Tribology Focus Topic
Room: 111 - Session TR-ThA

Advanced Tribological Materials
Moderator: S. Perry, University of Florida

2:00pm **TR-ThA1 Nanomechanics and Nanotribology of ZrB_2 Thin Films Deposited by DC Magnetron Sputtering**, ***E. Broitman***, *H. Högberg, L. Hultman*, Linköping University, Sweden

The microstructure, nanomechanical and nanotribological properties of ZrB_2 thin films grown by DC magnetron sputtering have been studied as a function of Ar pressure, substrate bias, and substrate temperature. Films, ~ 500 nm thick, were deposited onto Si (001) and Al_2O_3 (0001) substrates from a compound target using an industrial chamber CC-800/9 from CemeCon operated at a fixed target-to-substrate distance of 7 cm.

X-ray diffraction patterns show that 0001-oriented films can be obtained on both substrates at a substrate bias of -80 V without any external heating. Transmission electron microscopy of samples grown at different conditions reveal the presence of an amorphous 100-300 nm thick layer close to the substrate, followed by the nucleation of ZrB_2(0001). The same oriented structure appears for samples grown up to 150 ^oC, but at higher temperatures this orientation is gradually degraded. At 500 ^oC, cross-sectional scanning electron microscopy shows a columnar microstructure with re-nucleation during the growth. For films grown at 100 °C, little impact on the texture is observed when the substrate bias is changed from floating to -200 V.

Nanomechanical and nanotribological properties measured with a Hysitron Triboindenter^{TM} TI 950 reveal that the films have high hardness and elastic recovery, and low friction. For films grown at low temperature, the hardness, reduced elastic modulus, and elastic recovery decrease from 25 to 19 GPa, 290 to 200 GPa, and 96 to 92%, respectively, when the amorphous interface increases from 100 to 300 nm. Nano-frictional tests were done in a load-controlled feedback mode using a force of 1 mN; a total of 40 reciprocating passes were performed for each test using a diamond 90º probe with a 1 µm tip radius. The friction tests reveal a friction coefficient µ in the range 0.10-0.13 for ZrB_2 samples grown at different conditions, in contrast of µ = 0.6 for a pure Zr film.

2:20pm **TR-ThA2 Structural and Tribological Properties of CrN_{MPP}/TiN_{DCMS} Multilayer Coatings**, ***J. Paulitsch***, *C. Maringer, D. Holec, P.H. Mayrhofer*, Montanuniversität Leoben, Austria

Deposition processes like the high power impulse magnetron sputtering (HIPIMS) indicate high metal ion ratios in the plasma, which result in increased structural and mechanical properties. The generally low deposition rate, compared to direct current magnetron sputtering (DCMS), narrows the industrial application range of this technology. The modulated pulse power (MPP) deposition technique on the other hand uses multiple complex pulsing steps to increase the metal ion ratio in the plasma without dramatically reducing the deposition rates as compared to DCMS.

Recently we showed that a multilayer architecture of CrN and TiN, deposited using the hybrid HIPIMS/DCMS deposition technique, results in coatings exhibiting friction coefficients in the range of diamond-like-carbon (DLC) coatings when tested at RT and ambient air conditions. Here we show results of MPP/DCMS deposited CrN/TiN multilayer coatings indicating comparable mechanical and tribological properties, hardness values around 25 GPa and coefficient of friction below 0.05. Furthermore, investigations on their dependence to the atmospheric conditions used during dry sliding as well as theoretical investigations of the layered structure using density function theory simulations were carried out.

2:40pm **TR-ThA3 Tribological Properties of Plasma Electrolytic Oxidation (PEO) Coatings on an Aluminum A356 Alloy**, *J.F. Su*, ***X. Nie***, University of Windsor, Canada

To reduce the fuel consumption and pollution of passenger vehicles, the aluminium engines have been increasingly used throughout the last 30 years. Since most technical aluminium alloys provide only poor wear resistance, various technical solutions exist to generate a wear-resistant cylinder bore surface against the sliding piston ring. A Plasma Electrolytic Oxidation (PEO) process has been developed in our group to produce oxide coatings on an Al alloy A356 for Al engine block, to battle against the wear attack. Primary results showed that the PEO coatings, thinner than 8 µm, are promising candidates to resist wear at elevated temperature. In this work, further study was carried on a serial of PEO coatings on the A356 alloy. The surface morphology, coating thickness and tribological properties were tailored by adjusting the PEO process parameters. The tribological performance of the PEO coatings was better than a Plasma Transferred Wire Arc (PTWA) coating which is currently used for engine applications. Based on this study, selections on optimal thickness and morphology of PEO coatings for better wear resistance were proposed.

3:40pm **TR-ThA6 Tribochemical and Microstructural Evolution during Friction and Wear of Nanocomposite Coatings**, ***T. Scharf***, The University of North Texas **INVITED**

Friction and wear mitigation is typically accomplished by introducing a shear accommodating layer (e.g., a thin film of liquid) between surfaces in sliding and/or rolling contacts. When the operating conditions are beyond the liquid realm, such as in extreme environments, attention turns to solid coatings. The focus of this talk is how contacting surfaces and subsurfaces change both structurally and chemically in order to accommodate interfacial shear for two multifunctional coating systems: nanocomposite MoS_2/Sb_2O_3/Au and Ni/TiC/graphite. It was determined that the coatings exhibit velocity accommodation modes (VAM) of interfacial sliding and intrafilm shear, as determined by advanced electron microscopy (3-D focused ion beam serial cross-sectioning, HAADF-STEM, and HRTEM) and spectroscopy (Raman, Auger and EDS wear maps) techniques.

In the case of amorphous-based MoS_2/Sb_2O_3/Au nanocomposite sputtered coatings, the main mechanism responsible for low friction and wear in both dry and humid environments is governed by the interfacial sliding between the wear track and the friction-induced transfer film on the counterface ball. In dry environments, the nanocomposite has the same low friction coefficient as that of pure MoS_2 (~0.007). But unlike pure MoS_2 coatings which wear through in air (50% RH), the composite coatings showed minimal amount of wear with wear factors of ~1.2-1.4 x 10^{-7} mm^3/Nm in both dry nitrogen and air. Cross-sectional TEM of wear surfaces revealed that frictional contact resulted in amorphous to crystalline transformation in MoS_2 with 2H-basal (0002) planes aligned parallel to the sliding direction. In air, the wear surface and subsurface regions exhibited islands of Au. The mating transfer films were also comprised of (0002)-orientated basal planes of MoS_2 resulting in predominantly self-mated 'basal-on-basal' interfacial sliding, and thus low friction and wear.

In the case of laser deposited Ni/TiC/graphite composite coatings, it was determined during sliding that a wear-induced tribochemical and structural change from microcrystalline graphite to amorphous carbon/nanocrystalline graphite hybrid layer resulted in decreased friction and wear. Other novel insights were determined from 3-D microstructural evolution during wear, such as a mechanically mixed layer developed consisting of predominately refined nanocrystalline Ni grains (~10 nm grain size) and disordered carbon below this hybrid layer. The formation of these low interfacial shear strength films and recrystallized zones were responsible for intrafilm shear VAM to achieve low friction coefficients (~0.09) and wear factors (~6.8 x 10^{-7} mm^3/Nm).

4:20pm **TR-ThA8 Study of Failure Mechanisms of a PVD TiAlN Coating by an Impact-Sliding Tester**, ***J.F. Su***, *X. Nie*, University of Windsor, Canada

Physical vapor deposition (PVD) coatings usually have high hardness and wear resistance and have been considered as necessary top layers of a wide variety of mechanical components to battle the wear problems. One of applications of hard PVD coatings is used as much-needed protective top layers on surfaces of stamping dies thereby to extend the tool life and improve the quality of the stamped products. Impact fatigue tests have been carried out to investigate the failure behavior of coating-substrate systems under simulated stamping force conditions in our group. However, machining forces on the work pieces are not always only perpendicular to the surface. Tangential or parallel forces are usually involved such as in stamping, milling and turning. In this work, an impact-sliding fatigue tester was proposed as an experimental technique to investigate the failure behavior of coating-substrate systems under shear combined conditions. Each impact-sliding cycle consisted of an 200 N impact force and a 200 N pressing force, respectively. One PVD TiAlN coating on D2 substrates was tested at this combination of impact/pressing loads for 1,500 cycles. Impact-sliding wear track was then observed at cross section obtained by Electrical discharge machining (EDM). Coating failure mechanisms were found to be chipping, peeling, fatigue cracks and material transfer. Fatigue cracks were particularly discussed from the point of view of fracture toughness.

4:40pm **TR-ThA9 A Study of Sliding Friction Across Velocity Regimes for Alternative MEMS-type Interfaces using Atomic Force Microscopy and Combined Nanoindentation / Quartz Microbalance**, *N. Ansari*, Auburn University, *S. Barkley*, Luther College, *C. Bouxsein*, *M. Deram*, *N. Eigenfeld*, Saint Olaf College, *O. Matthews*, Luther College, *A. Poda*, *W.R. Ashurst*, Auburn University, *E.E. Flater*, Luther College, ***B.P. Borovsky***, Saint Olaf College

As mechanical devices have shrunk to microscopic sizes, the need for a more fundamental understanding of friction and other surface phenomena has become urgent. While the emerging technology of microelectromechanical systems (MEMS) shows promise as the mechanical counterpart to integrated circuits, progress remains slow as structural materials and lubricant strategies continue to be developed. We report on the results of a collaborative effort to study the frictional properties of organic monolayers deposited on metal oxide surfaces. These interfacial systems have the potential to offer an alternative to silicon-based device fabrication. Both a nanoindenter-quartz crystal microbalance (NI-QCM) as well as an atomic force microscope (AFM) in lateral force mode have been used to perform tribological experiments at sliding velocities spanning the range from microns per second to meters per second. Our studies have investigated two different self-assembled monolayers chemisorbed onto aluminum oxide surfaces with realistic contact roughnesses and sizes: octadecylphosphonic acid (ODP) and octadecyltrichlorosilane (OTS). Both monolayers are observed to exhibit substantially reduced friction as compared to the bare interface, at both low as well as high sliding speeds. However, the films appear to fail upon exceeding a threshold contact pressure. We compare the tribological responses of the bare and monolayer coated interfaces of different systems and discuss insights into the molecular-level mechanisms responsible for the observed behaviors.

5:00pm **TR-ThA10 Nanotribological Characterization of Percolating Lead Films Above and Below T_c**, ***K. Stevens***, *J. Krim*, North Carolina State University

Friction at the nanoscale shows a strong and complex relationship to surface roughness and atomic disorder [1]. Recent research in superconductivity dependent friction [2-5], along with reports that quantum size effects [6] can influence diffusion (and thus friction) of adsorbed layers, has motivated our investigation. In particular, we have performed friction measurements of adsorbed nitrogen and helium films sliding on nanostructured lead films substrates that have been deposited on titanium, a substrate that lead does not wet. Varying the lead coverage results in a spectrum of percolated morphologies. We prepare these films on a quartz crystal microbalance (QCM) and probe their topologies by means of adsorption onto the surface [7].

Measurements have been recorded on nanoclustered lead films with coverages crossing the critical concentration for percolation. We study the substrate in the superconducting and normal states, which allows us to isolate and quantify the contribution of electronic and phononic dissipation to the total friction present [2]. Submonolayer adsorbate converages have allowed us to probe the edge effects of surface nanoclusters, while multilayer coverages have let us explore the strength and proximity effects of surface roughness. We compare our measurements to those reported by Pierno et al. on films of ordered Pb(111) terraces, where atomic step edges are present [3], and conclude that the variation in reported values of friction on nanostructured lead is due to phononic effects at the step edges.

Funding provided by NSF DMR.

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[5] M. Kisiel et al., Nature Materials **10**, 119-122 (2011).

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5:20pm **TR-ThA11 Advanced SP3EC Carbon Nanocomposite Coatings**, ***J. Larson***, United Protective Technologies

United Protective Technologies (UPT) has developed a room temperature plasma assisted chemical vapor deposition (PACVD) coating process to build carbon based coatings. Versions of this coating are in use for infrared optical applications, galvanic corrosion barriers and tribological modifications to critical components. The process used to produce SP^3EC^{TM} coatings is compatible with a wide range of materials including semiconductors, metals, polymers and composites. Deposition parameters for the SP^3EC™ process can be controlled to produce a wide range of carbon based thin films, ranging from 120 nm diamond crystalline grains to low friction glassy amorphous carbon films. Layered “nano-composite” structures constructed of these films have been proven to improve the life; performance and reliability of components under high wear conditions and corrosion conditions. Additionally, the SP^3EC™ process is non-toxic and environmentally friendly.

Current applications for SP^3EC^{TM} coatings include targeting optics with improved durability for the AH-64 along with corrosion and wear coatings for UH-60 rotor components. Tribological coatings for improved efficiency of helical gears have been developed in conjunction with advanced wear coatings for aluminum and steel components. This presentation will include details on the mechanical and barrier characteristics of SP^3EC^{TM} coatings for various applications, current process capabilities and developmental applications of this coating process.

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