Friday Morning, November 8, 2002

Nanometer Structures
Room: C-207 - Session NS-FrM

Novel Surface Nanoprobes
Moderator: S.A. Smallwood, Wright Patterson Air Force Base

8:20am **NS-FrM1 Correlated Spectroscopic and Scanning Probe Microscopy Approaches to Studying Molecular Assemblies at Interfaces**, ***C. Yip***, University of Toronto, Canada **INVITED**

The rational design of protein-based supramolecular architectures requires careful consideration of not only intramolecular structure but also the intermolecular interactions that control their self-association into higher order structures. We are particularly interested in the role of interfacial structure and chemistry in defining the nucleation and growth of these systems and specifically the synthesis of extended two-dimensional protein arrays. While scanning probe microscopy provides an excellent tool for studying these processes in real-time, there is an obvious need for integrated instrumentation that provides correlated analytical capabilities. We report here on the development and implementation of a multi-modal coaxial instrumentation platform that enabled the simultaneous acquisition of spectral, optical, and probe microscopy data. Examples will be drawn from studies of lipid phase transitions and protein assembly into two and three-dimensional architectures.

9:00am **NS-FrM3 ATR Vibrational Spectroscopy Towards Single Molecule Sensitivity and Molecular Level Spatial Resolution**, ***M. Futamata***, National Institute of Advanced Industrial Science and Technology (AIST), Japan **INVITED**

Recent developments in SPM (Scanning Probe Microscopy) or SXS (Surface X-ray Scattering) provide us atomic level information on structural changes at solid/liquid interfaces. However, it is in principle difficult to identify adspecies or to characterize interaction between adspecies and substrates even with these techniques. Vibrational spectroscopy gives valuable information on this point, if inherently low sensitivity is overcome. We have studied highly sensitive ATR (Attenuated Total Reflection)-IR and Raman spectroscopy utilizing surface plasmon polariton (SPP) with higher spatial resolution beyond diffraction limit by combining with SNOM (Scanning Near-field Optical Microscopy). Recently, ATR-IR spectroscopy allowed us to elucidate water molecules at hydrophobic SAM (Self-assembled monolayer)/solution interfaces, whose hydrogen-bond network are completely broken to give a sharp O-H stretching band at quite high-frequency region. In addition, amphiphilic organic nanotubes in solution substituting the water result in multilayer formation of the monomers. On single molecule characterization with micro-SERS (Surface Enhanced Raman Scattering) on Ag nanoparticles, we found the blinking (drastic intensity fluctuation with time) for adenine molecules without using electronic resonance effect. Raman image from aggregated Ag particles shows the parallel polarization to connecting axes gives significantly larger enhancement than perpendicular direction. These results are in good agreement with the theoretical evaluation of the local electric field using FDTD (Finite Difference Time Domain) method. (3) ATR-SNOM Raman spectroscopy utilizing SPP yields the enhancement of Raman signal up to 300 times and enables us to obtain the SNOM-Raman image with ca. 50 nm of spatial resolution.

9:40am **NS-FrM5 Accurate Real-Space Measurements of Surface Lattice Parameters**, ***J.A. Kramar***, *G.M. Witzgall, V.P. Scheuerman*, National Institute of Standards and Technology

At the National Institute of Standards and Technology (NIST), we have built a metrology instrument called the Molecular Measuring Machine (M^3) with the goal of performing nanometer-accuracy two-dimensional feature placement measurements over a 50 mm by 50 mm area. The instrument uses a scanning tunneling microscope to probe the surface topography, and a Michelson interferometer system to measure the lateral probe movement, both having sub-nanometer resolution. The lateral position is servo controlled, based on the interferometer readings, using a digital signal processor. The instrument environment includes temperature control, a vacuum system with a base pressure below 10^{-5} Pa, and seismic and acoustic vibration isolation. Several artifacts have been measured to validate the instrument performance. Initially, an average pitch measurement was made on a grating that was produced by laser-focused atomic deposition of Cr.[1] The average line pitch for this grating was measured to be 212.69 nm, with an estimated fractional standard uncertainty of 25 x 10^{-6}. This estimate was derived from a consideration of the sources of uncertainty for a 1 mm point-to-point measurement, including the effects of interferometer and sample alignments, Abbé errors, motion cross-coupling, and temperature variations. Most recently, M^3 measurements were made of the surface lattice parameters of a conducting organic crystal and compared to the bulk lattice constants as determined by x-ray crystallography. In initial small-area measurements, the lattice constants of nominally 1.02 nm and 750 pm were in agreement to within 70 pm. These data represent a major achievement in performing a direct, real-space measurement of crystal lattice parameters using ultra-high accuracy interferometry.

[1] J.J. McClelland, R.E. Scholten, E.C. Palm, and R.J. Celotta, "Laser Focused Atomic Deposition," Science, Vol. 262, pp. 877-880, 1993.

10:00am **NS-FrM6 Microwave Evanescent Microscope with Coupled Shear-field Topography Measurement**, ***S.W. Robey***, *S.J. Stranick*, National Institute of Standards and Technology

Near-field probes are being developed to combat the diffraction limit in a wide spectral range from the visible through the IR and into the microwave regime. We are developing evanescent capabilities at GHz frequencies for a variety of applications. Two design criteria are broadband capability and the ability to independently measure sample topography. A system based on a coaxial transmission line resonant structure evanescently coupled to the sample via a scanning tunneling microscope tip will be described. Standing wave resonances in the structure provide high sensitivity with quasi-broadband coverage from ~ 1 GHz to 20 GHz. While previous designs have used soft contact or employed the capacitive feedback to provide height control, we have successfully implemented shear-force measurement to provide the necessary independent topographic information on either conducting and insulating materials. A variable single stub mechanism allows tuning to critical coupling at a selected resonance once near-contact is achieved. The design and implementation of the microscope will be discussed, with comparison to other microwave evanescent systems. Measurements on compositionally graded thin films of $Ba_xSr_{1-x}TiO_3$, buried metallic lines on IC's, and investigation of correlations in topographic and dielectric contrast will illustrate capabilities.

10:20am **NS-FrM7 Factors Influencing the Capacitance-Voltage Characteristics Measured by the Scanning Capacitance Microscope**, ***G.H. Buh***, National Institute of Standards and Technology and Seoul Nat'l Univ., Korea, *J.J. Kopanski, J.F. Marchiando, A.G. Birdwell*, National Institute of Standards and Technology, *Y. Kuk*, Seoul National University, Korea

The scanning capacitance microscope (SCM) can be used to measure two-dimensional dopant profiles in semiconductors with nanometer scale resolution. Dopant concentration information is extracted from the local capacitance-voltage (C-V) characteristics measured between the SCM tip and the semiconductor sample. Two important artifact effects on C-V curves measured via SCM are discussed. It is found that the stray light from the laser of the atomic force microscope (AFM) dramatically affects the measured C-V curve. The difference between the usual SCM C-V curves measured under this high stray light condition and SCM C-V curves measured in a true dark condition will be shown and discussed. The distortion of C-V curves caused by the lock-in modulation voltage will also be discussed. After reducing these effects, SCM C-V curves are obtained that show markedly different behavior from that of conventional one-dimensional C-V curves. These measured C-V curves have a much stretched-out shape and non-zero dC/dV signals in the depletion and inversion region. Measured C-V curves are compared with three-dimensional calculations of the capacitance between the tip and sample. Determination of the dopant density directly from SCM C-V curves will be discussed. Finally, we will discuss optimal SCM imaging conditions, which overcome effects from surface charge and work function variation, and produce more accurate dopant profile measurements.

10:40am **NS-FrM8 Two-Dimensional Dopant Profiling by Novel Scanning Capacitance Force Microscopy**, ***K. Kobayashi***, *K. Kimura, H. Yamada, K. Matsushige*, Kyoto University, Japan

We have newly developed scanning capacitance force microscopy (SCFM), which is capable of mapping local differential capacitance (dC/dV) without external capacitance sensors, based on electrical force detection. While an electric field alternating at a fixed frequency (f) is applied between a tip and a sample, an induced electrostatic force (ESF) oscillating at its third harmonic frequency (3f) is detected using a lock-in amplifier (LIA). Because the magnitude of the induced ESF is proportional to the product of the square of the applied electric field and the capacitance of the sample (C) which can be modulated at f by the applied electric field especially in

semiconductors, the amplitude and the phase of the induced ESF alternating at (3f) contain information on the differential capacitance (dC/dV) of the sample. SCFM works both in contact mode and dynamic mode. Since the sensitivity of SCFM is inherently high owing to the extremely high force sensitivity in scanning force microscopy (SFM), SCFM can be a high-resolution dopant-profiling technique for semiconducting samples. For demonstration of SCFM, a silicon test sample having several microfabricated patterns of p-type, n-type and heavily-doped n-type regions was imaged. Moreover, we demonstrated that SCFM could be also an important analytical tool for high-resolution characterization of ferroelectric domains in ferroelectric material such as a ferroelectric copolymer thin film.

11:00am **NS-FrM9 Imaging Subsurface Reflection Phase with Quantized Electrons**, *I.B. Altfeder, V. Narayanamurti,* ***D. Chen***, Harvard University

Lead quantum wells (QW) epitaxially grown on annealed Pb/Si(111) interface form a model system for the study of interactions between quantized electrons and adiabatically modulated boundaries. Tunnel spectra of this system reveal a previously unknown adiabatic shift of QW resonances due to lateral variations of the electronic reflection phase at the buried interface. With this effect, lateral distribution of the subsurface reflection phase can be probed, using scanning tunneling microscopy.

I. B. Altfeder, V. Narayanamurti, and D. M. Chen, Phys. Rev. Lett. 88, 206801 (2002).

11:20am **NS-FrM10 Spectroscopic Scanning Tunneling Microscopy Using Semiconductor Tips with Engineered Electronic Structure**, ***P.W. Sutter***, *J.S. Palmer, P. Zahl, E.A. Sutter,* Colorado School of Mines

III-V semiconductors and heterostructures are proposed as a new class of materials for use as probe tips for scanning tunneling microscopy (STM). Compared to the metal tips used conventionally, semiconductors with carefully tuned electronic properties have the potential to significantly increase energy resolution and contrast in spectroscopic STM, particularly in emerging applications such as single molecule vibrational microscopy and spectroscopy.[1] We are exploring InAs as a candidate probe material for spectroscopic STM with ultrahigh energy resolution. Using cleaved InAs probes, we demonstrate atomic-resolution STM imaging on highly oriented pyrolithic graphite (HOPG) and on clean semiconductor surfaces, such as Si(111) 7x7. Tunneling spectroscopy with InAs probes on these materials shows clear signatures of the band structure of the semiconductor tips in local conductance spectra. Routes are studied to adjust the Fermi-level in semiconductor tip materials, thus creating a tunneling distribution that is tunable and significantly narrower than that obtainable with a conventional metal tip.

[1] B.C. Stipe, M.A. Rezaei, and W. Ho, Science 280, 1732 (1998).

11:40am **NS-FrM11 First SEM, SAM and Combined SEM/STM Results of a Novel UHV Compatible Electron Column with Sub 3 nm Resolution**, ***J. Westermann***, *M. Maier, G. Schaefer,* OMICRON NanoTechnology GmbH, Germany, *J. Bihr,* LEO Elektronenmikroskopie GmbH, Germany, *J. Zach,* CEOS GmbH, Germany, *T. Berghaus,* OMICRON NanoTechnology GmbH, Germany

Scanning Electron Microscopy has been a proven tool for a huge variety of scientific applications for decades. Recently, new challenging applications are emerging from the fields of Semiconductor- and Nanotechnology. A key issue for these applications is the non-destructive imaging of the typically very sensitive, small and thin structures with nanoscale dimensions, as well as the characterisation of their chemical composition and electrical properties. Here, we present electron optical concepts and first results of a true UHV compatible version of an SEM column designed to meet the new requirements of ultra low outgassing, low beam voltages, and high resolution with high beam currents. Performance checks on nanostructured samples prove an ultimate lateral resolution below 3 nm at 15 keV beam energy and still below 5 nm at 3 keV at a working distance being compatible with electron energy analysers, and sample currents being suitable for Auger electron analysis. Beam currents in the nA range can be achieved with spot sizes below 10 nm at 1kV beam energy, thus enabling to use this column as an excitation source for chemical characterisation with ultimate spatial resolution in Scanning Auger Microscopy. First static Auger and SAM results demonstrating the outstanding spatial resolution will be shown. Furthermore, we report on the combination of this SEM column with simultaneous Scanning Probe Microscopy (SPM). This combination allows a continuous zoom from mm scale down to the atomic level on the same sample position, precise positioning of the SPM probe, as well as electrical contacting of nanosized structures (e.g. nanotubes or semiconductor devices).

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