuning the resonance peak to He-Ne laser wavelength is possible by varying the particle size. Moreover, antibodies and other biological molecules can be readily attached to the surface of AuNP. Also, absorption cross-sec tion and light scattering by AuNP is several orders higher than that of conventional absorbing dyes and strongly fluorescing dyes respectively. These properties of AuNP have been exploited in photothermal therapeutic applications. We have calculated theoretically the rise in temperature at the AuNP site due to heat generation as a function of incident light intensity. The rise in temperature has been estimated to be 15 K for a particle size of 80 nm.

11:20am **C3-11 Anisotropic Optical Properties of Sculptured Thin Films Grown by Glancing Angle Deposition**, ***D. Schmidt*** *(Daniel Schmidt [schmidt@huskers.unl.edu]),* University of Nebraska-Lincoln, *B. Booso,* University of Dayton, *T. Hofmann,* University of Nebraska-Lincoln, *A. Sarangan,* University of Dayton, *E. Schubert, M. Schubert*, University of Nebraska-Lincoln

Three-dimensional (3D) structure design on the nanoscale is in the focus of modern material science and engineering because intriguing applications are foreseen for such nanostructured films in various fields ranging from optics, electromechanics or electromagnetics. We utilize glancing angle physical vapor deposition, which allows for "bottom-up" fabrication of three-dimensional nanostructures arranged in sculptured thin films (STFs). Geometric shadowing and varying particle incidence azimuth is exploited to grow nanostructures with different 3D morphologies such as (slanted) columns, screws or spirals. We will present the anisotropic (structure-related) optical properties of STFs from various metals grown on silicon substrates by electron-beam evaporation at an oblique angle of incidence. Generalized spectroscopic ellipsometry is employed to determine the anisotropic optical constants of the thin films in the spectral range from 400 nm to 1000 nm. All nanostructured thin films show strong form birefringence and large dichroism and differ drastically from their bulk material. Columnar thin films from metal are found to be monoclinic[1,2,3].

[1]D. Schmidt, B. Booso, T. Hofmann, A. Sarangan, E. Schubert, and M. Schubert, Appl. Phys. Lett. 94, 011914 (2009).

[2]D. Schmidt, B. Booso, T. Hofmann, A. Sarangan, E. Schubert, and M. Schubert, Opt. Lett. (submitted, 2009).

[3]D. Schmidt, T. Hofmann, A. C. Kjerstad, M. Schubert, and E. Schubert, Mat. Res. Soc. Symp. Proc. (Fall 2008).

Tribology and Mechanical Behavior of Coatings and Thin Films
Room: California - Session E3-2

Tribology of Nanostructured and Amorphous Films
Moderator: J. Fontaine, Ecole Centrale de Lyon, T.W. Scharf, The University of North Texas

8:00am **E3-2-1 Characterization and Modeling of Self-Lubrication in Nanocrystalline Nickel**, ***C.C. Battaile*** *(ccbatta@sandia.gov), S. Prasad, J.R. Michael*, Sandia National Laboratories

Wear experiments on bare, single-crystal Ni films indicate that a thin (approximately 250 nm) layer of nanocrystalline Ni can form at the wear interface, and that this layer serves to lubricate the contact. Furthermore, the phenomenon is qualitatively sensitive to the crystallographic orientation of the frictional loading. For example, when a 1 N normal load and 3.75 mm/s tangential speed are applied to a 1/8" diameter Si_3N_4 ball in contact with electropolished single-crystal Ni, the measured friction coefficient is usually in the range 0.6 to 0.8. However, when the Ni surface is of the {110} type and the sliding direction is <211>, the friction coefficient gradually decreases from 0.6 to 0.5 during the first 400 cycles, at which point a sustainable nanocrystalline film forms and the friction coefficient abruptly drops to 0.3, where it remains indefinitely. Modeling of the wear behavior, based on crystal plasticity, microstructure formation, and grain bounda ry sliding, suggests that this self-lubrication phenomenon is due to the capacity of ultra-fine-grained microstructures to support grain rotation. Friction experiments on bulk nanocrystalline Ni deposits confirm this hypothesis by demonstrating low friction coefficients (around 0.3) and virtually no wear-in under low loads and sliding speeds, and higher friction (around 0.6) under high loads and speeds. This presentation will provide an overview of the experiments and modeling of nanocrystalline film formation on single-crystal Ni, detail the results from friction experiments on bulk nanocrystalline Ni, and discuss model validation of the phenomenon's strain rate sensitivity.

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.94AL85000.

8:20am **E3-2-2 Nanocomposite Nickel Coatings with Silicon Carbide Reinforcement: Friction and Wear Behaviour at 298 and 493 K**, ***M. Shafiei*** *(shafieim@uwindsor.ca), A.T. Alpas*, University of Windsor, Canada

Friction and wear behaviour of electrodeposited nanocrystalline (nc) Ni coatings reinforced with micro- and nano-particles of SiC were investigated at 298 K and 493 K to determine whether high temperature wear resistance of nc Ni coatings could be improved. Composite coatings were produced using 30 g/l SiC particles of either 5 μm or 50 nm in diameter. At 298 K, pin-on-disc tests showed that the coating reinforced with micro-particles had higher wear rate and coefficient of friction (COF) than the unreinforced nc Ni film, due to the abrasive effect of the micro-particles. However, the wear rate and COF of the coating reinforced with nano-particles were comparable to those of the unreinforced nc Ni film. Sliding tests at 493 K resulted in two orders of magnitude increase in the wear rate of the nc Ni. Compared to micro-particles, nano-particles were more effective in reducing high-temperature wear rate and COF of nc Ni. The subsurface microstructures were investigated b y cross-sectional focussed ion beam (FIB) and transmission electron microscopy (TEM). The wear mechanisms responsible for high temperature wear of the composites were delineated by paying particular attention to the role that nano-particles played in improving high temperature wear resistance.

8:40am **E3-2-3 Nanoscale Deformation Mechanism of Nanocomposite Thin Films**, ***J. De Hosson*** *(j.t.m.de.hosson@rug.nl), C. Chen, Y. Pei, K. Shaha*, University of Groningen, Netherlands **INVITED**

This paper concentrates on the deformation behavior of amorphous diamond-like carbon (DLC) composite materials. Combined nanoindentation and ex situ cross-sectional transmission electron microscopy (XREM) investigations are carried our on TiC/a-C nanocomposite films, with and without multilayered structures deposited by pulse DC magnetron sputtering. It is shown that rearrangement of nanocrystallites and displacement of a-C matrix occur at length scales from tens of nanometer down to 1 nm. At submicrometer scale homogeneous nucleation of multiple shear bands has been observed within the nanocomposites. The multilayered structure in the TiC/a-C nanocomposite film contributes to an enhanced toughness.

9:20am **E3-2-5 Tribological Investigation of New Low Wear Coating Systems on Ti6Al4V**, ***K.J. Kubiak*** *(K.Kubiak@leeds.ac.uk),* Ecole Centrale de Lyon, France and University of Leeds, United Kingdom, *T.G. Mathia,* Ecole Centrale de Lyon, France, *B.G. Wendler, W. Pawlak*, Technical University of Lodz, Poland

Titanium alloys are attractive constructional materials due to low density, high strength and high electrochemical corrosion resistance. On the other hand poor tribological properties like high coefficient of friction and tendency to seizure during dry sliding against numerous metals and alloys, results in many limitations in use of titanium alloys in engineering applications. Attempts to overcome these disadvantages by hardening of surface and near-surface zone with use of interstitial C, N, or O atoms don't improve the tribological properties and in particularity the resistance against fretting wear. In order to significantly improve the tribological properties of Ti alloys a novel original multiplex hybrid treatment of Ti alloys has been developed at Technical university of Lodz. As an example Ti6Al4V substrate has been diffusion hardened with interstitial O or N atoms by glow discharge plasma in the atmosphere of $Ar+O_2$ or $Ar+N_2$, than the gradient TiC@s ub x@N_y intermediate coating has been deposited and as an external layer a thin (200nm) amorphous a-C hard coating has been deposited. The morphology, microstructure, chemical and phase composition, chemical bonds, microhardness and tribological properties during dry and boundary lubricated friction of the alloy after multiplex treatment have been investigated with use of SEM, EDS, XRD, XPS, Vickers diamond indenter, ball-on-plate and fretting wear tests. An important increase of hardness of

the near surface zone of the Ti6Al4V alloy has been achieved (from 350VHN to 1000 VHN). Also a good adhesion between the gradient TiC_xN_y coating and the Ti6Al4V substrate has been achieved. The proposed coating system can be considered as a solid lubricant. The multiplex treatment of the Ti6Al4V alloy and gradient coating is a promising way to decrease dry friction coefficient against high carbon bearing steel to a value ~0.15 and to increase the wear resistance of the T i alloy.

9:40am **E3-2-6 Wear of C/CrC Coating Against Alumina at Room Temperature**, ***Z. Zhou*** *(z.zhou@sheffield.ac.uk), I. Ross, L. Ma, W. Rainforth,* University of Sheffield, United Kingdom*, P.Eh. Hovsepian*, Sheffield Hallam University, United Kingdom

Nanoscale hydrogen free C/CrC coatings have been produced by unbalanced magnetron sputtering of graphite and Cr metal targets in Ar gas. The coating possesses a nanocomposite structure with amorphous carbon embedded in a CrC matrix. The nano-scale amorphous carbon clusters self-assemble into layers alternated by CrC, giving a multilayer appearance. However, very little research has been conducted on its wear performance. Reciprocating wear was performed using a ball-on-disc apparatus at room temperature. The coating showed friction coefficient of 0.16 against alumina. Worn surface microstructure was characterised using scanning electron microscopy, transmission electron microscopy and Raman spectroscopy. A large number of roll-shaped wear debris was present on the surface with evidence of wear induced graphite, which was determined by Raman spectroscopy. Cross sectional microstructure of the rolls were revealed using focused ion beam extracted TEM specimens, and correla ted with the Raman spectroscopy. A tentative mechanism of the origin and evolution of the wear debris is proposed.

10:00am **E3-2-7 Constitution, Microstructure, and Tribological Properties of Nanocrystalline Reactive Magnetron Sputtered V-Al-C-N Hard Coatings**, ***C. Ziebert*** *(carlos.ziebert@imf.fzk.de), M. Stueber, H. Leiste, S. Ulrich*, Forschungszentrum Karlsruhe, Germany

The design of carbon-based nanostructured composite coatings being composed of nanocrystalline metastable hard phases such as fcc (Ti,Al)(C,N) homogeneously dispersed in an amorphous carbon matrix or covered by an amorphous carbon grain boundary phase is an emerging new approach for the development of advanced protective coatings. Nanocrystalline V-AI-C-N hard coatings were deposited by reactive r.f.-magnetron sputtering in an Ar/CH_4 plasma. In order to design and deposit different coating microstructures, ranging from metastable solid solutions to multi-phase nanocomposites, a combinatorial materials science approach was applied. In each experiment, six coatings of different composition and/or microstructure were obtained simultaneously by placing six substrates in individual positions relative to a segmented target, composed of ceramic VC and AlN half plates. The CH_4 flow rate was systematically varied up to CH_4 volume fractions of 8 % in the process gas. The chemical composition of the coatings was determined by electron microprobe analysis and the crystal- and microstructure of the films were characterized by X-ray diffraction, scanning and transmission electron microscopy and Raman spectroscopy. The surface topography has been investigated by atomic force microscopy and the correlation with the mechanical and the tribological properties of the coatings was studied by nanoindentation and ball-on-disk tribometer tests against 100Cr6 steel balls. Significant changes in the coatings topography, microstructure and in the related mechanical and tribological properties were observed both as a function of the sample position and of the carbon content. In particular, the successful variation of the hardness (15-35 GPa), the reduced elastic modulus (120-600 GPa) and the friction coefficient (0.15-0.45) on a wide range was achieved and correlated with constitution, microstructure and effective wear mechanisms.

10:20am **E3-2-8 Surface Morphology and Tribological Properties of Multilayer TiAlN Coatings Deposited by Reactive Magnetron Sputtering**, ***M. Wang*** *(wangmei64@yahoo.com), T. Toihara, M. Sakurai,* OSG Corporation, Japan*, W. Kurosaka, S. Miyake*, Nippon Institute of Technology, Japan

In this work, TiAlN monolayer and TiAlN multilayer coatings were deposited by reactive unbalanced magnetron sputtering. The surface morphology and tribological properties of the TiAlN monolayer and TiAlN multilayer coatings were characterized by atomic force microscopy (AFM) and high-frequency linear-oscillation (SRV) friction experiments. It was found that the TiAlN multilayer coatings exhibited a smaller grain diameter and a better surface roughness than the TiAlN monolayer coating owing to the suppression of the TiAlN grain growth in the multilayer. With increasing the rotation of the sample holder, the grain diameter and surface roughness of the TiAlN multilayer decreased. As a result, the SRV test results show that the wear resistance and frictional coefficient of the TiAlN multilayer coatings are better than those of the TiAlN monolayer coating deposited under similar conditions.

10:40am **E3-2-10 CrN-Ag Nanocomposite Coatings: High Temperature Tribological Properties**, ***C.P. Mulligan*** *(c.mulligan@us.army.mil),* Benet Laboratories, U.S. Army ARDEC*, T.A. Blanchet, D. Gall*, Rensselaer Polytechnic Institute

5-µm-thick CrN-Ag composite layers with 22 at.% Ag were deposited by reactive magnetron co-sputtering on Si(001) and 304 stainless steel substrates at growth temperatures T_s = 500, 600, and 700°C. The composite microstructures consist of a CrN matrix containing Ag segregates with an average size that increases from <25 nm grains at T_s = 500°C to lamellar grains, 100 and 200 nm thick and 200 and 300 nm wide for T_s = 600 and 700°C, respectively. Friction and wear are measured in air at T_t = 25-700°C, using ball-on-disk tests against alumina. The tribological tests indicate various temperature-friction regimes: At T_t < 300°C, the microstructure with finely dispersed Ag lamellae yields the highest solid lubricant surface coverage and, in turn, the lowest friction and wear. At 300°C < T_t < 500°C, the enhanced Ag mobility within the composite causes the transient friction coeffic ients µ to drop to 0.05-0.10 for the finest grained composite coating with T_s = 500°C, compared to µ = 0.25-0.30 for pure CrN and @mus@ = 0.3-0.4 for the coarser grained composites. The variation in µ for the fine grained vs. coarse grained composites is attributed to the decreasing Ag mobility and lubricant transport as Ag segregate size increases. At $T_t \geq$ 600°C, µ increases to 0.3 for the finest grained composite and 0.5-0.6 for pure CrN and the coarser grained composites. The increases at high temperature are attributed to oxidative degradation that is facilitated by higher porosity for $T_s \geq$ 600°C. These results suggest that both the residual porosity and the as-deposited Ag segregate size determine diffusive transport and oxidation rates and, in turn, control the high-temperature tribological performance of this adaptive self-lubricating nanocomposite coating system.

Applications, Manufacturing, and Equipment
Room: Sunrise - Session G2-2

Coatings for Automotive and Aerospace Applications
Moderator: R. Mertens, Oerlikon Balzers AG, G. Dadheech, General Motors

8:00am **G2-2-1 TiN Multilayer Systems for Compressor Airfoil Sand Erosion Protection**, ***A. Feuerstein*** *(Albert_Feuerstein@praxair.com), A. Kleyman*, Praxair Surface Technologies, Inc.

Frequently, aircraft, tank and helicopter gas turbine engines are operated in a desert environment where the gas turbine compressor rotor blades and vanes are exposed to erosive media such as sand and dust. These erosion effects lead to increased fuel consumption, efficiency loss, and can cause damage to compressor and turbine hardware. Erosion resistant coatings such as TiN, TiCN, TiZrN, TiZrCN, TiAlN and TiAlCN, applied by cathodic arc physical vapor deposition or other PVD processes, can be used to prolong the life of compressor airfoils in a sand erosion environment. Praxair Surface Technologies, Inc. has developed unique multilayered TiN coating systems with optimized erosion resistance compared to conventional mono block layers. The multilayer structure can be tailored to the respective erosion media particle size distribution. The key features of two selected coating architectures are outlined. Selected erosion performance data with different erosion media are presented. The aspects of high quality mass production are addressed.

8:20am **G2-2-2 Lubricated PVD Coatings for Automotive Applications**, *K. Bobzin, N. Bagcivan, N. Goebbels,* ***K. Yilmaz*** *(yilmaz@iot.rwth-aachen.de),* RWTH Aachen University, Germany*, B.-R. Hoehn, K. Michaelis, M. Hochmann*, University of Munich, Germany

The demand on a better efficiency and higher performance under environment friendly aspects is an actual trend for automotive applications and machine components. This demand can be achieved by vacuum coatings such as DLC (Diamond like Carbon), which offer excellent results in e.g. fuel injection systems, piston pins, cam follower, gears and bearings. At the beginning the focus of DLC coatings was mainly on dry lubrication capability. Nowadays this kind of coating become very interesting for lubricated tribological contacts, because of their excellent tribological performance under boundary lubrication, which provides low friction losses and high wear resistance. In addition to DLC, the CrAlN coatings promise also performance increase especially for the lubricated tribological contacts, where high ductility and wear resistance is needed. Surface properties of DLC and CrAlN coatings are not analogous to uncoated steel surfaces; thereby their wettability with lubricants is different than conventional steel surfaces. Hence finding an optimal PVD coating/lubricant pair is still a challenge with regard to improvement of system performance and efficiency. In this research the wettability of the PVD coatings with

different lubricants and the effect of the wettability on tribological behavior are investigated. The PVD coatings are WC/C and CrAlN, the lubricants are Polyalphaolefin, Polyglycol, synthetic ester and mineral oil, which are mixed with additives ZnDTP, MoDTP and S-P. The wettability of the PVD coatings with lubricants was determined by means of spreading coefficient and adhesion energy. For the tribological tests two different types of Tribometer, Pin on Disk and Twin Disk Tribometer are used. The Pin on Disk Tribometer delivers elementary information about the tribological performance of the PVD coatings with lubricants, whereas the twin disk Tribometer simulates the different working points of PVD coated gears for lubricated applications. By measuring friction coefficients, the tribological behavior of PVD coatings with the lubricants for different working points, such as different Hertzian pressure, slip velocity and lubricant temperature, are determined. The wear rates of the PVD coatings are measured and the tribological results are compared with the wettability. It is found that the wettability of the PVD coated surface can be optimized by means of adapting the adhesion energy, which in return leads to optimized friction behavior.

8:40am **G2-2-3 Multiphase Wear and Erosion Resistant Coatings for Aerospace Applications**, ***J. Nainaparampil*** *(jnain@ues.com), A.K. Rai, R. Bhattacharya*, UES Inc.

Sand, pollutants, and moisture may all affect the smooth functioning of a Gas Turbine (GT) engine. Since present day designs look for increased efficiency, lightweight and high temperature operation the choice of materials systems is limited. Since Ti, Ni and their alloys fulfill most of the requirements, critical engine parts are made of these alloys. Protective coatings are needed to enhance the erosion resistance of these components. A tough, super adherent, hard and chemically stable protective coating with high temperature resistance can preserve the design tolerances and surface finish. In this work, a nano-structured TiN based composite thin film with varied architecture is deposited with direct arc vacuum evaporation and used as a protective coating for Ti6Al4V and Inconel 718 substrates. The mechanism used to attain the protection is the manipulation of hardness to elastic modulus ratio, which in turn determines the resistance to high-speed impact erosion[1]. This can be achieved by incorporating a hard ceramic phase with an amorphous or ductile phase of another soft material. Hard and tough films have been developed by numerous researchers in the past mainly by mixing two types of material systems. A) nc-MeN/a-nitride, boride (e.g. a-Si_3N_4, a-TiBN, etc.) b) nc-MeN/metal (e.g. Cu, Ni, Y, Ag, Co, etc.) where Me stands for any transition metal, and 'a' stands for amorphous. The ductile/amorphous component forms the separation medium between the ceramic hard phases.. Manipulation of toughness can be achieved by proper selection of the ductile/amorphous phase and overall microstructure of the films. High temperature, high speed erosion data coupled with toughness and hardness values of these films will be presented. Results from Tribological characteristics and Microstructure study will also be presented. An attempt is made to correlate the results with existing models of toughening mechanisms.

[1]S. Veprek, P. Nesla´dek , A. Niederhofer , F. Glatza, M. Jılek, M. Sıma, Surface and Coatings Technology 108–109 (1998) 138.

9:00am **G2-2-4 Investigations on the Interfacial Strength of Chromium Adhesion Layers in DLC Coating Systems for High Load Applications**, ***J. Schaufler*** *(jens.schaufler@ww.uni-erlangen.de), K. Durst,* University Erlangen Nürnberg,, Germany*, R. Mertens,* Oerlikon Balzers AG, Liechtenstein*, M. Göken*, University Erlangen Nürnberg, Germany

DLC coatings play an important role in many high load applications. Their unique properties such as hardness, low friction coefficient and chemical inertness make them suitable for such applications. During service, the interfacial strength between the coating and the substrate is of a great importance. A chromium - chromiumcarbide adhesion layer is frequently used to enhance the adhesion between the coating and the steel substrate. However, the structure and quality of these adhesion layers mainly depend on the conditions and parameters of the deposition process. The interfacial strength and quality is usually determined by a Rockwell-C indentation test, using the cracking patterns to qualify the interfacial strength. In this work a detailed structural characterization of various adhesion layers with different interfacial strength, ranging from poor to very good has been conducted. This characterization includes TEM investigations of the microstructure in combination w ith EDX analysis. The internal stresses in the coating systems were measured with a cantilever deflection method, using a focused ion beam workstation. The residual damage zone around the indentation is analyzed in FIB cross sections and with a TEM. There it is found, that the analyzed weak coating system mainly fail in the chromiumcarbide zone, whereas the adhesion layer with the strong interfacial strength shows a great stability against internal cracking. By analyzing the failure mechanism a crosslink between the microstructure, the internal stress and the interfacial strength of the coating systems can be determined.

9:20am **G2-2-5 Innovative Surface Technologies for Advanced Automotive Applications: From Super-Hard and –Low Friction Coatings to Super-Fast Surface Treatments**, ***A. Erdemir*** *(erdemir@anl.gov), O.L. Eryilmaz, G. Kartal,* Argonne National Laboratory*, K. Kazmanli, S. Timur, M. Urgen*, Istanbul Technical University, Turkey **INVITED**

During last decade or so, there has been an overwhelming interest in the development and diverse utilization of super-hard and –low friction coatings for a wide range of automotive applications. Within the same period, great strides have been made in deposition technologies by which these multifunctional coatings are produced. With these advances, it is now possible to produce nano-composite and/or –layered coatings that can meet the ever increasing property and performance requirements of advanced automotive applications. In this paper, we will concentrate primarily on the design and development of super-hard and low-friction nano-composite coatings that can make a huge positive impact on the fuel efficiency and durability of advanced engine systems that are subjected to increasingly more severe applications conditions than before. Specifically, we will introduce a crystal-chemical model that can help identify the kinds of coating ingredients that are needed in suc h nano-composite architectures for achieving ultra-low friction and wear on lubricated surfaces. Recent results from bench-top and fired engine tests will be presented in support of much superior tribological properties for these designer coatings over a broad range of sliding conditions. Surface treatments (like, nitriding, carburizing, and boriding) are used extensively by industry in all types of engine components despite being very time and energy consuming. In this presentation, we will also present very briefly a super-fast surface treatment process which can complement or displace some or all of the existing surface treatment processes. Initial results from selected engine parts and their tribological properties will be presented and the future outlook for both the super-hard coatings and super-fast surface treatment technologies will be provided.

10:00am **G2-2-7 Sliding Wear Behavior of CrC and TiCrN Coatings Produced by Large Area Filtered Arc Deposition**, ***C. Bowman*** *(cbowman@arcomac.com), V.I. Gorokhovsky*, Arcomac Surface Engineering, LLC

Advanced aircraft and automotive design continues to produce new tribological challenges for bearings and gears. The desire for greater load capacities, higher operating temperatures, better corrosion performance, and better component lifetime all must be addressed in a unified engineering approach. Solutions to multifunctional design requirements of this nature are particularly suited to physical vapor deposition (PVD) coating surface engineering methods. The Large Area Filtered Arc Deposition (LAFAD) process has demonstrated promising coatings and high deposition rates over large surface areas. In addition to the inherent advantages of conventional filtered arc technology (high hardness, improved adhesion, low density of defects), the LAFAD technology allows functionally graded, multilayer, and nanocomposite architectures of multi-elemental coatings via electro-magnetic mixing of two plasma flows composed of different metal vapor ion composition. In the present study, chromium contained binary Cr-C and ternary Ti-Cr-N coating systems were deposited on M50 bearing steel and Pyrowear 675 carburized steel and characterized in sliding wear. Ball-on-disc testing results are presented over a range of loads and sliding distances for baseline metal to metal contact and combinations of coating to metal contacts. In addition wear testing results are presented for coating to coating contacts which are relevant for gearing applications where corrosion protection is necessary for both wearing surfaces. The observed wear mechanisms are discussed along with a brief overview of parallel design requirements for coating fatigue life and corrosion protection.

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Claus Rebholz – Symposia C & H
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Mechanical & Manufacturing Engineering
75 Kallipoleos
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+357-22-89-2282, Fax: +357-22-89-2254
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Hans-Joachim Scheibe – Symposia A & D
Fraunhofer IWS Dresden
Winterbergstrasse 28
Dresden, D-01277, Germany
+49 351 83391-3455, Fax: +49 351 83391-3314
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+49-7247-82-3889, Fax: +49-7247-82-4567
michael.stueber@kit.edu

PROGRAM CHAIRMAN
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