

## Nanometer Structures

### Room 308 - Session NS-WeA

#### Nanotribology

**Moderator:** R.W. Carpick, University of Wisconsin - Madison

**2:00pm NS-WeA1 MEMS Tribological Coatings and Wear Debris Analysis, S.A. Smallwood**, Air Force Research Laboratory; *K.C. Eapen*, University of Dayton Research Institute; *J.S. Zabinski*, Air Force Research Laboratory  
Among the most significant challenges to developing reliable MEMS that have mechanical components are stiction, friction, and wear. One potential solution to this problem is to protect the devices with coatings, such as monolayers and hard self-lubricating materials. An essential aspect to developing these materials is to understand how surface chemistry, surface forces, wear, and MEMS performance are interrelated. Due to the small size and complex structure of MEMS, it is difficult to develop a fundamental understanding of these phenomena. Wear debris often has dimensions of < 1 mm and it may be hidden under the MEMS structures. In addition, few surface analytical techniques are available to precisely determine tribochemistry and wear mechanisms on MEMS structures. To combat tribological problems, perfluorinated ethers, esters, and mono- and di-alcohols were deposited on MEMS and their chemical/physical properties were studied and related to MEMS performance. AES, microRaman spectroscopy, and IR spectroscopy were used to probe surface chemistry. SPM and surface force measurement systems were used to understand forces at the MEMS scale. A technique to adequately coat complex MEMS structures will be discussed along with the mechanisms responsible for increasing MEMS electrostatic output motor lifetimes by up to three orders of magnitude.

**2:20pm NS-WeA2 Nanotribological Properties of Ultrananocrystalline Diamond, D.S. Grierson**, A.V. Sumant, University of Wisconsin-Madison; *J.E. Gerbi*, *J.A. Carlisle*, *O. Auciello*, Argonne National Laboratory; *R.W. Carpick*, University of Wisconsin-Madison

There is an increasing demand for materials applicable to rolling and sliding contacts in MEMS technology. Ultrananocrystalline diamond (UNCD) films have unique mechanical and tribological properties that exhibit great potential for increasing the reliability of micro and nanoscale devices. Fabrication of MEMS components with UNCD has already been successfully demonstrated, but a detailed understanding of the nanotribological behavior of UNCD surfaces has not yet been established. This study is aimed at probing both the top "as-grown" surface and the back surface (exposed by etching the substrate) of UNCD films. We will explore the effects of hydrogen termination on both surfaces by examining the morphological, adhesive, and frictional properties of the H-terminated vs. untreated UNCD films as a function of load, sliding speed, and relative humidity. Atomic force microscopy is used to examine the nanotribological properties of these surfaces, and XPS, AES, and SEM are used to characterize the surface structure and chemistry. We will discuss the relationships between nanotribological behavior, surface morphology, and surface chemistry of UNCD. These studies could also be of interest for bio-sensing applications involving H-terminated diamond surfaces.

**2:40pm NS-WeA3 Effect of Electric Fields on Nanoscale Friction Properties of Self-assembled Monolayers: Towards Active Friction Control of Interfaces, S. Sundararajan**, K.K. Kanaga Subramanian, P. Hattan, Iowa State University

The ability to actively control friction properties of an interface is of great interest to both nanotechnology and macroscale applications. We present our investigations on the effect of an external electric field on the micro/nanotribological properties of selected self-assembled monolayers (SAMs) using scanning probe microscopy (SPM). It is theorized that an external field would affect the structure of the SAMs, thereby affecting their tribological behavior. The SAMs studied include -CH<sub>3</sub>, -COOH and oligo (ethylene glycol) terminated alkanethiols, and amino terminated silane on Au/Mica due to their well known structures and tribological properties. Micro/nanoscale friction and adhesive forces between the SAMs and a Si@sub3@N@sub4@ tip are studied as a function of applied external AC and DC fields to identify switchable friction states of the SAMs. This study would provide a better understanding of lubrication mechanisms at the molecular level and direct us to realize surfaces for active friction control.

**3:00pm NS-WeA4 Impact of Atomic Corrugation on Sliding Friction as Probed by QCM, T. Coffey**, North Carolina State University; *S.M. Winder*, University of South Carolina; *J. Krim*, North Carolina State University

At the atomic scale, friction is believed to originate primarily via sliding induced excitation of phonons. Theoretical predictions of the magnitude of phononic dissipation have been related to the atomic corrugation of the adsorbate/substrate potential. Using the Quartz Crystal Microbalance (QCM), we have measured the sliding friction of xenon adsorbed at 77 K onto three different surfaces. From lowest to highest atomic corrugation of the adsorbate/substrate potential, the surfaces are: Ag(111), Cu(111), and Ni(111). The QCM probe of sliding friction is the slifetime, which measures the slippage of the adsorbate atop the oscillating surface of the QCM. For monolayer coverages, we observed the following slifetimes: 2 ns for Xe/Ag(111), 1 ns for Xe/Cu(111), and 0.5 ns for Xe/Ni(111). We compare our slifetime results to published values of the atomic corrugation for these systems. We also discuss theoretical predictions for the impact of atomic corrugation on sliding friction. Fundamentals of Friction; Macroscopic and Microscopic Processes, ed. I.L. Singer and H.M. Pollock, Kluwer, Dordrecht (1992). M. Cieplak, E.D. Smith, and M.O. Robbins, Science 265, 1209 (1994). J. Krim and A. Widom, Phys. Rev. B 38, 12184 (1986).

**3:20pm NS-WeA5 Wear Structures on the Nanometer Scale, R. Bennewitz**, E. Gnecco, A. Socoliuc, E. Meyer, University of Basel, Switzerland

Friction force microscopy has been used to study the onset of wear. Abrasive wear could be detected down to single atomic layers at surfaces of ionic crystals. The wear debris was found to perfectly recrystallize in registry with the underlying surface. Furthermore, we have found a characteristic formation of nanometer-scale ripples perpendicular to the direction of scratching. These ripples are built in an interplay between strain-induced erosion, transport by the action of the tip, and diffusion. In a second part, we will present recent results of atomic friction experiments. The relation between atomic stick-slip instabilities and energy dissipation will be discussed based on experiments which exhibit very little dissipation.

**3:40pm NS-WeA6 Tribological Investigation of AlNiCo Decagonal Quasicrystals by UHV AFM/STM, J.Y. Park**, D.F. Ogletree, M. Salmeron, Lawrence Berkeley National Laboratory; *C.J. Jenks*, *P.A. Thiel*, Iowa State University

Tribological properties of the 10-fold and 2-fold surfaces of AlNiCo decagonal quasicrystals have been investigated in ultra-high vacuum using an atomic force microscope with WC-coated conducting tips. Using stiff cantilevers either tunnel current (STM mode) or force (AFM mode) can be used for feedback. The quasicrystal surfaces were cleaned by sputtering and annealing in UHV and characterized with low energy electron diffraction (LEED) and Auger electron spectroscopy (AES). Adhesion and friction of decagonal quasicrystal surface were measured by using contact mode AFM. From force-distance measurements we found that tip-sample adhesion on the clean 2-fold surface is two orders of magnitude higher than that of the air-oxidized surface. Because of this high adhesion, STM contrast was used to image surface morphology. The effects of in situ oxidation and carbon passivation on quasicrystal friction and adhesion will also be discussed.

**4:00pm NS-WeA7 The One-Two Punch: Combining Chemical and Mechanical Stimuli at the Nanometer Size Scale, J.T. Dickinson**, Washington State University

Bond breaking at surfaces due to stimuli such as exposure of materials to electrons, ions, photons, mechanical stress, or chemical agents are well established. We discuss in general the role of multiple stimuli (the "one-two punch") in the degradation and modification of materials and solid surfaces. We then show the nanometer scale consequences of combining localized mechanical stress (due to sliding contact with a scanning force microscope-SFM tip) and exposure to aqueous solutions. These nanotribological methods offer a top-down approach to controlled surface modification. Emphasis will be on results concerning tip induced material removal, recrystallization (at small normal forces), and unique patterning produced by scanning in super-saturated aqueous solutions. We also present results on the influence of an SFM tip in contact with polymers immersed in organic solvents. We show a new method for introducing very small amounts of highly localized patches of chemicals into a polymer surface. Finally, we show new evidence of a "1-2-3 punch", where we introduce radiation as a third stimulus on inorganic crystalline surfaces. The results shown have possible applications in sensors, nanofluidics, and

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optoelectronics. Models will be presented to explain the observed nanometer scale surface modifications.

4:40pm **NS-WeA9 The Wear of NaCl in Humid and Anhydrous Environments**, *P.E. Sheehan, L.J. Whitman*, Naval Research Laboratory

A recent report on the mechanism of dip pen nanolithography (DPN) indicated that wear on NaCl reveals the presence of a residual meniscus even under dry nitrogen. We have studied the wear of a NaCl single crystal by an AFM tip as a function of environmental humidity. In an anhydrous nitrogen environment, we find that the wear mechanism was not meniscus-based but rather mechanical, with the wear rate well-described by a thermal-activation model. By examining the wear track while varying both the temperature and the load, the activation volume and the activation energy could be measured. Introducing a slight amount of water into the anhydrous system dramatically changed the wear mechanism. In this case, salt dissolution and reformation occur, creating islands that are several atomic layers thick with clearly-observable step edges. Interestingly, the morphology of the islands indicates that they are fluid while in contact with the AFM tip and presumably its water meniscus; however, when the AFM tip is quickly removed, the islands solidify and can be imaged by a rapidly moving tip. This observation may lead to a method of patterning NaCl on the nanometer-scale. @FootnoteText@@footnote 1@ Rozhok, S.; Piner, R.; Mirkin, C. A.; J. Phys. Chem. B. (2003) 107 751-757.

5:00pm **NS-WeA10 Nanotribology - Tribochemical Wear of Muscovite Mica in Aqueous Solution**, *J.M. Helt*, The City University of New York College of Staten Island and the Graduate Center; *J.D. Batteas*, National Institute of Standards and Technology

Part I: Atomic force microscopy (AFM) is used to probe defect nucleation, prior to gross wear, of muscovite mica under aqueous environments. Nucleation is found to present itself initially as surface charging due to stress-induced tribochemical scission of the terminating surface bonds. As the surface bonds are continually cleaved, an ensemble of defects contribute to the  $\sim 5.2 \text{ \AA}$  to  $\sim 3 \text{ \AA}$  crystal lattice reconstruction observed in AFM topography and frictional force micrographs. Following lattice restructuring, abstraction of mica surface materials ensues, yielding visible wear scars ranging from  $\sim 2 - 10 \text{ \AA}$  in depth. Environmental [OH<sup>-</sup>] profoundly affects the efficacy of wear events, which is illustrated by the acceleration or inhibition of wear with adjustment of pH under identical load and scan conditions. Part II: In AFM wear trials the area scanned is defined by the length of the slow ( $L_{\text{sscan}}$ ) and fast scan axis. The ratio of  $L_{\text{sscan}}$  to image resolution ( $\text{res}$ , lines/image) defines the magnitude of the line step ( $LS = L_{\text{sscan}}/\text{res}$ ). A contact radius - LS relationship indicates that overlap of successive scans will result if the contact radius - line step ratio (CRLS) is  $< 1/2$ . Consequently, the history associated with a single image scan is not equivalent at various loads due to the contact radius being proportional to the load<sup>1/3</sup>. Theoretical & experimental analysis on muscovite mica with a Si<sub>3</sub>N<sub>4</sub> tip illustrate the effects of scan overlap. The CRLS model, derived from the Hertz contact mechanics theory, shows that scan overlap is considerable. AFM wear trials on mica under pH 5 conditions legitimize this development, with CRLS readily predicting the additional scan history from scan overlap for the mild wear regime. CRLS theory, however, consistently underestimates severe wear trends, which is to be expected in light of Hertzian principles employed.

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