Tuesday Morning, November 5, 2002

Nanometer Structures

Room: C-207 - Session NS+SE+SS+MM-TuM

Nanotribology

Moderator: K.J. Wahl, Naval Research Laboratory

8:20am NS+SE+SS+MM-TuM1 Ultralow Friction Coatings and Surfaces, J.M. Martin, Ecole Centrale de Lyon, France INVITED From a technological point of view, very low friction in solid lubrication may be interesting in micromechanisms requiring neither friction noise nor instabilities, together with low power consumption. Theoretical approaches at the atomic scale coupled with experimental approaches using proximal probe techniques have been developed to study atomic scale friction behaviors and energy dissipation modes. The two limiting factors for friction reduction at the macro-scale are S0 (shear strength of the interface film) and a (pressure coefficient).¹ Approaching very low friction requires the reduction of both S0 and a below the MPa range. Thus lowering to zero friction would require the vanishing of both the adhesive and the external pressure. However these conditions are unlikely to be perfectly achieved in practice. Thus zero friction may not be possible. However, friction values in the 10-3 range or even less (near-frictionless sliding) have been experimentally reached in some practical situations. Here we examine ultralow friction by using a macro-scale sphere/plane contact configuration (maximum pressure of 1 GPa). Friction in the 0.001 range is associated with a shear strength of 1 MPa. We report experimental evidence of superlow friction with different coatings: pure molybdenum disulfide MoS2,2 molybdenum dithiophosphate (Modtp) tribofilms and hydrogenated diamondlike carbon a-CH.

¹ I. Singer, J. Vac. Sci. Technol., A12(5), (1994) 2605.

²J.M. Martin, C. Donnet, Th Le Mogne and Th Epicier, Physical Review B 48, No 14, (1993) 10583.
³ C. Donnet et al, Surface and Coating Technology, 94, (1997) 456.

9:00am NS+SE+SS+MM-TuM3 Frictional Properties of Small Model Lubricant Molecules Adsorbed on VC(100), L.C. Fernandez-Torres, S.S. Perry, University of Houston, B.-I. Kim, Sandia National Laboratories

The frictional modification of the non polar (100) of vanadium carbide (VC) surface through small molecule adsorption at room temperature has been investigated from a fundamental perspective. These molecules represent the functionalities incorporated into lubricants and used to appropriately tailor the lubricant's properties and enhance its performance. Ultrahigh vacuum atomic force microscopy (AFM) has been employed to determine the changes in frictional response and interfacial adhesion. Scanning tunneling microscopy (STM) has been used to elucidate surface morphology. X-ray photoelectron spectroscopy (XPS) has been utilized to determine the composition of the species formed by the interaction of these adsorbates with the VC surface. This successful methodology has been developed during a recent investigation of ethanol, and in this study has been extended to other low molecular weight alcohols as well as an ester. The results will be rationalized in terms of chemical reactivity, adsorbate layer composition, extent of coverage, and changes in the interfacial shear strength and discussed in terms possible lubrication schemes.

9:20am NS+SE+SS+MM-TuM4 Adhesion and Deformation in Nanoscale Contacts between W(110) and Au(110) in Ultra High Vacuum, S.A. Smallwood, R.J. Lad, W.N. Unertl, University of Maine

Tribological phenomena change as the contact area decreases from macroscopic to atomic dimensions, but these changes are not well understood. We report studies of the force versus deformation behavior of contacts with diameters up to about 50 nm using well-characterized metal surfaces in ultra-high vacuum. These contact sizes are intermediate between those previously studied. The contacting bodies were a Au(110) single crystal and sharp tips of W wires. The W probes were cleaned by field evaporation and their atomic structure determined using field ion microscopy (FIM). All were terminated by (110) planes and radii varied between 12 nm and 24 nm. The probes were mounted in double cross-hair force sensors. After cleaning by sputtering and annealing cycles, the Au was transferred to a piezoelectric tube scanner and moved into tunneling contact with the probe. Deflection of the force sensor and electrical current were measured as the Au crystal was brought into mechanical contact to a predetermined maximum displacement and then withdrawn. Prior to the first yielding event, the data is well described by elastic contact mechanics theory. The reduced modulus of 61 \hat{A} ± 26 GPa agrees with the value calculated assuming bulk properties. The work of adhesion has an upper bound of about 0.3 J/m2. The first observable yielding events occur at a mean normal stress of 12 $\hat{A} \pm 2$ GPa, comparable to the values reported for

larger probes, but half that reported for smaller contacts on Au(111). Hardness is about 6 GPa near the surface and decreases by about fifty percent at 8 nm indentation depth. Prior to first yield, contact conductance remains far below one quantum. Deformation is confined to the Au. FIM demonstrates that the W probe is not deformed for penetrations as deep as its radius. Scanning tunneling microscopy shows that the indentation holes are asymmetric and that pile-up extends about one indentation diameter beyond the indent.

9:40am NS+SE+SS+MM-TuM5 Chemical Force Microscopy of Aluminium Oxide Surfaces, T.T. Foster, M.R. Alexander, UMIST, UK, E. McAlpine, Alcan International, UK, G.J. Leggett, University of Sheffield, UK

The combination of wettability, chemical force microscopy (CFM) and friction force microscopy (FFM) has been used to analyse changes at the oxide-covered surface of aluminium after magnetron sputter deposition. A model self-assembled monolayer (SAM) system was first developed to enable comparisons to be made with the more complex aluminium system. The monolayers were produced by self-assembly on Au (111) and Ag (111) substrates. The gold-coated AFM tips were modified with SAMs of alkanethiols terminated in a methyl or carboxylic acid group. Friction coefficients were measured for SAMs varying in chain length and terminal group chemistry. Using carboxylic acid modified tips; measurements were performed on the surface of aluminium. Adhesion forces were found to decrease with storage time in a desiccated environment, attributed to the adsorption of contaminant molecules from the atmosphere. In contrast the friction coefficient showed no significant change with storage time, presumably because the sliding tip, under loading, is able to displace contaminant molecules. Contact angle goniometry was used to study changes in surface wettability on the aluminium surface. The water contact angle increased linearly with the log of storage time, supporting the hypothesis that adsorption of hydrophobic contaminants modifies the aluminium surface. Contact mode characterisation of the aluminium oxide surface provided clear images of the oxide surface. A nitric acid-based cleaning procedure was developed that was capable of removing adventitious contamination and returning the aluminium oxide surface to condition that appears similar to the freshly deposited surface. This study clearly demonstrates the capability of CFM for characterising complex aluminium surfaces and studying changes in surface chemistry.

10:00am NS+SE+SS+MM-TuM6 Nanotribology and Related Structural Changes During Wear of Diamond-like Carbon Films, J. Goldsmith, E.A. Sutter, J. Moore, B Mishra, Colorado School of Mines, M. Crowder, Maxtor Corporation

Diamond-like carbon (DLC) thin films are used for wear and corrosion protection of magnetic disks, micro-electro-mechanical systems (MEMS), and tool bits. Magnetic information storage density increases when the readwrite head gets closer to the disk. The magnetic layers degrade very quickly without a good protective interface. The use of DLC thin films becomes increasingly popular as they can provide a protective surface due to their excellent tribological properties as low friction and high hardness. In both magnetic disks and MEMS applications, the DLC films are in the thickness range of 2 - 5 nm and in most cases are amorphous in structure. Characterizing the tribological and structural properties and identifying the wear mechanisms of DLC films on the nanoscale is a challenge. Here we present results on the nanotribology of the DLC films performed using both an atomic force microscope and a nanoindenter. We investigate the wear behavior of the DLC films and the role of transfer film. We find that the formation of transfer film plays an important role in providing low-friction. The nanotribological investigations are correlated with the structural changes that occur in the DLC film as well as in the transfer film detected using Raman spectroscopy and cross-sectional transmission electron microscopy.

10:20am NS+SE+SS+MM-TuM7 Tribology and Surface Forces in MEMS, J.S. Zabinski, Air Force Research Laboratory, S.T. Patton, K.C. Eapen, UDRI, S.A. Smallwood, Systran, Inc. INVITED

Microelectromechanical systems (MEMS) offer the potential to provide new capabilities and products for commercial and military applications. Simple devices are already common in the marketplace, but friction, stiction, and wear prevent reliable operation of more sophisticated types of MEMS devices that have contacting surfaces in relative motion. These tribological problems are fundamentally difficult to solve and are magnified because MEMS are expected to operate in very harsh environments, such as at elevated temperature and in space. The performance and reliability of MEMS are strongly dependent on the environment in which they operate. For example, moisture can cause device failure by stiction or it can provide excellent lubrication, depending on the device and the relative concentration of water vapor (i.e., relative humidity). Operation in vacuum is particularly severe and the wear mechanisms are different than in dry or moist environments. Methods to control system tribology include lubricant coatings, monolayers, and new materials. The tribological mechanisms operating in moist air through vacuum will be discussed along with strategies to control friction, stiction, and wear that have significantly improved MEMS reliability. In addition, the effects of storage on device performance will be presented.

11:00am NS+SE+SS+MM-TuM9 Tribological Measurement on MEMS Platforms¹, *M.T. Dugger*, *S.V. Prasad*, Sandia National Laboratories INVITED

Microelectromechanical systems (MEMS) fabricated using surface micromachining (SMM) and other lithographic techniques such as LIGA have resulted in actuators, counter-meshing gears and other moving mechanisms having complex tribological interfaces that are rough on the nanometer scale and have unusual surface morphologies. Meaningful friction and wear measurements of microsystems must be made at loads and speeds relevant to MEMS operation. Since friction and wear are properties of systems, measurements must also involve interactions of surfaces having the morphology and chemistry present in real devices. Experimental techniques for acquiring friction data during sub-micron displacement and under nanoNewton forces are critical for the fundamental understanding of energy dissipation and wear mechanisms in MEMS. However, experimental investigation of surface interactions in MEMS under relevant contact conditions requires techniques beyond those that are currently available. MEMS friction measurement platforms which bring real MEMS surfaces into contact are needed to define the design space, to investigate aging and failure mechanisms, and to validate models of friction and wear derived from fundamental studies. We have therefore developed both SMM and LIGA devices containing isolated tribological contacts from which quantitative friction forces can be extracted. These structures are used to investigate interface performance, degradation and failure mechanisms. Methods of quantifying static and dynamic friction in SMM and LIGA micromachined contacts will be presented. Examples will be shown of how these structures are being used to investigate degradation of monolayer lubricants and hard coatings for SMM devices, as well as the tribological behavior of metallic contacts in LIGA.

¹Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

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