Tribology Focus Topic
Room: C4 - Session TR+SE-WeA

Advances in Surface Engineering for Friction and Wear Control
Moderator: S.S. Perry, University of Florida

2:00pm **TR+SE-WeA1 The Deposition of Highly Adherent Fullerene-Like CN_X Coatings on Steel Substrates of Complex Geometry**, ***E. Broitman***, Linköping University, Sweden & Carnegie Mellon University, *S. Schmidt, G. Greczynski*, Linköping University, Sweden, *Zs. Czigany*, Research Institute for Technical Physics and Materials Science, Hungary, *C. Schiffers*, CemeCon AG, Germany, *L. Hultman*, Linköping University, Sweden

Due to their superior wear resistance, high hardness, and low friction coefficient, carbon nitride (CN_x) coatings have been proposed as a candidate to replace diamond-like carbon (DLC) coatings. In this study we present the structural, morphological, and adhesive properties of fullerene-like (FL) and amorphous carbon nitride (CN_x) coatings synthesized by HIPIMS in an industrial CC-800/9 CemeCon equipment. The coatings were grown on steel substrates of complex geometry (including those with small diameter cavities and holes, and shapes such as bolts, nuts, and screws) to thickness of 2-3 µm. A novel HIPIMS pretreatment with two HIPIMS power supplies was used to increase the adhesion of the coatings: one power supply to establish the discharge and one to produce a pulsed substrate bias. The environment of the created Cr plasma sputter-cleans the surface and forms a Cr-containing gradual interface into the substrate. Subsequently, carbon nitride coatings were prepared by HIPIMS from a high purity graphite target in a N_2/Ar discharge at 3 mTorr with the N_2 fraction varied from 0 to 1, and the substrate temperature varied from ambient to 300 °C. X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), scanning transmission microscopy (STEM), and high resolution transmission electron microscopy (HRTEM) have been used to study the coating and the steel/Cr/CN_x interfaces. Identification of coating adhesion failures was done by the Daimler-Benz Rockwell-C adhesion test.

2:20pm **TR+SE-WeA2 Tribological and Mechanical Properties of Nanostructured Hydrogenated Amorphous Carbon and TiB2 Films**, ***B. Zhao***, *Y.W. Chung*, Northwestern University

Hydrogenated amorphous carbon films (CHx) are known to attain ultra-low friction performance only in dry environments. Our work demonstrated that sulfur doping of hydrogenated carbon films (CHx+S) 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 nanostructured coatings is known to improve hardness. With the aim to produce hard, low-friction coatings, we synthesized nanostructured films of CHx (or CHx+S) and titanium diboride using dual-target magnetron sputtering. Titanium diboride deposited by this method had a hardness >30 GPa. This paper will discuss the film structure as well as tribological and mechanical properties. Nanolayered films with a majority titanium diboride composition showed hardness improvement around rule-of-mixtures values with favorable low friction performance in humid air.

2:40pm **TR+SE-WeA3 Tribological Characteristics of a Tungsten Tip/ Au-Ni Alloy Interface, Studied by Means of Combined STM-QCM**, ***L. Pan***, *J. Krim*, North Carolina State University

A two-phase Au-Ni (20 at.% Ni) alloy has recently been suggested as a potential contact material for RF switch microelectromechanical systems (MEMS) switches. Tribological properties impact switch closure behaviour and heating at the interface impacts both the electrical and mechanical properties of the switch. We have thus performed a QCM STM study of heating and wear at the interface between a tungsten tip and a range of gold nickel alloys with varying compositional percentages.The comparison of nano heating generated via friction and surface response to the tip crash would be shown here.

3:00pm **TR+SE-WeA4 Quantitative Measurements of Adhesion Forces in Polycrystalline Silicon Surfaces via a Doubly Clamped Beam Test Structure**, ***I. Laboriante***, *B. Bush, G. Li, C. Carraro, R. Maboudian*, University of California - Berkeley

Current state of knowledge indicates that the prevalence of static adhesion in microstructures remains one of the major hurdles preventing a larger number of MEMS-based products from entering the mainstream, and quantitative understanding of this phenomenon is currently lacking. This is due to the fact that contact mechanics at the micro-/nanoscale are a complicated multiscale problem, in particular when one is dealing with rough and rigid impacting surfaces. The results of investigations aimed at elucidating the adhesion force between co-planar, impacting polycrystalline silicon surfaces will be presented using a microfabricated doubly clamped cantilever beam test structure. The effect of apparent area of contact will be examined via microfabricated dimples of varying size. Determination of adhesion forces through systematic optical interferometric measurements will be presented. The data reveal a weak dependence of adhesion on the apparent contact area, instead of scaling with the contact area. Possible mechanisms leading to this behavior will be discussed.

4:00pm **TR+SE-WeA7 Nanoscale Control and Understanding of Friction at High-Speeds for Future Disk Drive Head-Disk Interfaces**, ***C.M. Mate***, Hitachi San Jose Research Center **INVITED**

Tremendous progress has been made over the past several decades towards understanding the nanoscale origins of tribological phenomena [1], leading to numerous breakthroughs in friction and wear control. The impact of these breakthroughs has been particularly striking in the disk drive industry, where improved tribological engineering of the head-disk interface has led to the magnetic spacing being reduced from ~100 nm in 1995 to ~10 nm in 2009, enabling a 10^3 increase in storage areal density. In today's drives, recording head sliders fly reliably at incredibly small clearances (~2 nm during read-write operations) for many years at speeds > 10 m/s. As the industry moves to even smaller spacings, however, head-disk contact will become more frequent and improved surface engineering to control friction and wear will become more paramount.

In this talk, I will first review the key surface engineering features (slider air bearing surface, disk topography, overcoat, lubricant, etc.) that enable today's disk drive sliders to fly at nanometer clearances over disk surfaces. I will then describe work going on in our laboratory both to determine the nanoscale origins of friction and to use this understanding to develop future head-disk interfaces that are expected to run in continuous contact.

To determine the nanoscale origins of friction at high-speed sliding contacts (> 1 m/s), we have developed a *high shear rate apparatus* using technology from the disk drive industry [2]. With this new instrument, friction, adhesion, and wear can be studied at high sliding speeds (1 to 100 m/s) for nanometer thick lubricant films sandwiched between atomically smooth surfaces. Recently, this instrument has been modified to incorporate optical microscopy for in-situ visualization when a slider runs in contact with a transparent rotating disk. This technique has been particularly useful for studying the formation of nanoscale, non-equilibrium menisci at high speed contacts.

[1] C.M. Mate, Tribology on the Small Scale: A Bottom up Approach to Friction, Lubrication, and Wear, Oxford University Press, Oxford, 2008.

[2] C. M. Mate, R.N. Payne, Q. Dai, K. Ono, *Phys. Rev. Lett.* 97 (2006) 216104.

4:40pm **TR+SE-WeA9 The Effects of Humidity on the Tribological Properties of Nanocrystalline Diamond**, ***N. Theodore***, *K.J. Wahl*, Naval Research Laboratory

The tribological response of nanocrystalline diamond (NCD) coatings to variations in moisture content of the environment was examined under reciprocating sliding conditions. Surface roughness, structure, composition, and carbon chemistry were determined by atomic force microscopy (AFM), X-ray diffraction (XRD), and Raman spectroscopy. All coatings were nanocrystalline with crystallite sizes ranging from to 4 to 60 nm as measured by XRD. Visible wavelength Raman spectroscopy of the NCD coatings revealed various carbon chemistries. The NCD coatings could be classified in three subsets by Raman microscopy: (1) coatings exhibiting a single strong peak at 1332 cm^{-1} typical of crystalline diamond bonding; (2) coatings possessing broad peaks at 1340 cm^{-1} and 1550 cm^{-1} characteristic of the *D* and *G* peaks in sp^2 hybridized carbon along with the diamond 1332 cm^{-1} peak; and (3) coatings displaying additional peaks at 1135 cm^{-1} and 1470 cm^{-1}, commonly attributed to trans-polyacetylene bonding, along with the three previously described peaks. Reciprocating sliding tests using sapphire counterfaces in controlled humid environments resulted in low friction values for all coatings, between 0.02 and 0.09. Decreasing the humidity caused an increase in the number of cycles to run-in to low friction. These observed differences in run-in and counterface wear as a function of environment will be presented and related to NCD coating composition and microstructure.

5:00pm **TR+SE-WeA10 Crystallography-Dependent Self-Lubrication on Nickel Surfaces During Wear**, ***C.C. Battaile**, S.V. Prasad, J.R. Michael, P.G. Kotula*, Sandia National Laboratories

Wear experiments on Ni surfaces show that stable, nanocrystalline tribofilms can form under appropriate tribological conditions, even on single crystals. The presence of these nanocrystalline layers is qualitatively dependent on the crystallography of the surface and wear orientations, and are responsible for a marked reduction in friction on bare contact, suggesting numerous surface engineering possibilities. For example, when a 1 N normal load and 3.75 mm/s tangential speed are applied to a 1/8" diameter Si3N4 ball in contact with electropolished single-crystal Ni in a dry nitrogen environment, the measured friction coefficient is usually in the range 0.6 to 0.8. However, when the Ni surface is of the {110} type and the sliding direction is <211>, the friction coefficient abruptly drops to 0.3 after about 500 cycles, where it remains indefinitely. Modeling of this phenomenon, based on crystal plasticity, microstructure formation, and grain boundary sliding, suggests that the self-lubrication is due to the capacity of ultra-fine-grained microstructures to support grain rotation. Wear experiments on bulk nanocrystalline Ni deposits support this hypothesis by demonstrating low friction coefficients (around 0.3) and virtually no wear-in under low loads and sliding speeds, and higher friction (around 0.6) under high loads and speeds. We will provide an overview of the experiments and modeling of nanocrystalline film formation on single-crystal Ni, detail the results from friction experiments on bulk nanocrystalline Ni, and discuss model validation of the phenomenon's strain rate sensitivity.

5:20pm **TR+SE-WeA11 NEXAFS Characterization of Vapor Deposited Monolayer and Submonolayer Films on Si and Al_2O_3 for MEMS Friction Control**, ***C. Jaye**, D.A. Fischer,* National Institute of Standards and Technology, *B.M. DeKoven,* Surface and Interface Consulting, *J.D. Chinn*, Integrated Surface Technologies

Most microelectromechanical systems (MEMS) are fabricated using semiconductor and ceramic materials such as Si, and SiO2, and Al2O3 which are hard, brittle materials and are not commonly used for mechanical devices. MEMS components are very small and lack power or much inertia when in motion, so they are highly susceptible to the influence of adhesive and surface forces. MEMS lubrication schemes involving vapor phase lubrication have been proposed as a means of continuously replenishing lubricant films on MEMS surfaces.

We present synchrotron based near edge x-ray absorption fine structure (NEXAFS) spectroscopy results of vapor deposited monolayers and submonolayers on SiO2 and Al2O3 substrates under different process conditions. NEXAFS is a powerful non-destructive method in which soft x-rays are absorbed followed by the excitation (transition) of electrons from a core K- or L-level to partially filled into empty low-lying antibonding molecular states. Bond orientation information is deduced from the changes in the intensity of the resonances upon rotating the substrate normal in the plane of incidence of the polarized synchrotron beam.

Carbon K-shell NEXAFS performed at different incidence angles revealed that the vapor deposited fluorodecyltrichlorosilane (FDTS) molecules on silicon and alumina substrates produced self-assembled monolayered films that have high surface coverage and can be highly oriented. Using the NEXAFS technique, dichroic ratios (based on the σ***C-F** resonance) of the order of 0.5 have been obtained, thus revealing that these FDTS films have a high degree of molecular orientation. Orientation and coverage comparisons for water wiped and isopropylalcohol wiped FDTS as well as directly vapor deposited FDTS will be presented. The implications for the design of surfaces and interfaces for stiction control in MEMS devices will also be discussed.

Authors Index

Bold page numbers indicate the presenter