Tuesday Morning, October 26, 1999

Nanometer-scale Science and Technology Division
Room 612 - Session NS1-TuM

Nanomechanics

Moderator: B. Unertl, University of Maine

8:20am **NS1-TuM1 A Progress Report on the Road to Quantitative Nanotribology*, R.W. Carpick,*** Sandia National Laboratories **INVITED**

The goal of routinely acquiring reliable, quantitative nanomechanical and nanotribological measurements with scanning force microscopy (SFM) has not yet been reached. As we travel along the exciting road toward this goal, several signposts warning us of the complications and drawbacks of SFM techniques have been recorded. In this talk I will discuss the current state of affairs regarding accurate measurement of mechanical and tribological properties at the nanometer scale. The critical roles of force calibration, tip characterization, cantilever properties and accurate displacement control will be discussed. Particular attention will be paid to the case of normal and lateral force measurements with contact-mode SFM. I will investigate the application of continuum mechanics models to SFM measurements, including recent models relating fracture mechanics and sliding friction measurements. Finally, I will discuss advantages gained by using novel instrumental approaches. * Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

9:00am **NS1-TuM3 Contact Properties of Oxide Surfaces: Long Range Forces and Adhesion*, E. Barthel,*** *A.S. Huguet, R. Roquigny, S. Sounilhac,* CNRS / Saint Gobain Recherche, France **INVITED**

Surface Forces Microscopy (SFM) is useful to caracterize adhesive properties of surfaces. In practice, however, the usual relations between pull-off force and adhesion energy (JKR, DMT) may sometimes be inadequate, because the assumptions inherent to these models are not fulfilled. Using a general approach to the adhesive contact problem, based on Sneddon's results, and specific descriptions of the interaction(s) relevant to the cases under study, we will consider three non-trivial cases: 1) Silica/silica contact: in this case, both very short-range and long-range interactions contribute. We show how to split the total adhesion energy into its long-range and short-range contributions.@footnote 1@ 2) Surfactant monolayers: recent experiments@footnote 2@ have evidenced a power law dependance of the pull-off force as a function of driving velocity when surfactant monolayers are deposited on the surfaces. We provide an analytical model for this case, where viscoelastic losses are essential 3) Metal/oxide contact: using an UHV AFM, we have investigated the tungsten/oxide adhesion and long range forces.@footnote 3@ Short-range forces are shown to form the main contribution, although we did not achieve quantitative assesment. CONCLUSION Surface Forces measurements are a versatile tool for understanding interaction and adhesion properties of oxide surfaces. However, the bare pull-off force may reflect a variety of phenomena, which have to be properly taken into account in the data treatment. @FootnoteText@ @footnote 1@ Barthel E., Colloids and Surfaces A, 1999, 149, 99. @footnote 2@ Ruth M. and Granick S., Langmuir, 1998, 14, 1804. @footnote 3@ Sounilhac S., Barthel E. and Creuzet F., J. Appl. Phys., 1999, 85, 222.

9:40am **NS1-TuM5 Metallic Adhesion and Tunneling at the Atomic Scale*, A. Schirmeisen,*** *G. Cross, A. Stalder, P. Grutter,* McGill University, Canada*; U. Durig,* IBM Research Division, Zurich Research Laboratory

We have measured forces and currents between atomically defined W(111) tips and a Au(111) sample in ultra high vacuum at 150 K. The W tips are manipulated and characterized on an atomic scale both before and after the experiment by field ion microscopy (FIM). The force-distance curve shows a peak of the attractive, adhesive metallic force of 5 nN for a three atom tip. Unexpected for a metallic system, there is no spontaneous jump-to-contact (Cross et al., PRL 80. 4685 (1998)). An analysis of the tip by FIM after the approach reveals an atomically unchanged tip apex even for repulsive forces of up to several nN. From a fit of our data to the Maugis-Dugdale theory we can determine that our system is close to the rigid body Bradley limit. The experimental data is described very well with a scaled Rydberg function with an unexpected large decay length of 0.2 nm. The simultaneously measured tunneling current has the expected exponential dependence on tip-sample separation, giving a reasonable barrier height of 3.7 eV. After a model proposed by Chen (J. Phys. Cond. Mat 3, 1227 (1991)) there should be a direct correlation between the tunneling current and adhesion force. Using our experimental data we extract a value for the LDOS of the W trimer tip of 1 state/eV/atom at the Fermi energy, which is in agreement with theoretical predictions. Finally, we have measured the evolution of the tip-sample contact for a tip radius of 3 nm and repulsive loadings as large as 200 nN. Half of these measurements show little or no hysteresis, whereas in the remaining indications of reversible slip behavior was observed.

10:20am **NS1-TuM7 Interphase Nanomechanical Properties in a Model Epoxy-Silane-Glass Composite as Revealed by Interfacial Force Microscopy***, H. Cabibil, J.M. White,* University of Texas, Austin*; J.E. Houston,* Sandia National Laboratories*; **R.M. Winter,*** S.D. School of Mines and Technology

The interfacial force microscope (IFM), a scanning probe microscope utilizing a self-balancing differential capacitance force sensor, was used to measure directly the interphase elastic and visco-elastic properties in model epoxy-silane-glass systems. Model composites were fabricated from diglycidyl ether of bisphenol F, diethyltoluenediamine and gamma-aminopropyltrimethoxysilane; an epoxy, amine curing agent, and organosilane respectively and optical silica fibers as the reinforcement and chemical sensor. The elastic modulus was determined directly from the force profiles using a contact mechanics analysis. It was found that the elastic modulus varies significantly with respect to the bulk in a 1-5 micron region surrounding the 50 micron glass fibers. The relaxation and creep response of the interphase was probed to investigate the visco-elastic response of the interphase. An organosilane-epoxy-glass system was also developed to model the interphase region and was probed with the IFM. Visco-elastic analysis of the model interphase yields storage and loss moduli. Fourier transform infrared evanescent wave spectroscopy, utilizing the fibers or a parallelepiped (the model interphase substrate) as both a waveguide and an evanescent sensor, and x-ray photoelectron spectroscopy were used to characterize the bulk and interphase chemistry of the systems. The relationship between interphase chemistry and nanomechanical properties were examined and will be discussed. The portion of this work done at Sandia, which is a multiprogram laboratory operated by Sandia Corporation - a Lockheed Martin Company, was supported by the United States Department of Energy under Contract DE-AC04-94AL85000.

10:40am **NS1-TuM8 Nanoindentation Mechanism and Surface Recovery in an Ionic Crystal Surface, MgO(100)*, P.F.M. Teran Arce,*** *G. Andreu Riera, P. Gorostiza, F. Sanz,* Universidad de Barcelona, Spain

The atomic force microscope can be used to perform nanoindentations on surfaces. With the resulting indentation curves one is enabled to characterize at the nanometer level elastic and strength properties of materials. Furthermore, one is allowed to follow in situ the recovery of the surface after the indentation, therefore gaining insight into the processes affecting the dynamical behavior of the surface. We have carried out nanoindentations, only a few monatomic layers deep, on a semibrittle surface, MgO(100), utilizing an atomic force microscope both for indenting and imaging. Indentations were performed by scanning the piezo vertically until the tip contacts and eventually indents the surface. A force curve was recorded to characterize in situ the indentation. It was found that relative humidity, RH, plays a fundamental role in the kinetics of surface recovery after the indentation. Therefore, in order to avoid the influence of recovery processes in the measurement of cavity dimensions, indentations were carried out at 0 % RH when measuring elastic or strength properties. The force curves obtained show characteristic discontinuities associated with atomic layers being expelled by the tip penetrating the surface. Indentation curves extracted from the force plots show clearly two regions. One of them corresponds to the elastic deformation of the crystal. The other one starts with the onset of discontinuities and corresponds to plastic deformation. The Young modulus and hardness values of MgO(100) obtained from these experiments agree well with the known macroscopic values.

11:00am **NS1-TuM9 The Formation and Evolution of Pileup in Nanoscale Contacts*, K.F. Jarausch,*** North Carolina State University*; J.D. Kiely, J.E. Houston,* Sandia National Laboratories*; P.E. Russell,* North Carolina State University

The interfacial force microscope (IFM) was used to indent and image defect free Au surfaces, providing atomic-scale experimental observations of the onset of pileup. The images and load-displacement measurements demonstrate that elastic accommodation of an indenter is followed by two stages of plasticity. The initial stage is identified by slight deviations of the load-displacement relationship from the predicted elastic response. Images acquired after indentations showing only this first stage of plasticity indicate that these slight load-relaxation events result in permanent deformations 0.25 to 4.0 nm deep with no evidence of pileup or orientation dependence. The second stage is marked by a series of dramatic load-relaxation events and permanent deformations 10-100+ nm deep. Images acquired following this second stage document 0.25 nm high pileup terraces which reflect the crystallography of the surface as well as the indenter geometry. Attempts to plastically displace the indenter 4-10 nm deep into the Au surface were unsuccessful, demonstrating that the transition from stage one to stage two plasticity is associated with overcoming some sort of barrier. Stage one plasticity is consistent with previously reported models of dislocation nucleation. The dramatic load relaxations of stage two plasticity, and the pileup of material above the surface, require cross-slip and appear to reflect a dynamic process leading to dislocation intersection with the surface. The IFM measurements reported here offer new insight into the nucleation, structure and evolution of the dislocations associated with the very early stages of plasticity. This work was supported by the United States Department of Energy under Sandia Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company.

11:20am **NS1-TuM10 Measuring and Imaging Contact Stiffness Quantitatively at the Nanoscale using Force Modulation***, S.A. Syed Asif,*

University of Florida*; K.J. Wahl, R.J. Colton,* Naval Research Laboratory

Depth-sensing nanoindentation has been widely used to measure the nanomechanical properties of materials. However, measuring quantitative mechanical properties of surfaces and thin films on a scale below 10 nm is still a problem. In this presentation we show that combining force modulation with depth-sensing nanoindentation allows measurement of the mechanical properties of materials on the sub-nanometer scale. The stiffness sensitivity of the technique is ~0.1 N/m, which is sufficient to detect long-range surface forces and locate the surface of compliant materials. The tip-surface interaction during approach to contact, asperity deformation during contact and time-dependent deformation at the atomic scale can all be studied. We also present a novel quantitative stiffness imaging technique, which can be used directly to map the mechanical properties of materials with sub-micron lateral resolution. Quantitative stiffness imaging is particularly valuable for polymers, thin films, and other nanostructured materials.

11:40am **NS1-TuM11 SFM Studies of Environmentally Assisted Detachment of Strongly Adhering Particles@footnote 1@***, R.F. Hariadi,*

S.C. Langford, J.T. Dickinson, Washington State University

The production of flat surfaces is often hindered by small particles that adhere strongly to the substrate. We have undertaken a model study of the detachment of sub-micron particles via tribological interactions. Scanning force microscope, silicon nitride tips (typically about 50 nm in diameter) are used to apply stress to 10-100 nm single crystal NaCl crystallites grown on and strongly bonded to soda-lime glass substrates. In most experiments, the applied force is a combination of a compressive normal force and a lateral force, both measured simultaneously. In a time- and spatially-resolved fashion, we apply the stress and measure the response (e.g., detachment). Only the smallest particles can be detached in dry air. However, as the relative humidity (RH) is increased, the crystallites detach at considerably lower stresses. We also observe particle reattachment as a function of time and RH; all reattachments show only a fraction of the original adhesion. We model the detachment process in terms of environmentally enhanced crack growth (due to moisture); when the crack reaches a critical length that depends on particle size, catastrophic interfacial fracture follows. Numerical estimates of the necessary work of adhesion, based on a network of ions interacting via polar forces with the glass, are described. These calculations are in reasonable agreement with our data. @FootnoteText@ @footnote 1@Work supported by the NSF Surface Engineering and Tribology Program under Grant CMS-98-00230.

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