

Tuesday Morning, April 27, 2021

Live Session

Room Live - Session LI-TuM1

Surface Engineering - Applied Research and Industrial Applications Live Session

Moderators: Dr. Satish Dixit, Plasma Technology Inc., USA, Dr. Christoph Schiffers, CemeCon AG, Germany

10:00am **LI-TuM1-1 Award Chair's Welcome Address, Introductions, & Thank You to Sponsors, Ivan G. Petrov (petrov@illinois.edu)**, University of Illinois at Urbana-Champaign, USA

We hope you will enjoy the presentations from our three student award finalists and our invited speakers!

10:15am **LI-TuM1-2 Student Award Finalist Talk: Enhanced High-temperature Oxidation Resistance of Hard TiB₂-rich Ti_{1-x}Al_xB_y Thin Films, Babak Bakht (babak.bakht@liu.se)¹**, Linköping University, Sweden; I. Petrov, J. Greene, University of Illinois, USA, Linköping University, Sweden, USA; L. Hultman, J. Rosen, G. Greczynski, Linköping University, Sweden

Ultra-high-temperature refractory transition-metal (TM) diborides are considered as promising candidates for extreme environments. However, they typically exhibit insufficient high oxidation resistance required for harsh environmental conditions. Here, we study the effect of Al addition on the high-temperature oxidation resistance of TiB₂-rich Ti_{0.68}Al_{0.32}B_{1.35} thin films. The films, grown by hybrid high-power impulse and dc magnetron co-sputtering (Al-HiPIMS/TiB₂-DCMS) in pure Ar atmosphere at ~475 °C, exhibit hexagonal columnar nanostructure. While the column boundaries of TiB_{2.4} layers grown by DCMS are B-rich, the Ti_{0.68}Al_{0.32}B_{1.35} alloys consist of Ti-rich columns surrounded by an Al-rich Ti_{1-x}Al_xB_y tissue phase which is highly B deficient. The observed transition in the nanostructure is attributed to the lower formation enthalpy of AlB₂ than TiB₂ together with enhanced atomic mobility caused by intense Al⁺ ion bombardment during HiPIMS pulses. TiB_{2.4} films readily oxidize at temperatures above ~300 °C, as evidenced by X-ray photoelectron spectroscopy, with oxidation products consisting of a tetragonal rutile-TiO₂ structure filled with an amorphous BO_x phase. Air-annealing at 700 °C for 1 h results in the formation of a thick double-layer oxide scale on TiB_{2.4}, ~510 nm, where the outer layer is composed of sub-micrometer crystallites and the inner layer has a porous and V-shape columnar structure. Compared to TiB_{2.4}, Ti_{0.68}Al_{0.32}B_{1.35} alloys show significantly higher oxidation resistance. While air-annealing at 800 °C for 0.5 h results in the formation of an ~1900-nm oxide scale on TiB_{2.4}, the thickness of the scale formed on the Ti_{0.68}Al_{0.32}B_{1.35} alloys is ~470 nm. The enhanced oxidation resistance is attributed to the formation of a dense, protective Al-containing oxide scale which considerably decreases the oxygen diffusion rate by suppressing the oxide-crystallites coarsening.

10:30am **LI-TuM1-3 Student Award Finalist Talk: The Magneto-Plasmonic Properties of Ag-Co Composite Nanostructures, Hoang Luong (hoanglm@uga.edu)²**, T. Nguyen, Y. Zhao, University of Georgia, USA

Multifunctional materials and structures with both plasmonic and magnetic properties have been receiving substantial attentions. In this work, we investigated magneto-plasmonic properties of well-identified plasmonic nano-lattices based on Ag-Co composite materials. By combining shadowing nanosphere lithography and electron beam co-deposition, composite nanotriangle and nanohole arrays with different composition ratio of Ag and Co were fabricated. The composition-dependent optical transmission, polar magneto-optics Kerr effect, Faraday rotation (FR), and Faraday ellipticity of these structures in the visible to near infrared wavelength region were studied. Finite-difference time domain calculations were performed to confirm the experimental results and to give an insight on the relationship between magneto-plasmonic properties of nanostructures and their compositions. In particular, the Ag-Co composite nanohole array showed several FR peaks at the plasmonic-related wavelength positions, *i.e.*, where the Bloch wave condition and Wood – Rayleigh anomaly condition are satisfied. With the Co content of 30%, the composite nanohole array exhibited a significant enhancement of plasmonic - magneto-optics behavior compared to that of Ti-Co composite nanohole array. On the other hand, the enhanced magneto-optic effect of the Ag-Co composite nanotriangle array was observed to be coincident with the localized surface plasmon resonance (LSPR), *i.e.*, the maximum FR effect occurred at the LSPR wavelength, which is due to high local E-field. Thus, the magneto-optics response of these magneto-plasmonic systems behave differently, depending on the nature of the plasmon resonance that

the system supports (*i.e.* propagating surface plasmon wave in the nanohole array or localized surface plasmon wave in the nanotriangle array), and by adjusting the Co content, their magneto-plasmonic performances can be maximized. Such a magneto-plasmonic composite provides a new way on the design and application of magneto-plasmonic materials and devices.

10:45am **LI-TuM1-4 Student Award Finalist Talk: Nanoscale Stress and Microstructure Distributions across Scratch Track Cross-Sections in a Brittle-Ductile CrN-Cr Bilayer Film on Steel Revealed by X-ray Nanodiffraction, Michael Meindlhuber (Michael.Meindlhuber@oeaw.ac.at)³**, Montanuniversität Leoben, Austria;

J. Todt, J. Zalesak, Austrian Academy of Sciences, Austria; M. Rosenthal, ESRF, Grenoble, France; H. Hruby, eifeler-Vacotec GmbH, Germany; C. Mitterer, R. Daniel, J. Keckes, Montanuniversität Leoben, Austria

Nanocrystalline hard films benefit from a combination of extraordinary multifunctional properties, such as high hardness, elastic modulus, thermal stability and wear resistance. Although scratch tests are routinely used to obtain qualitative data on adhesion and abrasion of thin films, it is not trivial to correlate the scratch-test-response of the films with their composition, microstructure and residual stress state. In order to relate the microstructure and residual stress state to elastic-plastic deformation of a CrN/Cr film induced during a scratch test, cross-sectional X-ray nanodiffraction (CSnanoXRD) in transmission geometry and a beam size of 50 nm was applied. The experiment focused on the characterization of a brittle/ductile CrN/Cr bilayer thin film consisting of 1.2 and 2 μm thick CrN and Cr layers, respectively, deposited on a high-speed-steel substrate and loaded at 200 and 400 mN by a diamond sphero-conical indenter with a radius of 5 μm. In order to assess the microstructure variations and stress distributions in the deformed volume, cross-sections of the scratch traces were extracted and subsequently ex-situ probed by CSnanoXRD at the ID13 beamline of the European Synchrotron Radiation Facility in Grenoble, France. Crack patterns in individual layers were characterized by small-angle X-ray scanning microscopy and revealed crack formation in the CrN layer predominantly at the load of 400 mN, which corresponds to the complementary scanning and transmission electron microscopy data. The results further revealed a gradual increase of the compressive stress from ~-3 to -4 GPa from the interface towards the surface of the CrN toplayer and a rather constant stress state of ~-1 GPa within the Cr sublayer in the as-deposited state. On the contrary, complex variations of in-plane stress in the deformed volume were observed in the CrN toplayer reaching magnitudes up to -6 GPa in the near CrN/Cr interface region. The film volume next to the groove of the residual imprint close to the film surface was almost stress free, indicating full stress relaxation of the deformed zone. Within the Cr sublayer, tensile in-plane stress of ~0.5 GPa near the CrN/Cr interface and compressive stress of ~-1.5 GPa near the film/substrate interface were detected. Further insights into the deformation behaviour of the bilayer system during scratching were gained by correlating the experimental results with a finite-element model. In summary, the experiments revealed that the ductile Cr sublayer served as a stabilizing component for the CrN/Cr bilayer structure upon mechanical loading, effectively suppressing catastrophic failure of the otherwise brittle CrN.

11:15am **LI-TuM1-6 Silicon in Cutting Tools, Albir Layyous (alabyrinth@netvision.net.il)**, Layyous Consulting, Israel; L. Qiu, Central South University, China

INVITED
Silicon have been used widely in cutting tools, started as Silicon Nitride base, followed by SiC whiskers reinforcement of Al₂O₃, Silicon alloying of PVD and CVD coatings, has been proved to enhance the machining performance of TiAlN base coatings, furthermore Silicon Carbide thin layers were coated successfully as wear resistance or interlayer for diamond coating. Recently CVD TiSiN and TiSiCN coatings were prepared and investigated successfully in a commercial CVD hot-wall reactor, where hardness and oxidation resistance were increased. Importance of silicon usage in cutting tools was reviewed.

Key Words: Cutting Tool, Silicon Alloying, Physical Vapor Deposition, Chemical Vapor Deposition

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² Student Award Nominee

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11:45am LI-TuM1-8 **Stabilization of FCVAS Based Hybrid System for Deposition of Thick Tetrahedral Amorphous Carbon Films and its Applications**, *Jongkuk Kim (kjongk@kims.re.kr)*, Y. Jang, Korea Institute of Materials Science (KIMS), Korea, Republic of Korea; D. Kim, Y. Kang, Korea Institute of Materials Science (KIMS), Korea; J. Kim, Korea Institute of Materials Science (KIMS), Korea, Republic of Korea

INVITED

Diamond-like carbon (DLC) is used in various industrial applications such as automobile, mechanical machinery and optical lens, etc. due to their excellent physical, chemical and mechanical properties. In the case of automobile application, the coated films are required to have high thermal stability to prevent the film delamination during actual operating conditions.

However, many kinds of hard coatings (a-C, a-C:H) were very unstable under high temperature. During the operation, coated surface undergoes high mechanical, thermal and chemical stresses. Therefore, the surface quality degrades very quickly to an unacceptable level.

It is known that tetrahedral amorphous carbon (ta-C) is a hydrogen-free carbon coating with 70 ~ 80 % of sp³ phase, which results in smooth surface, good thermal resistance and wear resistance.

In particular, despite the high thermal stability and hardness, ta-C film deposited by vacuum arc method was difficult to be thickened due to high internal stress. Furthermore, it is hard to make thick coated layer as a carbon cathode became unstable when the coating process proceeds long time.

Our Research Group tried to improve the stability of the carbon cathodes for long-time coating process by controlling electric and magnetic fields. We have optimized the discharge stability so that the carbon arc target can be used stably at a discharge current of 160A for up to 24 hours.

The designed hybrid coating system consists of 1) Anode layer ion source (LIS) for the etching process, 2) Unbalanced magnetron sputter (UBM) for the deposition of interlayer, and 3) Filtered cathodic vacuum arc (FCVA) source for the deposition of ta-C film.

To adopt the designed hybrid coating process, the system was established with a single LIS, double UBMs, and eight FCVA with the maximum working area of 900 mm in diameter and 500 mm in height. For 5 μm coating of ta-C, the system can be operated for longer than 20 hours stably.

For the further application, we applied ta-C coating films on non-ferrous cutting tools (0.3 ~ 2 μm), piston rings (5 ~ 7 μm) of automotive engine parts, and semiconductor inspection probes with conductive ta-C (0.2 μm).

12:15pm LI-TuM1-10 **Low Interfacial Toughness Materials for Effective Large-scale Deicing**, *Kevin Golovin (kevin.golovin@ubc.ca)*, University of British Columbia, Canada

INVITED

Reducing the interfacial adhesion between ice and a surface could be beneficial to a wide range of commercial activities. Since the 1940s, the adhesion between ice and a surface has been defined by the force, F , required to de-bond an area of adhered ice, A , typically in shear. The shear ice adhesion strength is then defined as $\tau_{ice} = F/A$, and an increasing body of literature is available delineating the various strategies for minimizing τ_{ice} . In the first part of my talk, I will briefly discuss our efforts aimed at minimizing τ_{ice} , via mechanisms such as superhydrophobicity, interfacial cavitation, and engendering slip at the ice interface. In the second part of my part I will discuss why this definition of τ_{ice} contains an intractable scalability limit often ignored within the ice adhesion community – large areas of accreted ice will require extremely large forces to remove the ice. I then discuss our recent work understanding materials that circumvent this issue. Such materials, which exhibit Low Interfacial Toughness (LIT), offer the unique property that the force necessary to remove adhered ice becomes independent of the interfacial area – the force needed to remove a few square centimeters is the same as the force needed to remove a few square meters. We design LIT materials using a cohesive zone analysis of the ice-substrate interface mechanics. LIT materials are categorically dissimilar to traditional ice-phobic systems. For example, LIT materials become more effective with decreasing thickness and increasing shear modulus (the opposite is true for ice-phobic materials). These physical parameters make LIT systems particularly attractive for aerospace applications, which durability (requiring high modulus) and added weight (requiring low thickness) are major constraints.

12:45pm LI-TuM1-12 **Closing Remarks & Sponsor Thank You's!**, *Satish Dixit (dixsat@gmail.com)*, Plasma Technology, Inc., USA; C. Schiffers, CemeCon AG, Germany

Thank you for attending the session. Join us for the Post-Session Discussion from 1:00 - 2:30 pm EDT and be sure to return for tomorrow's Live Sessions at 10:00 am EDT.

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