Nanometer-scale Science and Technology Division
Room 321/322/323 - Session NS+AS-WeM

Innovative Force, Near-Field Optics, and Tunneling Measurements

Moderator: H.G. Craighead, Cornell University

8:20am **NS+AS-WeM1 Recent Progress in the Functionalisation of AFM Probes using Electron-Beam Nanolithography***, H. Zhou, G.M. Mills, B.K. Chong, L. Donaldson,* ***J.M.R. Weaver,*** Glasgow University, Scotland **INVITED**

Scanned probe microscopy has greatly expanded the range of contrast mechanisms available to microscopists. Until recently, however, the only techniquees available to the non-specialist user have been those which involve either the modification of the SPM instrumentation (for example Scanning Capacitance Microscopy) or relatively simple functionalisation of the probe (for example Magnetic Force Microscopy). More complex techniques, based on the fabrication of advanced probes, have largely remained confined to a relatively small number of groups. These include the Hall Probe Microscope,@footnote 1@ The scanning Single Electron Transistor@footnote 2@ and others. Recently progress has been made towards methods whereby probes may be modified using batch fabrication techniques such as focussed ion beam deposition,@footnote 3@ controlled etching processes@footnote 4@ or direct-write electron-beam lithography.@footnote 5@ This talk describes recent work in which the last named method has been used to fabricate Near-Field Optical (SNOM), Thermal (SThM) and Magnetic sensors. Results will be presented from SNOM and SThM sensors and progress in sensor technology will also be discussed. @FootnoteText@ @footnote 1@A. Oral et. al. J. Vac. Sci. Technol. B14 (2) p.1202-5 (1996) @footnote 2@M.J. Yoo et. al. Science 276 (5312) p.579-82 (1997) @footnote 3@K. Luo et. al. Appl. Phys. Lett. 71 (12) p.1604-6 (1997) @footnote 4@E. Oesterschulze Appl. Phys. A66, S3-9 (1998) @footnote 5@H. Zhou et. al. J. Vac. Sci. Technol B16 (1) p.54-58 (1998)

9:00am **NS+AS-WeM3 Surface Derivatization of Nanoscale Tungsten Tips for Interfacial Force Microscopy***,* ***K. Griffiths****, P.R. Norton, J.F. Graham, M. Kovar, F. Ogini, O.L. Warren,* University of Western Ontario, Canada

Interfacial force microscopy (IFM) is a novel technique not only for imaging surfaces at resolutions approaching those obtainable with atomic force microscopy, but also for the quantitative determination of the mechanical properties of a material such as elastic modulus, hardness etc., with lateral resolutions of ~nm and depth resolutions ~0.1 nm. The IFM force-compensated sensor permits the acquisition of quantitative force (f) versus distance (d) curves, which through appropriate analysis yield the mechanical properties. Because of the extreme pressures that can be attained in tip-surface contact (many GPa), it is essential to passivate the chemical interactions between the probe tip and the substrate under investigation to prevent strong adhesion effects such as metal-metal bond formation. Studies on Au surfaces are feasible because of the efficacy of self-assembled thiol monolayers on Au. However, convenient and effective protective monolayers are not generally available for many substrates, and it is best to develop a general procedure of passivating the probe tip. Our present studies involve parabaloidal tungsten tips of radii 25<r<200 nm. We have shown that it is possible to use silyl coupling agents (octadecyltrichlorosilane; OTS) to derivatize tungsten surfaces. Using the same techniques we have shown that the nm-scale W-tips can also be derivatized. Measurements were made of the f-d curves for the following tip-substrate couples: underivatized W-tip against underivatized Au(111) surface, underivatized W-tip against derivatized Au(111) surface (C-18 thiol SAM) and derivatized W-tip (OTS) against underivatized Au(111). The data clearly show that the OTS derivatized tips were passivated against adhesive contact even at pressures of many GPa, demonstrating the necessary stability for use in nanoindentation experiments.

9:20am **NS+AS-WeM4 Silicon Cantilevers for Ultrahigh-Density Data Storage***,* ***A. Kikukawa****, H. Koyanagi, K. Etoh, S. Hosaka,* Hitachi Ltd., Japan

In the past few years we have been working on applying atomic force microscopy (AFM) technologies in data storage. One of the most important issues is to increase the data transfer rate (DTR). Thus, it is required to increase the cantilever resonance frequency but keeping the spring constant sufficiently small. Also, an integrated sharp tip is required for reading the small recorded marks. The smallest one we have made so far is an equilateral-triangle cantilever 7 µm long and 0.1 µm thick. Its measured resonance frequency is 6.1 MHz, which is about two magnitudes higher than most of the cantilevers used in AFM, and the calculated spring constant is 0.75 N/m. It was fabricated from a SOI (silicon on insulator) wafer using anisotropic reactive ion etching (RIE) for cantilever shape etching, isotropic RIE for the tip etching, and KOH anisotropic etching for removing excess bulk silicon on the back side and making it a freestanding cantilever. The most difficult part in making such small cantilevers was to control the variation of their dimensions. They are caused mostly by the lateral variation of the wafer thickness and the alignment error (±4 µm at maximum) between the cantilever pattern defined on the active layer and the handling piece pattern defined on the bulk side. We reduced the variation to a sufficient level not by connecting the cantilever directly to the handling piece but by connecting the cantilever via a supporting region sufficiently thicker than the cantilever and whose shape was defined from the cantilever side. We also developed new type of optical lever that can focus the incident beam spot diameter as small as 5 µm and that can be operated with a bandwidth as wide as 10 MHz. From a noise characteristic analysis, the sensitivity of the system was obtained as 4.84 µrad at 10 MHz bandwidth which corresponds to 0.48 Å when a 10 µm long cantilever is used. That is, we now have basic technologies for demonstrating a DTR of 10 Mbps.

9:40am **NS+AS-WeM5 Capacitative Force Modulation Technique in Nanoindentation***,* ***S. Asif****, K.J. Wahl, R.J. Colton,* Naval Research Laboratory*; S.G. Corcoran,* Hysitron, Inc.

The sinusoidal force modulation technique for nanoindentation has been implemented using a three-plate capacitative force/displacement transducer developed by Hysitron, Inc. The force modulation technique can be used to detect the surface of the specimen very accurately with the stiffness sensitivity of 1N/m or less. The low spring mass (243mg), spring stiffness (120N/m) and the low damping coefficient (0.007 Ns/m) of the transducer allows one to measure the damping losses in most of the materials including metals. The experimental results on indium at room temperature indicate that the damping of the material influences the modulus measurement. The technique can be used to measure the loss and storage modulus of polymer materials (e.g. poly(vinylethylene)) and thin film systems. The experimental technique will be described together with the importance of system calibration and specimen mounting.

10:00am **NS+AS-WeM6 Nanoindentation as a Probe of Stress State***,* ***K.F. Jarausch****,* North Carolina State University*; J.D. Kiely, J.E. Houston,* Sandia National Laboratories*; P.E. Russell,* North Carolina State University

A dependence of elastic response on the local stress-state of a material has been demonstrated using the interfacial force microscope (IFM). This investigation was prompted by a previous IFM survey in which the mechanical response of Au thin films was found to correlate with the films' residual stress state and not with morphology or substrate adhesion. In order to better establish the details of this relationship a concentric ring bending device was built to investigate the dependence of IFM nanoindentation measurements on applied tensile and compressive stresses. The measured elastic modulus was shown to increase to 65 +-6MPa with applied compressive stress (50 +-10MPa) and decrease to 32 +-9MPa with applied tensile stress (-50 +-10MPa). The response of the unstressed film was 47 +-6MPa throughout the measurement sequence demonstrating that this change in response is not due to any permanent change in the film. Elastic response was also found to vary as a function of work hardening, indentation position relative to morphological defects, and ion implantation dose. Results from these five experiments will be discussed in terms of possible mechanisms, in an effort to identify how stress alters the measurement process and causes the variation of Au's nano-mechanical properties. These experiments suggest that the IFM has the potential for being able to measure stress state on a very local level. 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.

10:20am **NS+AS-WeM7 Nano-scale Observations of Stress-Enhanced Dissolution in Monoclinic CaHPO@sub4@ 2H@sub 2@O: Chemical vs. Mechanical Effects***,* ***S.C. Langford****, L. Scudiero, J.T. Dickinson,* Washington State University

In several mechanical wear situations, e.g., biomaterials in hip replacements and mechanochemical polishing (used extensively in the microlectronics industry), a surface experiences simultaneous tribological loading and corrosive chemical exposure. The combination can greatly increase wear rates. We examine single crystal brushite [CaHPO@sub 4@ 2H@sub 2@O] (a model biomaterial) in buffered aqueous solutions mechanically stimulated by the tip of a Scanning Force Microscope (SFM). Quantitative data on nanometer-scale wear of single atomic layer steps are readily obtained. The (010) faces of this material are strongly anisotropic, forming trianglular etch pits bounded by three crystallographically distinct steps in aqueous solution. Stress-enhanced dissolution is readily observed along all three steps. On each step, the wear rate is a highly nonlinear (essentially exponential) function of contact force; this function dependence is modeled in terms of stress-enhanced double kink nucleation. At low contact forces, etch pit growth principally involves dissolution along [210] steps; in contrast, the [101] steps are far more vulnerable to wear at high contact forces than the other steps. Damaged regions along [101] steps are especially vulnerable to subsequent chemical dissolution. We exploit this effect to produce atomically flat surfaces many microns in dimension. We also describe the influence of tip velocity and solution chemistry on the rates of corrosive wear. This highly anisotropic material provides a useful system for isolating aspects of the crystal structure which render it vulnerable to chemical etching from those which make it vulnerable to mechanical damage. This work is supported in part by a grant from the National Science Foundation, Grant CMS-9414405.

10:40am **NS+AS-WeM8 Conductance and Force at an Atomically Defined Junction*, G. Cross,*** *A. Schirmeisen, A. Stalder, P. Grütter,* McGill University, Canada*; U. Dürig,* IBM Research Division, Switzerland

We have simultaneously measured conductivity and force between an atomically defined tip and atomically flat sample in UHV. The sharp metal tips are manipulated and characterized on an atomic scale both before and after the sample approach by field ion microscopy (FIM). Conductivity over a large range is obtained by a multidecade nonlinear current amplifier,@footnote 1@ while simultaneously forces between the tip and sample are measured by an in-situ differential interferometer with sub-nN force sensitivity. We report on the conductivity and force vs. tip-sample separation relationships for specific atomic tip geometry. In particular, we have examined the precontact regime characterized by short-ranged attractive forces. In this regime, we find that for a trimer W tip approaching an Au(111) surface, the square of the force depends linearly on conductivity. This can be understood if one assumes that both tunneling and adhesion quantum mechanical exchange interactions are due to overlap of tip and sample wavefunctions.@footnote 2@ @FootnoteText@ @footnote 1@U. Dürig, L. Novotny, B. Michel, A. Stalder , Rev. Sci. Instr. 68, 3814 (1997) @footnote 2@C. Chen, J. Phys. Cond. Matter 3, 1227 (1991)

11:00am **NS+AS-WeM9 Chemical Imaging with Scanning Near Field Infrared Microscopy*, C.A. Michaels,*** National Institute of Standards and Technology, US*; R.R. Cavanagh, S.J. Stranick, L.J. Richter,* National Institute of Standards and Technology

The development of a scanning near field microscope that utilizes infrared absorption as the optical contrast mechanism will be discussed. This instrument couples the nanoscale spatial resolution of a scanned probe with the chemical specificity of vibrational spectroscopy. This combination allows the in situ mapping of chemical functional groups with subwavelength spatial resolution. Key elements of the microscope include; an ultrafast IR light source producing pulses with a FWHM bandwidth of 150 cm@super -1@, an infrared focal plane array based spectrometer allowing parallel detection of the entire pulse bandwidth with 4 cm@super -1@ resolution, and a near field probe fabricated from fluoride glass fiber allowing single mode transmission over the range 2.2 to 4.5 µm. Factors influencing the optical and topographic resolution characteristics of this microscope will be presented. Additionally, the performance of the microscope in discriminating chemical species based on their IR optical properties will also be described.

11:20am **NS+AS-WeM10 Tapping-Mode and Nonoptical Force Sensing Near-Field Scanning Optical Microscopy*, D.P. Tsai,*** *Y.Y. Lu,* National Chung Cheng University, Taiwan

We present a tapping-mode and nonoptical force sensing near-field scanning optical microscopy system. A high Q quartz tuning fork with resonance frequency of 32.768 kHz is used as a force sensing transducer. The piezoeletric current of the tuning fork is lock-in amplified and served as a signal for distance control. Excellent quality of tapping-mode sensing and imaging was obtained. The sensitivity of image is comparable to optical force sensing technique. Results show low background signal and high signal to noise (S/N)ratio for near-field optical contrast, and the elimination of possible optical excitations arising from the force sensing laser light source. Applications on the near-field optical writing and reading on the light sensitive samples show the advantages of this novel method.

11:40am **NS+AS-WeM11 Development and Application of a Dual-Probe Scanning Tunneling Microscope for Nanoscale Investigations of Materials*, H. Grube,*** *M. Allgeier, J.J. Boland,* University of North Carolina, Chapel Hill

Scanning tunneling microscopy has evolved into a valuable tool for the study of the structural and electronic properties of semiconductor and metal surfaces, as well as enabling fabrication of novel nanoscopic electronic devices. However, the single probe geometry of STM limits its application to local and static measurements of the local density of states (LDOS).@footnote 1@ Incorporation of a second electrically and mechanically independent STM tip within 100nm of the first is expected to enable measurements of surface properties that conventional STM cannot perform.@footnote 2,3,4@ To this end our lab has completed construction of one of the first dual probe STMs in which tips can be placed 10-100nm apart. Each tip is mounted on an independent tube scanner with independent piezo drivers, current preamplifiers and feedback controllers. The scanners have two and three degrees of freedom for coarse motion, achieved through the use of modified commercial inertial sliders. These five degrees of freedom allow for the precise positioning of the two probes into overlapping scanning ranges of the tubes. In this DP-STM configuration it is possible to inject a current into the sample at an arbitrary location with one tip and detecting a change of the electrical environment of the sample with the other probe arbitrarily positioned close by. Therefore it is possible to probe the transport properties of the medium or three terminal nanoscale device. Our DP-STM has been characterized by using each tip to scan its local surface environment and then overlaying the images obtained to determine the inter-tip separation. @FootnoteText@ @footnote 1@G. Binnig et al., Phys. Rev. Lett., 49 (1), 57 (1982) @footnote 2@Q. Niu, M.C. Chang and C.K. Shih, Phys. Rev., B 51 (8), 5502 (1995) @footnote 3@J.M. Beyers and M.E. Flatte, Phys. Rev. Lett., 74 (2), 306 (1995) @footnote 4@J.M. Beyers and M.E. Flatte, J. Phys. Chem. Solids., 56 (12), 1701 (1995)

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