Tribology Focus Topic
Room: 111 - Session TR-WeA

Emerging Interfaces of Tribological Importance
Moderator: T. Scharf, The University of North Texas

2:00pm **TR-WeA1 Highly Wear-Resistant Surfaces Based on Fluorinated Alkyne-Derived SAMs on Si(111)**, ***S.P. Pujari***, *H. Zuilhof*, Wageningen University, The Netherlands

Micro-electro-mechanical systems (MEMS) are considered to be an important technology for the development of several products in daily life such as electronics, medical devices, and packaging. Even after tremendous progress in fabrication of miniaturized devices based on silicon materials, the development of highly robust surfaces with low friction and resistance against wear is still a challenging subject of accomplishment . To accomplish this goal, new fluorine-containing terminal alkynes were synthesized and self-assembled onto Si(111) substrates to obtain fluorine containing organic monolayers. Such covalently bound organic monolayers have similar surface properties as polytetrafluoroethylene (Teflon), but these monolayers are more stable than traditionally coated PTFE. The combination of these properties yields a highly improved wear resistantance.

A combination of spectroscopic (XPS, IR), microscopic (AFM), and contact angle measurements shows these monolayers were to be ordered and highly hydrophobic. Increasing the amount of fluorine on the alkyne precursor resulted in monolayers with a greatly reduced adhesion to silica probes, as well as an almost 5-fold decrease in the coefficient of friction on the surface. Overall, this yields a friction coefficient that is – to the best of our knowledge – lower than reported for <u>any</u> other fluorine-containing monolayer. In addition, these fluorinated monolayers displayed no sign of wear at high loads. Therefore, the use of such highly durable fluorine-containing monolayers can significantly expand the range of applications for MEMS. Therefore, this work opens a route to design new materials with tailor-made properties for a wider range of applications in MEMS-based devices.

2:20pm **TR-WeA2 Composition and Friction Analysis of Copolymer Solution Treatments of Silicone Hydrogel Contact Lens Surfaces**, ***S. Perry***, *Y. Huo, A. Rudy*, University of Florida

The surface chemical compositions of three major brands of silicone hydrogel (SH) contact lenses were analyzed using X-ray photoelectron spectroscopy (XPS) prior to and following treatment in a test solution of diblock copolymer of polyethylene oxide and polybutylene oxide. Atomic force microscopy (AFM) was also employed to evaluate the surface topography and frictional properties of these lenses prior to and following similar solution treatments.For surface compositional analysis with XPS, lens surfaces have been prepared through a vacuum drying procedure, in which the hydrogel is taken from a fully hydrated state directly to an ultraclean, ultrahigh vacuum environment. Contact and tapping mode AFM were used to measure the frictional and topographical properties in aqueous environments. Prior to treatment, differences in surface elemental composition of the various lenses were found to reflect known bulk compositions and/or respective surface treatments. Following solution treatment, surface chemical modifications were apparent in balafilcon A (PureVision®) and lotrafilcon B (O_2 OPTIX®), especially in the distribution chemical functionalities present at the surface. Only modest changes in surface composition were observed for the senofilcon A (ACUVUE® Oasys®) system. AFM measurements in saline revealed large disparities between the coefficients of friction of the three lenses, with balafilcon A and lotrafilcon B exhibiting coefficients of friction approximately five times greater than that of senofilcon A. Lens surface treatment with the diblock copolymer test solution produced a significant reduction in the coefficients of friction of the two lenses exhibiting higher friction, yet only a small reduction in friction was observed for senofilcon A lens. Together, these results depict a strong correlation between the surface chemistry and frictional response of the lens systems as they relate to solution treatment with this specific diblock copolymer. This study indicated that diblock copolymers containing polyethylene oxide and polybutylene oxide may have a positive impact on the lubrication and wetting properties of silicone hydrogel lenses.

2:40pm **TR-WeA3 In Situ Studies of Cartilage Microtribology**, ***D.L. Burris***, *E.D. Bonnevie, V.J. Baro, L. Wang*, University of Delaware **INVITED**

The progression of local cartilage surface damage toward early stage osteoarthritis (OA) likely depends on the severity of the damage and its impact on the local lubrication and stress distribution in the surrounding tissue. It is difficult to study the local responses using traditional methods; *in-situ* microtribological methods are being pursued here as a means to elucidate the mechanical aspects of OA progression. While decades of research have been dedicated to the macrotribological properties of articular cartilage, the microscale response is unclear. An experimental study of healthy cartilage microtribology was undertaken to assess the physiological relevance of a microscale friction probe. Normal forces were on the order of 50 mN. Sliding speed varied from 0 to 5 mm/s, and two probes radii, 0.8 mm and 3.2 mm, were used in the study. *In-situ* measurements of the indentation depth into the cartilage enabled calculations of contact area, effective elastic modulus, elastic and fluid normal force contributions, and the interfacial friction coefficient. This work resulted in the following findings: 1) at high sliding speed (V=1-5 mm/s), the friction coefficient was low ($\mu = 0.025$) and insensitive to probe radius (0.8 mm – 3.2 mm) despite the 4-fold difference in the resulting contact areas; 2) The contact area was a strong function of the probe radius and sliding speed; 3) the friction coefficient was proportional to contact area when sliding speed varied from 0.05 mm/s-5 mm/s; 4) the fluid load support was greater than 85% for all sliding conditions (0% fluid support when V=0) and was insensitive to both probe radius and sliding speed. The findings were consistent with the adhesive theory of friction; as speed increased, increased effective hardness reduced the area of solid-solid contact which subsequently reduced the friction force. Where the severity of the sliding conditions dominates the wear and degradation of typical engineering tribomaterials, the results suggest that joint motion is actually beneficial for maintaining low matrix stresses, low contact areas, and effective lubrication for the fluid-saturated porous cartilage tissue. Further, the results demonstrated effective pressurization and lubrication beneath single asperity microscale contacts. With carefully designed experimental conditions, local friction probes can facilitate more fundamental studies of cartilage lubrication, friction and wear, and potentially add important insights into the mechanical mechanisms of OA.

4:00pm **TR-WeA7 "Going No Wear?"**, ***W.G. Sawyer***, University of Florida **INVITED**

There is a need for the development of wear-resistant, low-friction materials, and understanding the fundamental origins of wear across length scales will be necessary to guide the development of such materials. The events at buried interfaces that lead to wear entail extreme variability in interaction strength, contact duration, and frequency of occurrence. It has been long postulated that the ensemble of these transient interactions at weak buried interfaces ultimately lead to the ever-present macroscopic phenomena of wear. The quest to find high performance solid lubrication solutions continues. Traditional solid lubrication techniques rely on a pre-deposited coating of a lubricous and/or a protective material, but since these materials wear during operation, the life of the system is finite. In order to extend the operational life indefinitely and to potentially negate any mechanical contributions to wear, a stable lubricating tribofilm of sufficient chemistry and thickness must be maintained during operation. However, because sliding occurs in a buried interface, it has proven challenging to determine what materials processes are actively enabling stable performance and/or what to add to the system to improve lubrication. In this talk, results from a number of ultra-low wear systems (polymers, metals, and ceramics) that have been studied using a variety of active and *in situ* tribological instrumentation will be presented, along with a discussion of the various mechanisms that we believe to be responsible for this unique behavior.

5:00pm **TR-WeA10 Study on the Fatigue Wear Behaviour of TiN and WC DLC-coated Stainless Steel under Inclined Impact-Sliding Load Tests**, ***Y. Chen***, *X. Nie*, University of Windsor, Canada

WC/C diamond-like carbon (DLC) coatings and Titanium Nitride (TiN) coatings are widely used in industrial machinery and tools. Both of the two coatings have extremely hard surfaces comparing to the stainless steel substrate SS316L. The W: DLC coating gives low coefficients of friction against a number of counterfaces, has relatively high lubricity and resistance to adhesive wear comparing to the TiN coating. In this study, a new method which is called cycled inclined impact-sliding test is introduced and utilized to study the coating durability under a combined force of an Impact force *Fi* and Pressing force *Fp* (*Fi/Fp=200N/400N* and

200N/200N) and the fatigue wear behaviors after up to 1000 cycles impact tests. A 10mm steel (AISI 52100) bearing ball is used as the impact indenter. Due to the low coefficient of friction of W:DLC coating against steel counterface, greater impact cycle was endurable before the failure of the coating when impacted in dry air condition. Under inclined impact and sliding forces, fatigue cracking was first initiated, followed by chipping and pealling of the coatings. The SEM showed that different types of fatigue wear cracks were found which distributed in the different areas (head and tail parts) of the impact scars. Most of those scars, observed by 45º tilted cross-sectional SEM, penetrated the coating and caused hardening of the under layer (interface layer). Material transfer from the indenter ball could also be detected by EDX in some areas of the impact scar on both of the coating materials.

5:20pm **TR-WeA11 Scaling Laws of Structural Lubricity for Amorphous and Crystalline Nanoparticles**, *D. Dietzel, T. Moenninghoff, M. Feldmann,* Westfaelische Wilhelms-Universitaet Muenster, Germany, ***U.D. Schwarz,*** Yale University, *A. Schirmeisen*, Justus-Liebig University Giessen, Germany

In an effort to reduce the friction between sliding components scientists and engineers have developed a multitude of lubrication schemes. One of the most intriguing concepts is referred to as `structural lubricity', where flat surfaces are thought to slide past each other virtually frictionless if their atomic structures are incommensurate. In this talk, we analyze the fundamental mechanisms that govern the area-dependence of friction in extended but atomically flat contacts of dissimilar materials with a particular emphasize on the relation between structure (crystalline vs. amorphous) and friction. The resulting sublinear power laws, which link mesoscopic friction to atomic principles, are then confirmed by measuring the sliding resistance of gold and antimony particles on graphite [1,2]. The findings suggest that engineering surfaces with unprecedented low friction can be realized.

[1] A. Schirmeisen and U. D. Schwarz, ChemPhysChem 10 (2009) 2358

[2] D. Dietzel et al., Physical Review Letters 101 (2008) 125505

5:40pm **TR-WeA12 Auger Surface Analysis of Deposits Formed on Magnetic Tape Recording Head Surfaces**, ***F.E. Spada,*** University of California, San Diego, *D.F. Paul, J.S. Hammond*, Physical Electronics

The 2008 International Magnetic Tape Storage Roadmap[1] projects that the total magnetic spacing between the recording head and the tape magnetic layer must decrease from the current 43 nm spacing to about 23 nm by the year 2018 in order for tape to maintain its cost advantage as an information storage medium. Because tape drives are contact recording systems, interactions between head materials and components in the tape magnetic layer can detrimentally affect the head-tape separation via deposit formation on head surfaces as well as preferential erosion of critical recording head elements. Understanding the nature of these interactions is therefore essential for mitigating undesirable increases in the magnetic spacing. This study shows that deposition and erosion phenomena in tape heads can be varied *at the local level* by changing the electrical configuration of adjacent pole tip structures in multichannel heads, and that the composition of the head deposits depends on the electrical configuration of the pole tips. Using atomic force and electric force microscopy, we show that conductive deposits form on the “trailing edge” of pole tips which are electrically connected to earth ground or to the head substrate. The conductive deposits become non-conductive further “downstream” from the pole tips. Deposits adjacent to electrically isolated poles are always non-conductive. Auger analysis shows that the surfaces of the conductive deposit regions contain high levels of Fe and Co, and small amounts of P and Y, whereas the surfaces of the non-conductive deposits contain predominantly P and Y, with very low levels of Fe. Because all of these elements are present in the magnetic coating of the tape, and because the compositions of the deposits on heads having NiFe pole tips is similar to those on heads having CoZrTa pole tips, these results suggest that the deposits originate from components in the tape and not from metallic structures in the tape head.

1. *International Magnetic Tape Roadmap*, Information Storage Industry Consortium, September, 2008.

*Supported by the Information Storage Industry Consortium Tape Program

Tribology Focus Topic
Room: 111 - Session TR+AS+SS-ThM

Atomic-scale Characterization of Tribological Interfaces
Moderator: S. Perry, University of Florida

8:00am **TR+AS+SS-ThM1 Electrochemical Control of Atomic Friction**, ***F. Hausen***, INM - Leibniz Institute for New Materials, Germany, *A. Labuda,* McGill University, Canada, *N.N. Gosvami, R. Bennewitz*, INM - Leibniz Institute for New Materials, Germany

Electrochemical methods allow for fast and reversible modification of metal surfaces through deposition and dissolution of metal films, adsorption and desorption of anions, as well as oxidation and reduction. The surface composition and structure undergo dramatic changes in these processes, which should cause significant changes in the friction on the surface.

We present friction force measurements at the nanometer scale on Au(111) and Au(100) single crystal electrodes performed by means of friction force microscopy in various electrolytes. The resolution of atomic stick-slip events in an electrochemical cell is improved by the development of a dedicated instrument [1]. A significant difference in friction is found for the bare electrodes compared to the modified surfaces. Friction is extremely weak and exhibits almost no load dependence on clean Au(111) surfaces. Upon electrochemical oxidation of the surface, significant friction with linear load dependence is observed. This process is reversible and allows switching repeatedly between high and low friction [2]. In the regime of anion adsorption our results indicate a frictional response with threshold behaviour. The threshold depends on both applied normal load and the electrochemical potential [3].

After deposition of copper on gold by underpotential deposition in perchloric acid, the atomic stick-slip changes into a periodicity which indicates frictional response of CuCl with a linear load dependence. In chloride-free sulphuric acid a different behaviour is found, indicating competing effects of ion adsorption on friction forces at small scales.

[1] A. Labuda et al., Rev. Sci. Instruments 81, 083701 (2010)

[2] A. Labuda et al., Langmuir (2011, available online)

[3] F. Hausen et al., Electrochimica Acta (2011, in print)

8:20am **TR+AS+SS-ThM2 Surface Alterations Effects on Ice Adhesion Strength**, ***C. Ellis-Terrell***, *M. Miller,* Southwest Research Institute, *M. Zou,* University of Arkansas at Fayetteville, *R. Wei,* Southwest Research Institute, *S. Beckford,* University of Arkansas at Fayetteville, *G. Hatton*, Shell Global Solutions, Inc.

Ice adhesion is a serious problem in areas such as the oil, gas, and automotive industry, telecommunications and power line transmission. There is a significant amount of research directed towards designing a coating to reduce ice accumulation. This study focuses on measuring the effects of surface roughness and surface energy on ice adhesion strength. Surface texturing ranged from high to low surface roughness. A sandblasting technique was applied to the aluminum surface creating a high surface roughness. Surface energy changes were created by depositing a silicon doped hydrocarbon film, using plasma enhanced vapor deposition. A custom built apparatus was employed to specifically measure the adhesion force of an ice droplet. The results illustrate that the smoother as-received surfaces have lower ice adhesion strength than the rougher sandblasted surfaces.

8:40am **TR+AS+SS-ThM3 Atomistic Simulations of Nanoindentation and Nanoscratching of SiO_2/Si and HfO_2/Si Systems using COMB Potentials**, ***T.-R. Shan***, *X. Sun, S.R. Phillpot, S.B. Sinnott*, University of Florida

Oxides such as SiO_2, Al_2O_3 and HfO_2, are typically used together with Si in many high-performance electronic devices, including metal-oxide-semiconductor (MOS) devices/junctions and micro- and nano-electromechanical systems (MEMS/NEMS). The lack of precise control over mechanical properties can lead to the degradation of these materials. It is therefore critical to understand the nanometer-scale mechanical properties of materials or complex systems being considered for use in electronic devices. Nanoindentation and nanoscratching are important methods for investigating the mechanical behavior of small volumes of materials, such as thin film systems. Here, classical molecular dynamics simulations are used to examine the responses to nanoindentation and nanoscratching of thin films of SiO_2 and HfO_2 on silicon substrates. The goal is to determine the influence of thin film types and the structure of thin film and substrate interface on the responses. Because these systems consist of heterogeneous interface with significant changes in bonding as one crosses from one side of the interface to the other, the empirical charge optimized many-body (COMB) potential as implemented in large-scale atomic/molecular massively parallel simulator (LAMMPS) program is used to model the structural evolution, mechanical response and charge transfer in these systems in response to a nanometer-scale spherical indenter. Aspects of the SiO_2/Si and HfO_2/Si interfaces during nanoindentation and nanoscratching, including the mechanisms by which fracture and plasticity occurs, will also be addressed. We gratefully acknowledge the support of the National Science Foundation through grant numbers DMR-0426870 and DMR-1005779).

9:00am **TR+AS+SS-ThM4 Accelerated Molecular Dynamics Simulations of Nanoscale Friction**, *W.K. Kim,* University of Minnesota, ***M.L. Falk***, Johns Hopkins University **INVITED**

Accelerated molecular dynamics simulations are implemented to model the sliding process of atomic force microscope experiments and to lower the sliding speeds below those in a conventional MD simulation. In this study the hyperdynamics method, originally devised to extend MD time scales for non-driven systems, is applied to the frictional sliding system. This technique is combined with a parallel algorithm that simultaneously simulates the system over a range of slider positions so that the overall acceleration rate is approximately the number of processors multiplied by the boost factor from the hyperdynamics method. The new methodologies are tested using two-dimensional and three-dimensional Lennard-Jones AFM models. The methodology is then applied to simulated sliding between an oxidized silicon tip and surface achieving a range of six decades of velocity and reproducing the experimentally observed velocity dependence of the friction force. In doing so we learn something new about this system and about friction between amorphous surfaces in general. Unlike in the crystalline case, as increasing force is applied to the amorphous tip intermediate states arise. These intermediate states serve as critical transition pathways. The emergence of such states leads to the emergence of a plateau in sliding velocity at lower sliding speeds and higher temperatures. A simple theory based on these observations successfully describes both the experimental and the simulated data.

9:40am **TR+AS+SS-ThM6 Molecular Dynamics Simulations of Contact between Carbon-Based Materials: Isolating the Effects of Experimental Variables**, ***J.A. Harrison***, *K.E. Ryan, P.L. Keating,* US Naval Academy, *D.S. Grierson, J. Liu, K.T. Turner,* University of Wisconsin Madison, *R.W. Carpick*, University of Pennsylvania

The behavior of nanoscale contacts is complex and often cannot be understood through continuum mechanics alone. Here, parallel molecular dynamics (MD) simulations using the AIREBO potential for hydrocarbons to model indentation and friction, are used to investigate nanoscale contacts of carbon-based materials, such as diamond, DLC, and ultrananocrystalline diamond (UNCD). Specifically, the contact of carbon-based AFM probes is simulated to understand the effects of experimental parameters, including tip geometry and material selection, on the adhesion between the tip and sample. Results from the MD simulations will be compared to and discussed within the context of the complementary atomic force microscope experiments and finite element simulations. The tribological response of carbon-based materials is very sensitive to environmental conditions. For example, the presence of water has been shown to negatively impact the friction performance of hydrogenated DLCs but to improve the performance of nanocrystalline and ultrananocrystaline diamond. We have been working to develop a potential energy function that is capable of modeling carbon-based materials in the presence of water. This talk will also outline our current efforts at potential development.

10:40am **TR+AS+SS-ThM9 Modeling the Pressure Dependence of Shear Strength in Sliding, Boundary-Layer Friction**, ***M. Garvey***, *M. Weinert, W.T. Tysoe*, University of Wisconsin-Milwaukee

The pressure dependence of the shear strength of model alkali halide lubricant systems has been investigated at the density functional theory level. This is compared to the experimental dependence given by $S = S_0 + \alpha P$, where P is the contact pressure, S_0 is the zero-pressure shear-strength and α is the coefficient of pressure dependence. Sliding potentials were calculated and shear is found to occur between the film and the sliding interface. The heights of the potentials were calculated as a function of compression, allowing the lateral force to be calculated as a function of pressure. The calculated values of S_0 and α are in good agreement with experimental data.

11:00am **TR+AS+SS-ThM10 Lubrication Mechanisms of MoS_2 Fullerene-Like Nanoparticles: Coupling Computer and Experimental Works**, ***E.W. Bucholz***, University of Florida, *I. Lahouij, F. Dassenoy,* Ecole Centrale de Lyon, France, *S.B. Sinnott,* University of Florida, *J.M. Martin*, Ecole Centrale de Lyon, France

Inorganic fullerene (IF)-like MoS_2 nanoparticles have been shown to be good lubricating and anti-wear additives when dispersed in a base oil. This improved tribological performance appears to be a result of the size and structure of the nanoparticles along with the test conditions. Possible lubrication mechanisms include pure rolling to sliding to the exfoliation of lamellar MoS_2 sheets inside the contact. *In situ* transmission electron microscopy (TEM) experiments have been used to manipulate individual MoS_2 nanoparticles and investigate their responses to compression and friction under different conditions. However, the very small scale of the MoS_2 nanoparticles makes distinguishing the properties which affect the lubrication mechanism exceedingly difficult; thus, a computational approach is used to more fully understand the most important mechanisms. Therefore, classical molecular dynamics (MD) simulations of individual nested MoS_2 nanoparticles are performed where they are subjected to compression and shear forces between sulfur-terminated molybdenum surfaces. Two specific nanoparticle configurations are considered, with both structures containing three layers. The first configuration is a curved, ellipsoidal MoS_2 nanoparticle structure with a major and minor diameter of approximately 8.9 and 6.6nm, respectively. The second nanoparticle configuration is an octahedron with grain boundaries that are approximately 6.2 nm in length. MD simulations of these structures indicate the role of curved and faceted morphologies as well as grain boundaries on the rolling/sliding behavior and nanosheet exfoliation of the particles. The results are used to interpret the experimental TEM findings and predict the dominant mechanisms associated with enhanced lubrication through the addition of these particles to base oils. This work is supported by the Office of Naval Research .

11:20am **TR+AS+SS-ThM11 Shape-Independent Lateral Force Calibration**, *E.V. Anderson,* ***N.A. Burnham***, Worcester Polytechnic Institute

The primary problem with lateral force microscopy (LFM) has been the difficulty in calibrating the cantilever and tip in order to obtain quantitative friction data. Two recent review articles and several research articles have expressed this difficulty and the need for a simple, universally-accepted method [1,2]. The available procedures have numerous limitations. Some require specialized samples or setups. Others are difficult to perform. A number are indirect, or only suitable for certain cantilevers. Several risk damage to the tip or sample, or both, and might require the geometry of the cantilever, which can be hard to measure. We present a procedure that alleviates these problems [3]. The linear relationship between the detected voltage and lateral force is exploited to obtain the slope (calibration factor) and intercept that convert voltage to lateral force. The method is independent of sample shape, probe shape, and scan parameters (load force, gain, and scan rate). The accuracy was investigated on an order-of-magnitude level and was within 50% of torsional spring constants obtained from geometry, and the precision was under 10%. Small scan areas were also found to produce accurate calibration factors and could help to limit tip-sample wear. Quantification of nano-Newton friction forces might now become routine.

1. M. L. B. Palacio, B. Bhushan, *Crit. Rev. Solid State Mater. Sci.* **2010**, *35*, 73-104.

2. M. Munz, *J. Phys. D: Appl. Phys.* **2010**, *43*, 063001.

3. E.V. Anderson, S. Chakraborty, T. Esformes, D. Eggiman, C. DeGraf, K. M. Stevens, D. Liu, and N.A. Burnham, “Shape-Independent Lateral Force Calibration,” submitted April 2011.

11:40am **TR+AS+SS-ThM12 Atomic Stick-Slip Friction Studied by Optimally-Matched Accelerated MD Simulations and AFM Experiments**, ***Y. Dong***, Purdue University, *Q. Li, R.W. Carpick,* University of Pennsylvania, *A. Martini*, Purdue University

Atomic-scale stick-slip friction of platinum on gold (111) surface is quantitatively studied both experimentally and through optimally-matched accelerated molecular dynamics (MD). In order to make a direct comparison between simulation and experiment, many other factors are matched as closely as possible, such as misalignment, size effect of the tip, cantilever compliance, normal load and so on. The Parallel Replica Dynamic Method (ParRep) is used to accelerate the simulation so scan velocities can be decreased to scales approaching those used in atomic force microscope experiments. A logarithm dependence of friction on scanning velocity is observed both in Atomic Force Microscope (AFM) and MD reveals that at low speed the atomic friction lies in thermal activation regime. A further comparison shows that AFM and MD provide consistent energetics, which supports that MD can be used to interpret AFM results; but attempt frequencies differ by orders of magnitude, which is attributed to the inertia discrepancy.

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 $\mu = 0.6$ 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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5:20pm **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.

Tribology Focus Topic
Room: East Exhibit Hall - Session TR-ThP

Tribology Focus Topic Poster Session

TR-ThP1 Subsurface Characteristics of an Abraded Fe-0.4wt%C Martensitic Steel using Nanoindentation and Cross-Sectional TEM Techniques, ***F. Katsuki***, Sumitomo Metal Industries, Limited, Japan

The present investigation of unidirectional abraded surfaces of a martensitic (0.4 wt%C) steel with silicon (1.5wt%Si), chromium (1.5wt%Cr) and molybdenum (1.9wt%Mo) addition elucidates the work hardening and the softening near the surface layer caused by abrasion, particularly its relation to the wear behavior. The abrasion testing was performed using a pin-abrasion apparatus in which a small pin of the specimen was ground on an abrasive paper at an applied load of 2.1N and sliding speed of 0.66m/s. Crushed silica particles (size: 15-67µm) were used as the abrasive medium. The abraded surfaces were examined with a nanoindentation apparatus to evaluate the variation of nanohardness with sliding time on a nanometer scale. A cross sectional transmission electron microscope (TEM) technique was also employed to clarify the structural changes in the region close to the abraded surface. It has been found that abrasion induced work hardening with sliding time was observed in the case of chromium and molybdenum addition steels. A fine dispersion of molybdenum carbide (Mo_2C) was observed in the surface of the molybdenum steel after abrasion. Mo_2C precipitates at approximately 550°C, indicates that surface and near surface temperatures would be over the carbide formation temperature by the abrasion induced frictional heating. On the other hand, the softening has been indicated to be caused by the abrasion heating leading to some tempering effects in the case of the silicon addition steel. Work hardening and softening caused by abrasion induced subsurface deformation and frictional heating respectively seem to be two processes taking place simultaneously which counteract each other's effect. Metallurgical reaction such as precipitation and temper by frictional heating has been found to play a important role in controlling the wear characteristics of steels. The influence of the alloying element addition on the wear response of the martensitic steel will be discussed.

TR-ThP2 The Lubrication Characteristics of Molybdenum Disulfide Films by RF Magnetron Sputter, ***A. Kasahara***, *M. Goto, M. Tosa,* National Institute for Materials Science (NIMS), Japan*, T. Maruyama, J. Nakagawa, K. Endo*, Toyama Co., Ltd., Japan

For vacuum applications, lubricants which have low vapor pressure and good tribological performance are required. Solid lubricants have been widely adopted for vacuum applications due to their low vapor pressure. Thin film lubricants and high-temperature solid lubricants are discussed as the latest alternatives to fluorine grease. These lubricants feature lower out-gassing for clean and vacuum environments. One big drawback of solid lubrication is limited life. We investigate solid lubricant in order to develop high performance ultra-high vacuum (UHV) manipulation mechanism and high-temperature drive devices. We focused sputtering coating method and Molybdenum Disulfide (MoS_2) , since this material has been reported to be suitable in vacuum , but not well been investigated. As a first step, we have carried out basic study of MoS_2 thin film to find out suitable sputtering parameter. It was deposited on martensite stainless steel (SUS440C) substrates, using radio frequency magnetron sputter. Their tribological characteristics in UHV and high-temperature was evaluated by vacuum friction measurement system, high-temperature friction measurement system based on Bowden-Leben type and abrasion measurements by Stylus Surface Profiler.

TR-ThP3 Structure of Thin Diamond-Like Carbon Films and its Relationship to its Tribological Performance, ***F.J. Flores-Ruiz***, *F.J. Espinoza-Beltran, M.O. Vazquez-Lepe, A. Herrera-Gomez*, CINVESTAV-Unidad Queretaro, Mexico

Diamond-like carbon (DLC) films are promising materials for dry-contact applications where resistance to surface damage or lubricating performance is required. In the present work, the structure of 3 and 10 nm Focus Cathodic Arc (FCA) carbon films and 3 and 10 nm plasma carbon films grown on Si(100) was studied by angle-resolved X-ray photoelectron spectroscopy (ARXPS). The concentration and distribution of sp^2 and sp^3 carbon within the film was assessed with methods described elsewhere [i]. Atomic force microscopy (AFM) with a silicon tip was used to study the friction coefficient µ on the nanoscopic scale. It is defined by the Amonton law, $\mu=F_L/F_N$, where F_N and F_L are the normal and the lateral force applied to a probe [ii]. The calculation of the normal and lateral force applied during the process of sliding the tip over the sample was done according to the methodology proposed by Carpick [iii] . The results suggest a relationship between the chemical structure and the tribological performance for each film tested. The films grown by FCA presented the best tribological performance (µ~*0.02*) compared to films grown by plasma (µ~*0.06*) indicating a direct link between the distribution of sp^2 and sp^3 carbon and the nanoescale friction coefficient. Since during the friction tests there was no evidence of damage or wear on either the surface or the tip, the conventional interpretation for the origin of friction proposed by Bowden and Tabor [iv] grossly underestimates the energy loss in the sliding process. On the other hand, the energy loss can be quantitatively explained in terms of heat generation [v]. The tribological performance analysis indicates that the friction is related to the adhesion force between tip and sample. However, the friction coefficient values do not show significant changes as the strength of adhesion varies, indicating that the value of the friction coefficient depend on the contributions of atomic bonds at the surface.

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TR-ThP4 Tribological Investigations of Octadecylphosphonic Acid (ODP) and Octadecyltrichlorosilane (OTS) Self-Assembled Monolayers: A Comparative Study of MEMS-type Interfaces, *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*, B.P. Borovsky,* Saint Olaf College*,* ***E.E. Flater***, Luther College

Microelectromechanical systems (MEMS) are critically-limited by interfacial phenomena such as friction and adhesion. One strategy to reduce friction between MEMS surfaces is to coat them with molecularly-thin self-assembled monolayer (SAM) coatings. Historically, silicon MEMS have been coated with silane-based SAMs, such as octadecyltrichlorosilane (OTS). However, continued progress in the development of MEMS may require new material systems to be employed. Therefore, in this study, we have investigated the frictional properties of octadecylphosphonic acid (ODP) monolayers deposited on aluminum oxide surfaces, across speed regimes. Measurements using an atomic force microscope (AFM) and separately using a nanoindenter-quartz crystal microbalance system were performed each with a microsphere-terminated probe. This allows for a comparative study between different velocity regimes using contacts with similar sizes, pressures, surface roughnesses, and interfacial chemistries. AFM colloidal probe friction measurements indicate that for a bare tip sliding on various substrates, ODP-coated alumina surfaces exert a lower friction force than either bare or OTS-coated alumina substrates. We also observed strong evidence of transfer of the ODP molecules to the tip when the tip is uncoated. The results presented in this study are significant contributions towards our goal of better understanding the frictional properties of phosphonate SAMs in pursuit of alternative MEMS materials.

TR-ThP6 The Effect of Test Parameters on the Tribocorrosion Behavior of Multilayers, ***M. Flores****, O. Jiemnez, J. Garcia, E. Rodriguez,* Universidad de Guadalajara, Mexico*, L. Huerta*, Universidad Nacional Autononoma de Mexico

The multilayer coatings can improve the corrosion and wear resistance of materials for biomedical applications. The tribocorrosion behavior of TiALN and $TiAlPt_xN$ coatings and $TiAlPt_xN$-$TiAlPt_x$ multilayers immersed in a corrosive environment was investigated. The coatings were deposited on 316L stainless steel and Ti6Al4V alloys by magnetron sputtering. The period thickness of multilayers was 300 nanometers and the total thickness was 3.6 microns. In order to evaluate the influence of the environment, the corrosion was studied using open circuit potential (OCP) measurements and potentiodynamic polarization techniques in saline and a Ringer ´s solutions. For the tribocorrosion test a counterbody of Alumina with 10 mm diameter was used. The loads used were from 1 to 5 N, the oscillating frequencies were 1Hz to 5 Hz. The electrochemical noise measurements were performed during, and after the sliding and scratch tests. The structure and composition of multilayers were studied by means of XRD, XPS and RBS techniques. It was found that the codeposition of Pt and TiAlN-TiAl multilayer can improve the wear-corrosion resistance of materials for biomedical applications.The tribocorrosion behavior results in terms of the coefficient of friction showed a dependence against the force and the sliding frequency.

TR-ThP7 Tribological Properties of Electron Beam Excited Plasma Nitrided Surfaces, ***P. Abraha***, *J. Miyamoto*, Meijo University, Japan

Tribological properties play significant role on the performance of tool steel surfaces. Here, application of electron beam excited plasma nitriding and its effect on tribological properties is described. The technique eliminates the formation of the brittle and rough compound layer that is common in nitriding processes. The hardening process is done through diffusion of the plasma species in to the subsurface of the treated material without altering the initial surface finish. The applications of the process can be in areas of hard coating where adhesion of the coating material with the tool steel is of significant importance.

The experimental tool steel material is SKD 61 with a chemical composition of 0.36% C, 5.05% Cr, 1.21% Mo, 0.83% V, 0.92% Si, 0.43% Mn, 0.008% P, >0.001% S, Fe bal. The sample was heat treated, hardened and triple tempered to a hardness of 630 Hv. The sample was then treated in a nitrogen plasma produced by a beam current of 8 A under a working pressure of 0.4 Pa. The temperature was set at 500 degrees centigrade throughout the treatment time. The experimental set up includes bias terminals that reduce the ion density within the vicinity of the tool steel material. This is done to reduce nitriding due to ion and increase the chance of nitriding due to neutral species within the plasma. The cross sectional hardness distributions and wear measurements of the nitrided tool steels were examined to determine the mechanical and tribological surface properties. The surface has no trace of the compound layer that is usually observed in the ion nitriding processes. This is also confirmed from the X-ray Diffraction peaks, as there is no visible Fe_3N and Fe_4N peaks observed. These results are attractive as they open new areas of application especially in the coating industry where adhesion remains to be the limiting factor in lots of the hard coatings that protect cutting and forming tools against wear.

TR-ThP8 Parameter Optimization of Ion Plated Nickel-Copper-Silver Lubrication, ***M. Danyluk***, *A. Dhingra*, University of Wisconsin Milwaukee

In this paper we present a connection between argon ion flux with ion mixing and rolling contact fatigue (RCF) life of a thin solid nickel-copper-silver film lubrication on ball bearings tested in high vacuum. Using a Langmuir probe we measure plasma properties and ion flux and then calculate plane stress within the film during deposition using a validated model found in the thin film science literature. Experiments reveal that there is an inverse relationship between ion flux and RCF life for most deposition voltage and pressure combinations tested, specifically, 15.5 to 18.5 mTorr and 1.5 to 3.5 kV. For voltages up to 2.5 kV, RCF life decreases as deposition voltage and ion flux increase. Experiments also confirm that as ion flux increases deposition rate decreases due to sputter removal from the ball surface. For voltages between 2.5 and 3.5 kV interlayer mixing and contamination of the 100 nm thick coating reduces RCF life even as ion flux decreases with decreasing process pressure within a constant power process. At ion energy greater than 2.5 keV and ion flux above 10^{15} cm^{-2} s^{-1}, SRIM results suggest elemental mixing of copper and nickel at the interface and this is confirmed using Auger Electron Spectroscopy (AES) on steel and Si_3N_4 5/16" diameter balls.

TR-ThP9 Shear-induced Tribofilm Formation: Boron Containing Molecules on Copper, ***B.P. Miller***, *O.J. Furlong, W.T. Tysoe*, University of Wisconsin Milwaukee

The push for greener lubricants has steered focus away from compounds containing sulfur and phosphorus. The tribological chemistry can depend critically on the nature of the substrate so that a good lubricant additive for one type of surface may not be applicable to another. In particular, the lubrication of sliding copper-copper interfaces in electrical motors provides a challenge due to the requirement for a conducting interface. Boron containing molecules have been proposed as potential green lubricants since environmentally safe boric acid is a prominent decomposition product in the presence of water. The following investigates the chemistry and frictional properties of alkoxy dioxaborolane (borolane) on copper surfaces in ultrahigh vacuum (UHV) to determine the precursor at room temperature that can potentially form a tribofilm needed for the lubrication of sliding copper-copper contacts. Temperature programmed desorption (TPD) and X-ray photoelectron spectroscopy (XPS) experiments provide the background information for investigating the frictional properties of borolane. A sliding copper-copper interface is exposed to borolane under UHV conditions, and a significant reduction in friction is found from the clean-surface values. A lasting tribofilm persisted even after the borolane dosing was stopped, and carbon was found on and below the surface in the wear scar region by *in-situ* Auger spectroscopy. Because the interfacial temperature rise under the experimental conditions used to measure friction is <1 K, the tribofilm formation is shear- and not thermal-induced

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