Tribology

Room: 617 - Session TR4+SE-FrM

Friction and Wear of Engineered Surfaces Macro- to Nanoscale Approaches

Moderator: N.D. Theodore, Naval Research Laboratory, North Carolina State University

8:00am **TR4+SE-FrM1 Mechanical, Chemical, and Tribochemical Etching of Silicon Studied by Atomic Force Microscopy[1]**, ***F. Stevens***, *S.C. Langford, J.T. Dickinson*, Washington State University

Commercial silicon nitride tips for atomic force microscopy (AFM) were used as model asperities to study the mechanical and tribochemical wear of a Si(110) surface. Aqueous sodium hydroxide and tetraethyl ammonium solutions were employed as chemical etchants. Under these conditions, tip wear is a significant issue; a new tip was employed for each wear measurement. Changes in tip contact stress were estimated by characterizing tip shapes before and after wear. In basic solution, the native oxide can be patterned by the AFM tip to expose the more vulnerable underlying Si to the chemical etchant.[2] Features 20 nm deep, with lateral dimensions less than 100 nm, are readily produced. The rate of oxide wear is a strong function of force applied to the AFM tip; even at low contact forces, scanning significantly accelerates oxide wear. Initial penetration of the native oxide is not uniform and produces deep pits—presumably at pinholes or similar defects in the oxide. Once the oxide is fully penetrated in the scanned region, subsequent tribochemical etching produces depressions with flat, smooth bottoms. For a given contact force and solution, the final wear depth relative to the surrounding, chemically etched material depends only on the number of times the AFM tip has passed over the surface; changing the tip velocity has no significant effect. Thus the tribochemical component of wear is not limited by chemical reaction rates under the conditions of this work. To characterize mechanical wear apart from chemical effects, hydrogen-terminated Si surfaces were scanned in inert solutions and atmospheres; images after wear in argon show wear debris, consistent with abrasive wear. The observed abrasive wear is sufficient to account for 5-10% of the Si wear observed in the presence of basic solution. Tribochemical effects during Si wear at a contact force of 300 nN in 0.1 M NaOH can easily enhance the total etch rate by a factor of two over the sum of the chemical and mechanical wear rates.

[1]This work was supported by the US National Science Foundation under Grant CMS-0409861.

[2]S. Miyake and J. Kim, Nanotechnology 16, 149 (2005).

8:20am **TR4+SE-FrM2 New Techniques for the Quantitative Determination of nm/hr Wear Rates of Materials**, *P.R. Norton, G. Pereira,* ***Y.-R. Li***, University of Western Ontario, Canada, *A. Lachenwitzer,* Cameco Corporation, *A. Alpas,* University of Windsor, Canada, *W. Capehart*, General Motors

Much effort has been spent on the chemical and mechanical characterization of antiwear films formed from ZDDP additives in automobile engines and on the mechanisms of their formation. By contrast, much less effort has been devoted to evaluating the wear itself. This is largely because the wear rates required in the engine are of order nm/hr, and accelerated testing under high loads is unacceptable because of the ubiquity of wear-rate transitions which make extrapolation to low loads difficult or impossible. The absence of relevant low-load (ultra-mild) wear data means that it is currently not generally possible to correlate the film characterization and wear rate studies, creating a huge gap in our understanding of wear. We are addressing this unsatisfactory situation by developing new techniques for directly measuring wear rates down to nm/hr in tests lasting a few hours. These techniques must be capable of measuring both the initial and long-term wear rates, be valid in the presence of surface films and take account of retention of material during wear. The strategy involves the implantation of Au atoms into the near-surface 100 nm region of a material (currently 52100 steel or an Al-Si alloy), and the determination of the loss of gold from the samples by means of Rutherford Backscattering (RBS) and Heavy Ion Backscattering (HIBS), and the accumulation of gold in the lubricant by Neutron Activation Analysis (NAA). The initial profiles can be either uni-modal or multi-modal with depth, to provide sensitivity to short and long-term behaviour. The analysis of both sample and lubricant/debris quantitatively accounts for all implanted gold, and the depth resolution of HIBS permits determination of the location of the residual gold in the samples.

8:40am **TR4+SE-FrM3 Nano-scale Friction and Wear of Carbon-Based Materials**, ***R.W. Carpick***, University of Pennsylvania, *D.S. Grierson, A.R. Konicek, P.U.P.A. Gilbert,* University of Wisconsin-Madison, *A.V. Sumant, O. Auciello,* Argonne National Laboratory, *R.J. Cannara,* IBM Zürich, Switzerland, *T.A. Friedmann,* Sandia National Laboratories, *W.G. Sawyer,* Univeristy of Florida, *J.A. Carlisle*, Advanced Diamond Technologies **INVITED**

Nanostructured carbon-based materials, such as nanocrystalline diamond and atomically smooth, nanometer-thick diamondlike carbon films, have outstanding and in many cases unrivalled tribo-mechanical properties. The atomic structure of the surfaces, verified by detailed surface spectroscopy, critically affects nano-scale friction and adhesion. We will specifically discuss how hydrogen termination, sp2-content, and crystal orientation affect adhesion and friction at the nanoscale. Next, we discuss how surface spectroscopy and imaging can be simultaneously applied to study wear of carbon films using photoelectron emission microscopy (PEEM)/X-ray absorption near edge structure (XANES). From these studies we test the hypotheses of ultralow friction in these materials, specifically, the extent to which chemical passivation of dangling bonds and convertion to graphitically bonded carbon occur. Finally, we will discuss taking advantage of this knowledge for nanoelectromechanical systems and advanced nanoprobe applications.

9:20am **TR4+SE-FrM5 Wear in MEMS-Based Microball Bearings**, ***B. Hanrahan***, *M. McCarthy,* University of Maryland, *C. Zorman,* Case Western Reserve University, *A.V. Sumant,* Argonne National Laboratory, *R. Ghodssi*, University of Maryland

PowerMEMS devices supported on microball bearings have been successfully demonstrated at low operating speeds. Continued research on the tribological properties of these support structures is necessary for the realization of robust high-speed devices. Critical dimensions need to be maintained within tight tolerances when operating high speed micromachines. The wear between stationary and moving elements must, therefore, be minimized to reduce the change in critical dimensions over the lifetime of the device. Accordingly, the mechanism of wear between silicon and hardened steel microballs is the focus of this work. Silicon test structures, 23 mm in length, were fabricated with 300 µm wide deep-etched rectangular trenches acting as housings for the microballs. The 285 µm steel balls were sandwiched between two test structures under an applied normal loading. Oscillatory motion is generated between the two structures using an externally controlled stepper motor at 400 rpm and a traveling distance of 4mm. At periodic time intervals, the test device is disassembled and inspected using an optical profilometer to determine changes in the surface topography. To the best of our knowledge, this is the first investigation of wear in MEMS-fabricated ball bearings. Initial results show both deposition and removal of material within the microfabricated trenches. At 50,000 cycles, equivalent to 200 meters of travel, an increase in average surface roughness from 7.5 nm to 30.2 nm is observed. These results suggest the adhesion and subsequent shearing of contacting materials, increasing the overall roughness. The properties of the mating materials play a critical role in determining the wear mechanism. To expand the scope of the study, solid thin film lubrication has been explored. Silicon carbide and ultra-nanocrystalline diamond are ideal candidates for wear resistant films because of their high hardness, low interfacial energy, and compatibility with MEMS fabrication processes. In our analysis we will present a detailed investigation of the wear mechanisms of steel balls on silicon with and without solid lubrication.

9:40am **TR4+SE-FrM6 Microstructure and Tribological Behavior of W-DLC Coated Rubbers**, ***Y.T. Pei***, *X.L. Bui,* University of Groningen, the Netherlands, *X.B. Zhou,* SKF Research and Development B.V., the Netherlands, *J.T.M. De Hosson*, University of Groningen, the Netherlands

W-DLC coatings have been deposited on FKM, ACM and HNBR rubbers via unbalanced magnetron reactive sputtering from a W target in C_2H_2/Ar plasma. The surface morphology and fracture cross sections of uncoated and coated rubbers are characterized with high resolution SEM. The tribological behaviors of uncoated and coated rubbers have been investigated with ball-on-disc tribotest under dry sliding against 100Cr6 ball. The coefficients of friction (CoF) of uncoated rubbers are very high ($\mu > 1$). Relatively high CoF of W-DLC coated FKM (about 0.6) is observed due to the gradual fracture and delamination of the coatings. In contrary, W DLC coated HNBR rubbers exhibit superior tribological performance with a very low CoF of 0.2-0.25 (comparable to that of Me-DLC coatings deposited on steel substrates). After 10000 sliding laps, almost no damage of the coatings is observed on the wear tracks. The micro-crack networks as deposited facilitate the flexibility of the coatings. The different surface roughness and mechanical properties of the rubber substrates explain the differences in the tribological performances of the coated rubbers. For soft and flexible substrates such as rubbers, metallic interlayer does not enhance the interfacial adhesion but negative effects are observed.

10:00am **TR4+SE-FrM7 Adaptive Tribological Nanocomposite Coatings**, ***C. Muratore***, *J.J. Hu, A.A. Voevodin*, Air Force Research Laboratory **INVITED**

Plasma processing allows precise control of the composition, microstructure and architecture of nanocomposite coating materials, enabling the design of materials that automatically adapt to variable aerospace environments, including humid and dry ambient air, space, and high temperature oxidizing conditions. Self-adaptive materials are of particular interest in tribological applications because most solid lubricants are effective in only a narrow range of ambient conditions. Thoughtful selection of multiple solid lubricant phases and the development of novel lubricant delivery mechanisms such as controlled diffusion, catalysis and chemical reactions with the surrounding atmosphere enhanced by contact between moving surfaces have recently been realized to achieve adaptation in diverse environments. For example, nanocomposite yttria-stabilized zirconia (YSZ) coatings containing Ag, Mo and other nanosized inclusions depend on temperature-activated adaptations to yield low friction (<0.2) from 25-700 °C in air. For YSZ-Ag-Mo nanocomposites, lubrication below 500 °C results from diffusion and coalescence of silver at the surface. Above 500 °C, the silver is pushed out of the wear contact, exposing a limited quantity of molybdenum in the coating to air, and initiating the controlled formation of MoO_3 and other compounds exhibiting low shear strength and thus easy sliding at high temperatures. While these adaptations at the surface reduce friction, irreversible compositional and structural changes resulting from the migration of film components can compromise the mechanical properties of the coating, thus reducing its utility over long periods at high temperature or through multiple thermal cycles. Moreover, some lubrication mechanisms occur over the entire coating surface in addition to the area experiencing wear, thus wasting the limited quantity of lubricious material stored in the coating. Coating architectures that control adaptation rates or selectively inhibit adaptation on coating surfaces unaffected by wear have been developed to increase the lifetime of adaptive tribological materials. Additionally, smart coatings with wear sensing capability have been developed to facilitate coating development and to improve reliability in critical applications.

10:40am **TR4+SE-FrM9 Hard Nanostructured Sulfur-Doped CHx-TiB2 Coatings for Improved Friction and Mechanical Performance**, ***B. Zhao***, *Y.W. Chung*, Northwestern University

Hydrogenated amorphous carbon films are known to attain ultra-low friction performance only in dry environments. Our work demonstrated that sulfur doping of hydrogenated carbon films results in 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 nanolayer or nanocomposite coatings is known to improve hardness. With the aim to produce hard, low-friction coatings, we synthesized nanostructured films of sulfur-doped hydrogenated carbon and titanium diboride using dual-target magnetron sputtering. This paper will discuss the film structure and how such structure correlates with its tribological and mechanical properties.

11:00am **TR4+SE-FrM10 The Relation of Hydrogen to Superlubricity of DLC Films**, ***O.L. Eryilmaz***, *A. Erdemir*, Argonne National Laboratory

In this study, we investigated the critical role of hydrogen in friction and wear mechanisms of hydrogen-free and -poor films in dry nitrogen and high-vacuum environments. Using TOF-SIMS and XPS, we found a very important relationship between friction coefficient and the degree of hydrogenation in these films. Specifically, friction and wear test results showed that the films grown in hydrogen-free or -poor plasmas, the friction coefficients were very high (more than 0.5)and un-steady; and these films wore of very quickly during the sliding tests. However, when these films were subjected to very short-duration (a few minutes) post-hydrogen plasma treatment, their friction coefficients became very low (i.e., less than 0.1) and they were able to last very long during sliding tests. Surface analytical characterizations of the films and sliding contact surfaces were done using XPS and TOF-SIMS as well as Raman Spectroscopy and the chemical and structural findings were correlated with the tribological performance of the films. In particular, TOF-SIMS results revealed very close relationship between surface chemistry and tribological performance of DLC films.

11:20am **TR4+SE-FrM11 Friction and Wear Properties of Nanocrystalline Diamond Coatings**, ***C.C. Baker***, *N.D. Theodore*, Naval Research Laboratory, *T. Feygelson*, GeoCenters Incorporated, *J.E. Butler, K.J. Wahl*, Naval Research Laboratory

The tribological behavior of nanocrystalline diamond (NCD) coatings was studied under both unidirectional and reciprocating sliding conditions. Coatings were deposited by microwave plasma chemical vapor deposition onto Si substrates under varying growth conditions to thicknesses between 1-2 microns. Friction behavior was investigated using pin-on-flat geometry with sapphire counterfaces at average contact pressures of 0.43-0.74 GPa. Wear volumes of the ball counterface and coating wear tracks were determined with optical interferometry. Coating microstructure, chemistry, and surface morphology were examined using X-ray diffraction (XRD), micro-Raman spectroscopy, and atomic force microscopy (AFM). We have found that friction coefficients for the sapphire-NCD sliding interface are low, ranging from 0.03 to 0.1. However, there were large differences in friction run-in, with run-in from high to low friction taking between 50 to as many as 10000 cycles over a wide range of NCD deposition conditions. The role of coating microstructure, bonding chemistry, wear, and roughness of worn and unworn surfaces on run-in friction behavior of NCD will be discussed.

11:40am **TR4+SE-FrM12 Effects of Annealing on Anti-Wear and Anti-Bacteria Behaviors of TaN-Cu Thin Films**, ***J.H. Hsieh***, *M.K. Cheng, Y.K. Chang*, Mingchi University of Technology, Taiwan, *S.H. Chen*, National Chiayi University, Taiwan, *P.C. Liu*, Mingchi University of Technology, Taiwan

TaN–Cu nanocomposite films were deposited by reactive co-sputtering on Si and tool steel substrates. The films were then annealed using RTA (Rapid Thermal Annealing) at 400 ?C for 2, 4, 8 minutes respectively to induce the nucleation and growth of Cu particles in TaN matrix and on film surface. C-AFM (Conductive Atomic Force Microscopy) and FESEM (Field Emission Scanning Electron Microscopy) were used to confirm the emergence of Cu nano-particles on the surface of TaN-Cu thin films. The effects of annealing on anti-wear and anti-bacteria properties of these films were studied. The results reveal that annealing by RTA can cause Cu nano-particles with various dimensions to emerge on the TaN surface. Accordingly, hardness and friction coefficients will change, as well as the anti-bacterial behavior.

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