Tuesday Morning, October 3, 2000

Processing at the Nanoscale/NANO 6
Room 302 - Session NS+NANO6+MM-TuM

Nanomechanics
Moderator: W.N. Unertl, University of Maine

8:20am **NS+NANO6+MM-TuM1 Nanomechanical Properties of Molecular Organic Thin Films***, J. Caro,* Institut de Ciencia de Materials de Barcelona (CSIC), Spain*; P. Gorostiza, F. Sanz,* Universitat de Barcelona, Spain*; **J. Fraxedas,*** Institut de Ciencia de Materials de Barcelona (CSIC), Spain

Using Atomic Force Microscopy we have studied the nanomechanical response to nanoindentations of surfaces of highly-oriented molecular organic (MO) thin films (thickness < 1000 nm). Fundamental parameters as the Young' s modulus E, unknown for most MO materials, can be estimated from the elastic deformation using Hertzian mechanics. In the case of the quasi-one-dimensional MO conductor tetrathiafulvalene tetracyanoquinodimethane (TTF-TCNQ) we obtain E ~ 22 GPa, in excellent agreement with previous reported values obtained on single crystals using neutron scattering (E ~ 20 GPa).@footnote 1@ Above ~ 200 nN the surface deforms plastically as evidenced by discrete discontinuities in the indentation curves (~ 1 nm) associated to molecular layers being expelled by the penetrating tip. The estimated critical shear stress @tau@ is ~ 2 GPa. Nanoindentation permits the determination of nanomechanical parameters of MO metastable polymorphs. This is illustrated with the MO radical p-nitrophenyl nitronyl nitroxide (p-NPNN). The @alpha@-phase of p-NPNN, stabilized as thin film,@footnote 2@ exhibits values of E and @tau@ two times smaller than the corresponding values of the thermodynamically most stable @beta@-phase. Measurements were performed with the same tip under the same experimental conditions, thus eliminating the uncertainty associated to the cantilever constant and tip radius. @FootnoteText@@footnote 1@J. P. Pouget et al. Phys. Rev. B 19 (1979) 1792. @footnote 2@J. Fraxedas et al. Europhys. Lett. 48 (1999) 461.

8:40am **NS+NANO6+MM-TuM2 Quantitative Imaging of Dynamic Mechanical Properties by Hybrid Nanoindentation***, **S.A.S. Asif,*** University of Florida*; K.J. Wahl, R.J. Colton,* Naval Research Laboratory

In this paper, we present a novel quantitative stiffness imaging technique and demonstrate its use to directly map the mechanical properties of materials with nm-scale lateral resolution. This is a powerful new approach that can eliminate tedious point-by-point analyses of indentation arrays to obtain quantitative mechanical properties of surfaces. For the experiments, we use a 'hybrid' nanoindenter, coupling depth-sensing nanoindentation with AFM scanning capabilities. AC force modulation electronics have been added, enhancing instrument sensitivity and enabling measurements of time dependent materials properties (e.g. loss modulus and damping coefficient) not readily obtained with DC techniques. Tip-sample interaction stiffness images are acquired by superimposing a small AC force (10's of nN) onto the DC imaging force (1-2 µN), and recording the AC displacement amplitude and phase as the surface is scanned. Combining a dynamic model of the indenter (having known mass, damping coefficient, spring stiffness, resonance frequency and modulation frequency) with the AC response of the tip-surface interaction allows evaluation of complex stiffness maps. We will demonstrate the use of this approach to obtain quantitative loss and storage stiffness images for elastic and viscoelastic surfaces, as well as discuss a method to directly determine loss and storage moduli from the images.

9:00am **NS+NANO6+MM-TuM3 Force-Modulated Nanoindentation of Fluorinated Polymer Thin Films Grown by PECVD***, S.A.S. Asif,* University of Florida*; E.J. Winder, K.K. Gleason,* Massachusetts Institute of Technology*; **K.J. Wahl,*** Naval Research Laboratory

Thin polymer films have been of considerable interest recently in applications for electronics packaging, solid lubrication, MEMS devices, antifouling and adhesives. However, evaluating the mechanical properties of polymer thin films is difficult due to the low elastic moduli and viscoelastic behavior typically observed with polymers. In this paper, we present an approach for measuring the mechanical and dissipative properties of thin, compliant polymer films using AC force-modulated nanoindentation.@footnote 1@ The dynamic response of the indenter is monitored during tip-sample approach, enabling sensitive detection of the surface. Adhesive interactions, contact stiffness and damping are monitored during force-displacement measurements, and hardness and modulus evaluated. In this study, we apply the above approach to investigate the correlation between polymer thin film deposition conditions and the resulting mechanical properties. The thin polymer films were deposited on Si wafers using pulsed plasma-enhanced chemical vapor deposition (PECVD). Two different source gases were used, HFPO (hexafluoropropylene oxide) and HFC-134 (1,1,2,2, tetrafluoroethane); growth conditions were varied by altering the plasma duty cycle during deposition (plasma on-time/plasma off-time). Film thickness was measured by ellipsometry and profilometry, and chemistry examined by XPS and FTIR. Film thickness varied between 100 and 400 nm. Hardness of the films varied between 0.04 to 0.2 GPa, and complex modulus between 2 and 20 GPa, with considerable damping losses observed. Comparisons between the film deposition conditions and resulting chemistry and mechanical properties will be presented and discussed. @FootnoteText@ @footnote 1@ S.A.S. Asif, K.J. Wahl, and R.J. Colton, Rev. Sci. Instrum. 70 (1999) 2408.

9:20am **NS+NANO6+MM-TuM4 Dynamic Contacts to Adhesive Viscoelastic Materials***, **M. Giri,** W.N. Unertl,* University of Maine

Dynamic mechanical contacts with nanometer to micrometer dimensions are important in scanned probe microscopy, ultra-low load indentation, microelectromechanical systems, compact discs, etc. These contacts are poorly understood if they involve adhesive viscoelastic materials such as polymers. We have studied contacts to styrene-butadiene latex films with Tg in the range 253-301 K. Contact times were in the range 0.01-1000 s and loads were up to 1 mN. Nanoindentation was used, rather than scanned force microscopy, because of its well-defined geometry and capability to control the applied load while simultaneously measuring the displacement. Diamond probes with Berkovich and spherical end shapes were used. Load vs. displacement data showed substantial adhesion hysteresis between the loading and unloading portions. The hysteresis is at least partially due to creep as indicated by the continued increase in penetration after the start of unloading. Works of adhesion were estimated by extrapolating the measured pull-off forces to long times as suggested by Johnson.@footnote 1@ Theoretical models that include adhesion but neglect long-range creep effects could not fit the data over an entire loading-unloading cycle. Creep tests were carried out under constant load. The model of Hui, Baney, and Kramer (HBK),@footnote 2@ which predicts the response of an adhesive viscoelastic contact under increasing load, was used to extract a Mode I stress intensity functional. When this functional is normalized by the indentation strain rate, it has a simple universal time-dependence. This result supports the suggestion of HBK that the stress intensity functional may be a simpler alternative to surface energy for characterization of adhesion of viscoelastic materials. @FootnoteText@ @footnote 1@K.L. Johnson in Microstructure and Tribology of Polymers,Eds. V.V. Tsukruk and K.J. Wahl (ACS Books, 2000). @footnote 2@C.Y. Hui, J.M. Baney, E.J. Kramer, Langmuir 14 (1998) 6570.

9:40am **NS+NANO6+MM-TuM5 Precision Nanoscale Machining with STM-QCM***, **J. Krim**, B. Borovsky,* North Carolina State University

We have constructed an apparatus which allows us to investigate the nanoscale machining of metal surfaces resulting from the contact of a sharp tip with a high speed vibrating surface (maximum speeds over 1 m/s).@footnote 1@ The tip (tungsten or platinum alloy) is that of a Scanning Tunneling Microscope (STM), and the surface is that of a metal film deposited onto a Quartz Crystal Microbalance (QCM). The STM-QCM combination enables machining-and-imaging experiments in which the topography of the substrate is compared before, during, and after tip-surface rubbing contact at well-defined locations. The rubbing contact is either direct tip-surface contact or tunneling contact through an oxide layer. While the STM tip alone is able to machine softer materials (such as copper), the high speed vibrations of the QCM greatly enhance machining of more durable materials and oxide films. Specially prepared surfaces permit the creation of sharper, more detailed structures with 10 to 100 nm dimensions, as is demonstrated using copper and silver surfaces exposed to oxygen gas. Our talk focuses on the robustness of resulting structures compared to the ease with which they were machined. Research supported by the NSF and the AFOSR. @FootnoteText@ @footnote 1@ B. Borovsky, B. Mason, and J. Krim, submitted to J. Appl. Phys.

10:00am **NS+NANO6+MM-TuM6 Size-Dependent Mechanical Properties of MoO@sub 3@ Nanoplates***, **J. Wang**, K.C. Rose, J.W. Hutchinson, C.M. Lieber,* Harvard University

The mechanical properties of materials on the nanometer scale are of great interest both for furthering our fundamental understanding as well as for use in a wide range of micro- and nano-mechanical systems. Previous experimental studies have focused on one-dimensional systems, including carbide nanorods and carbon nanotubes. For example, atomic force microscopy (AFM) has been used to show that silicon carbide nanorods have similar Young's moduli to defect free macroscopic crystals and that carbon nanotubes are much stiffer than carbon whiskers and fibers. Here, we used AFM to determine the bending stiffness of individual, structurally-isolated molybdenum oxide (MoO@sub 3@) nanocrystal nanoplates (5-16 nm thick). These nanoplates were pinned to molybdenum disulfide (MoS@sub 2@) surfaces on one side and were suspended freely over MoS@sub 2@ steps on the other side. Bending forces were measured versus displacement on the unpinned side of these MoO@sub 3@ nanoplates. Finite element analysis revealed that the effective Young's moduli of these MoO@sub 3@ nanocrystals are significantly smaller than that of bulk MoO@sub 3@ single crystals and that the moduli decrease with decreasing nanocrystal thickness. This novel behavior was further substantiated in subsequent experiments where it was shown that MoO@sub 3@ nanocrystals (1.4 - 5 nm thick) had enormous flexibility when slid over multilayer MoS@sub 2@ steps. These results have important implications for the sliding of nanoscale structures on rough surfaces and even for the fabrication and manipulation of smaller mechanical systems evolving in nanotechnology.

10:20am **NS+NANO6+MM-TuM7 High Frequency Nanomechanical Systems***, **D.W. Carr,*** Lucent*; L. Sekaric, A. Olkhovets, S. Evoy, J.M. Parpia, H.G. Craighead,* Cornell University **INVITED**

Nanofabricated mechanical systems are highly useful tools for research in physics, optics, and dynamics. We have developed fabrication processes that allow us to make suspended nanostructures in silicon and silicon nitride. We can actuate motion in these structures using electrostatic forces, and this motion is detected optically using interferometric effects. This measurement technique is sensitive to sub-nanometer motion. We have measured doubly-clamped silicon beams with fundamental resonant frequencies as high as 380 MHz. Such structures are being considered for use as chemical and biological sensors, force gauges and frequency filters. One of the obstacles for practical applications are intrinsic losses which lower the mechanical quality factor (Q-factor) of these devices. We see a strong dependence in the Q-factor on the width of these beams. As the width decreases below 100 nm, the Q factor drops sharply, indicating that the dominant energy loss mechanisms are surface related. We are also focusing on surface treatment and the effects of device geometry on dissipation. We have conducted a study of the effects of amorphous metal layers that are used in driving and detection schemes for NEMS and found that the metal layers have a detrimental impact on the devices' mechanical quality factor. We are also studying the effects of various levels of doping in single-crystal silicon on dissipation and driving schemes, a study significant for industrial use in integration with electronic devices. We have also studied the effect of parametric amplification in very small torsional resonators. An applied bias voltage effectively changes the spring constant of the system. Oscillating this bias at a specific frequency results in an amplification of the resonant motion. Swept-frequency measurements show interesting properties of the resonant spectrum, and these results agree well with the theory. Such systems may have interesting application in resonant sensors and surface probes.

11:00am **NS+NANO6+MM-TuM9 Quantum Well Micromechanical Photon Detectors***, **P.G. Datskos,** S. Rajic, L.R. Senesac,* Oak Ridge National Laboratory*; I. Datskou,* Environmental Engineering Group, Inc.

We have developed a method of fabricating quantum well microstructure arrays for a variety of sensing applications. Microstructures with quantum wells allow real-time manipulation of energy states using external stress. For example this can result in an effective and rapid change in electron energy levels in photon detection devices. Such changes make possible tuning the levels to respond to desired wavelengths. We applied such GaAs/GaAlAs micromechanical quantum well arrays to detection of photons and especially uncooled infrared detection. We will present and discuss our results.

11:20am **NS+NANO6+MM-TuM10 Nanomechanical Systems***, **M.L. Roukes**[1],* California Institute of Technology **INVITED**

Microelectronics technology is now pushing deep into the submicron regime, yet, for the most part, work on micromachines still remains back at the few micron scale, or larger. The time is ripe to embark upon a concerted exploration of mechanical systems at the nanoscale. In this talk will highlight the promise and intrigue of this domain. Nanoelectromechanical systems, or NEMS, are MEMS scaled to submicron dimensions. In this size regime, it is possible to attain extremely high fundamental frequencies while simultaneously preserving very high mechanical responsivity (small force constants). This powerful combination of attributes translates directly into high force sensitivity, operability at ultralow power, and the ability to induce usable nonlinearity with very modest control forces. In this overview, I shall provide an introduction to NEMS and will outline several of their exciting initial applications. Our recent efforts at Caltech have culminated in nanomechanical devices with potential for new applications in electronics and metrology. These include development of the first VHF (very high frequency) mechanical resonators;@footnote 1@ the development of mechanical electrometers yielding sensitivity below a single electron charge;@footnote 2@ explorations of thermal transport and energy equilibration in nanoscale devices,@footnote 3@ which have recently culminated in the measurement of the quantum of thermal conductance;@footnote 4@ and development of mechanically-detected magnetic resonance imaging.@footnote 5@ However, a rather stiff entry fee exists at the threshold to this new domain, new engineering is crucial to realizing the full potential of NEMS. Our work also serves to indicate some of the most crucial issues that must be addressed before the full potential of nanomechanical systems can be realized. An important example is that certain mainstays in the methodology of MEMS will, simply, not scale usefully into the regime of NEMS. Most problematic among these issues are the size of the devices compared to their embedding circuitry, their extreme surface-to-volume ratios, and their unconventional "characteristic range of operation". These give rise to some of the principal challenges in developing NEMS. Prominent among these are the need for: ultrasensitive, very high bandwidth displacement transducers; an unprecedented control of surface quality and adsorbates; novel modes of efficient actuation at the nanoscale; and precise, yet robust and reproducible approaches to surface and bulk nanomachining. Ultimately nanomechanical devices will permit access to a regime where mechanics is determined by atomistic properties; where quantum, rather than thermal, fluctuations predominate; where force and displacement detection can meet, or even exceed, the standard quantum limit; and where thermalization involves "granular" heat flow via individual phonons.@footnote 6@ I will conclude by making some projections about this domain that initially seems exotic, but is, in reality, imminent. @FootnoteText@ @footnote 1@ A.N. Cleland and M.L. Roukes, "Fabrication of High Frequency Nanometer Scale Mechanical Resonators from Bulk Si Substrates", Appl. Phys. Lett., 69, 2653 (1996). @footnote 2@ A.N. Cleland and M.L. Roukes, "A Nanometre-Scale Mechanical Electrometer", Nature 392, 160 (1998). @footnote 3@ T.S. Tighe, J.M. Worlock, and M.L. Roukes, "Direct Thermal Conductance Measurements on Suspended Monocrystalline Nanosturctures", Appl. Phys. Lett. 71, 2678 (1997). @footnote 4@ K. Schwab, E.A. Henriksen, J.M. Worlock, and M.L. Roukes, "Measurement of the Quantum of Thermal Conductance", Nature 404, 974 (2000). @footnote 5@ P.C. Hammel, Z. Zhang, G.J. Moore, and M.L. Roukes, "Subsurface Imaging with the Magnetic Resonance Force Microscope", J. Low Temp. Phys. 101, 59 (1995). / P.C. Hammel, Z. Zhang, M.Midzor, M.L. Roukes, P.E. Wigen and J.R. Childress, "The Magnetic Resonance Force Microscope", in "Frontiers in Magnetism of Reduced Dimensional Systems", B.G. Bar'yakhtar et al., eds., (Kluwer Academic, 1998). @footnote 5@ M.L. Roukes, "Yoctocalorimetry: Phonon Counting in Nanostructures", Physica B: Condensed Matter 263-264, 1 (1999).

[1] **Featured Speaker - Science and Technology in the 21st Century**

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