Nanometer-scale Science and Technology
Room 2016 - Session NS-MoM

Nanoscale Imaging Techniques

Moderator: M.C. Hersam, Northwestern University

8:00am **NS-MoM1 A Novel Scanning Probe Microscope with MOS Transistor and Nano Tip***, **S.H. Lee**, G. Lim, W. Moon,* Pohang University of Science and Technology, Republic of Korea

A novel probe with the MOS transistor and the nano tip is fabricated for the SPM (Scanning Probe Microscope). The probe measures the electric field with high-aspect-ratio nano tip compared with the previous ones. It can be fabricated by the common semiconductor process and the FIB (Focused Ion Beam) process, and the resulting device can rapidly detect the electric signal with a high sensitivity. The SPMs have been developed and applied to various fields including the nano-scale measurement.@footnote 1,2@ However, sometimes the limited scanning speed and the requirement of additional equipments may become the major obstacles to such an application as data storage.@footnote 2@ To overcome these difficulties, the depletion phenomenon has been reported for the probes, but the resolution of tens or hundreds of nanometers is not solved.@footnote 3@ In this paper, we integrate the MOS transistor and the nano tip into a micro cantilever for maximizing electric signals from a localized region. Since the MOS transistor has high working frequency, the sensing speed cannot be a problem. The sensitivity is also high, and no bulky device such as lock-in-amplifier is required. Moreover, the nano tip by FIB has nanometer scale tip radius, and the resolution is very high. Therefore, the probe may increase various kinds of applications of the SPM technology since it can rapidly detect small localized charges with high sensitivity and high resolution. The device properties are investigated with the various electric signals. The measured data represent the well-established electric properties of the device, and it shows the promising aspect of the local electric field detection with high sensitivity and high resolution. @FootnoteText@ @footnote 1@ H. K. Wickramasinghe, Acta Mater., Vol. 48, No. 1, pp. 347-358, 2000@footnote 2@ M. Lutwyche et al., Sensors and Actuators, Vol. 73, pp. 89-94, 1999@footnote 3@ H. Park et al., App. Phys. Lett., Vol. 84, No. 10, pp. 1734-1736, 2004.

8:20am **NS-MoM2 Simulation of Electrostatic Force Microscopy Imaging Modes***, **D.F. Ogletree**,* Lawrence Berkeley National Laboratory

Non-contact electrostatic imaging is an important mode of operation for scanning force microscopy (SFM). Electrostatic SFM has been used to image ultrathin liquid films, static charge distributions on insulating surfaces, spreading of liquid-crystal films, solvation of surface ions and surface potential variations of self-assembled molecular films, to list a few applications. For dynamic force microscopy in vacuum, it is important to minimize electrostatic interactions by nulling the tip-sample contact potential difference to better resolve atomic-scale structure. Various modes of SFM operation for non-contact electrostatic imaging are possible. One can drive the cantilever on or off resonance by applying an oscillating potential between the cantilever tip and the sample. One can combine off-resonance electrostatic excitation with on-resonance mechanical excitation. One can adjust the DC offset of the tip-sample potential to null the contact potential difference (Kelvin Probe operation). A report comparing experimental results for different electrostatic imaging modes has recently been published.@footnote 1@ The results of numerical simulations of the non-linear tip-sample interaction for the various electrostatic operation modes will be presented. The simulations include the cantilever dynamics and the response of the signal detection electronics, in addition to the tip-sample force. The advantages of the different operation modes will be analyzed, and the resolution limits for contact potential imaging will be discussed. @FootnoteText@ @footnote 1@ M. Luna, D. F. Ogletree and M. Salmeron, Nanotechnolgy 16, S1-7 (2005).

8:40am **NS-MoM3 Chemical Specificity and Defect Characterization on MgO(001)***, **O.H. Pakarinen**,* Helsinki University of Technology, Finland*; A. Ishiyama,* Osaka University, Japan*; A.S. Foster,* Helsinki University of Technology, Finland*; N. Oyabu, M. Abe, O. Custance,* Osaka University, Japan*; R.M. Nieminen,* Helsinki University of Technology, Finland*; S. Morita,* Osaka University, Japan

MgO remains one of the most technologically essential surfaces due to its importance in catalysis. It has also been the subject of intense recent research as a substrate in nanocatalysis. Oxygen vacancies (F-centers) on the surface have been proposed to play an important role as nucleating centres for catalytic nanoparticles. Dynamic force microscopy (DFM) has the potential to image both the surface and any defects or adsorbates in atomic resolution, providing unprecedented details into surface processes. In this work we compare low temperature atomic resolution DFM imaging of the MgO (001) surface in UHV with first principles simulations of the tip-surface interaction. The comparison between theory and experiment is made possible by experimentally obtained site-specific force spectroscopy, which can be directly compared to computational predictions of the short range interaction with different tip models, therefore leading to unambiguous characterization of surface species and point defects. Tips are nanofabricated from silicon, but during DFM imaging, material is exchanged with the surface as well as with the ambient. Hence, we construct a set of probable tip models including adsorbates such as hydroxyl groups, hydrogen, magnesium and oxygen, as well as clean silicon tips. After testing imaging of a perfect area of the MgO(001) surface with two dozen most probable tip models, we establish a best fit to experiment and further simulate imaging of the characteristic defects seen on the surface in experiments.

9:00am **NS-MoM4 QPlus Sensor AFM at Low Temperatures with Atomic Resolution on NaCl***, A. Bettac, **M. Maier**, M. Wittmann, A. Feltz, T. Berghaus,* Omicron NanoTechnology GmbH, Germany

Over many years, low temperature STM has been established as an advanced imaging and spectroscopy tool in various scientific fields. However, the creation and investigation of nano-structures on insulating surfaces gains more and more interest an thus push AFM as an alternative and complementary imaging technique. Ideally, the used AFM probe should simultaneously or alternatively work in STM and STS modes. Based on a proven LT STM platform, we have integrated a Qplus@footnote 1,2@ sensor for atomic resolution AFM while maintaining ease of use and level of STM performance. Especially at low temperatures and related spatial constraints, this self-sensing AFM technique is an ideal alternative to cantilever based optical detection. The QPlus sensor@footnote 1,2@ employs a quartz tuning fork for force detection in non-contact AFM operation mode. One prong of the tuning fork is fixed while the SPM probe tip is mounted to the second prong. It thus acts as a quartz lever transforming it's oscillation into an electrical signal as a result of the piezo-electric effect. The feedback signal is based on frequency shift originating from tip-sample force interaction. A dedicated pre-amplification technique ensures distance control based on the pure vibrational signal. In addition, extremely low signals require the first amplification stage to be very close to the sensor, i.e. to be compatible with low temperatures. Measurements on Si(111) 7x7 show that tunnelling current and vibrational signal are clearly separated. In addition, benchmark measurements on NaCl with a typical corrugation of approx. 10pm prove that resolution on insulation samples is competitive to best cantilever based AFM results. @FootnoteText@ @footnote 1@ Franz J. Giessibl, Appl. Phys. Lett. 73, 3956 (1998)@footnote 2@ Franz J. Giessibl, Appl. Phys. Lett. 76, 1470 (1998).

9:20am **NS-MoM5 Nanoscale Spectroscopy with Optical Antennas***, **L. Novotny**, N. Anderson, P. Bharadwaj,* University of Rochester **INVITED**

In optics, lenses and mirrors are used to redirect the wavefronts of propagating optical radiation. But because of diffraction, propagating radiation cannot be localized to dimensions much smaller than the optical wavelength. Borrowing concepts developed in the radiowave and microwave regime, we use antennas to localize optical radiation to length-scales much smaller than the wavelength of light. We place a laser-irradiated optical antenna, such as a bare metal tip, a few nanometers above a sample surface in order to establish a localized optical interaction and a spectroscopic response (fluorescence, absorption, Raman scattering, ..). A high-resolution, hyperspectral image of the sample surface is recorded by raster-scanning the antenna pixel-by-pixel over the sample surface and acquiring a spectrum for each image pixel. This type of near-field optical spectroscopy has been applied to map out phonons and excitons in individual single-walled carbon nanotubes (SWNT) with a resolution of 10nm. The method is able to resolve defects in the tube structure as well as interactions with the local environment. The proximity of the antenna influences the local light-matter interaction and affects the selection rules, the quantum yield, and momentum conservation. Using the fluorescence from a single molecule we are investigating these effects and we characterized the trade-off between fluorescence enhancement and fluorescence quenching as a function of the separation between the antenna and the molecule.

10:20am **NS-MoM8 Ultra High-Stability SPM to Study Molecules at Variable Temperature***, A.V. Belyayev, S.A. Saunin,* NTMDT, Russia*; **Y.A. Bobrov**,* Nanotechnologies America, Inc.

AFM experiments are subject to noise, image distortion, and long term drift caused by environmental temperature variation, creep, and mechanical and acoustic vibrations. A newly designed AFM system significantly reduces thermal and vibration sensitivity. Capable of imaging in contact and non contact modes the new system is available in two versions: the low temperature (thermal range from -300 C to 800 C) or the high temperature versions (from ambient to 2000 C). Both versions exhibit thermal drift less then 15 nm/C in XY and 10 nm/C in Z direction and temporal drift in X, Y and Z direction less then 3 nm per hour. These extremely low thermal drifts and noise parameters significantly improve image quality, even during long term experiments (force spectroscopy, thermal properties of polymers, lithography, etc). A key feature of this design is a copper cell which encloses the sample and cantilever. The two are heated simultaneously and kept at the same temperature within 0.0050C during all measurements. The special triangular design further minimizes drift caused by thermal expansion and its asymmetry. Minimizing air flow around the cantilever, coupled with small temperature gradients, significantly reduces noise compared to conventional open configuration. Designed for 10x10x5micron â?oscan-by-sampleâ?� imaging, the scanner is equipped with special low-noise capacitance sensors that enable atomic lattice resolution even in closed-loop scanning mode. Additionally, its high resonant frequencies significantly reduce sensitivity to external mechanical and acoustic vibration. The unmatched properties of this device are illustrated with several examples.

10:40am **NS-MoM9 Mapping Atomic-Scale Interaction Potentials***, **A. Schirmeisen**, D. Weiner, H. Fuchs,* University of Muenster, Germany

On the atomic level even a perfectly flat surface is not structureless but reflects the electronic structure of the underlying molecular lattice. This potential energy landscape governs molecule adsorption and diffusion,@footnote 1@ which is responsible for a multitude of dynamic surface processes like crystal growth, vicinal to faceting transitions and catalytic action. However, direct experimental access to the spatial variation of the interaction potential energy at the atomic level is difficult. We use an ultrahigh vacuum atomic force microscope in non-contact mode to scan the three-dimensional force field@footnote 2@ of a NaCl (100) surface at room temperature. The interaction forces are systematically probed over a wide range of tip-sample distances, from attractive to strongly repulsive forces. The surface was imaged before and after the 3D spectroscopy experiments with atomic resolution, including single atom defects, thus ensuring an unmodified single atom terminated tip apex. From the force spectroscopy measurements on a predefined grid on the surface we obtain a quantitative characterization of the atomic scale potential energy landscape.@footnote 3@ The energy diagram allows us to identify distinct energy minima at the site of one ion species and extract the effective energy barrier between two adjacent minima. This barrier is directly linked to the dynamics of adsorbed surface atoms and atomic scale friction processes. Furthermore we calculate from the energy landscape the vertical as well as lateral tip-sample forces of the single atom contact. We observe the emergence of a mechanical relaxation process of the investigated single atom contact, which shows a reversible yet hysteretic characteristic. @FootnoteText@ @footnote 1@ R. Gomer, Rep. Prog. Phys. 53, 917 (1990)@footnote 2@ H. Hoelscher et al., Appl. Phys. Lett. 81, 4428 (2002)@footnote 3@ A. Schirmeisen, D. Weiner, H. Fuchs, submitted (2006) .

11:00am **NS-MoM10 Electromechanical Imaging of Ferroelectric Materials in a Liquid Environment: Ultrahigh Resolution and Novel Physics***, B.J. Rodriguez, S. Jesse, A.P. Baddorf, **S.V. Kalinin**,* Oak Ridge National Laboratory*; B. Mirman,* Suffolk University*; E.A. Eliseev, A.N. Morozovska,* National Academy of Science of Ukraine

High resolution imaging of ferroelectric materials is demonstrated using piezoresponse force microscopy (PFM) in an aqueous environment. In the last decade, PFM has been established as a powerful tool for nanoscale imaging, spectroscopy, domain patterning and lithography of ferroelectric thin films, as well as the characterization of capacitors used for ferroelectric memories and data storage. Recent work has demonstrated the applicability of PFM to biological systems where it is possible to image structural properties and molecular orientation with a sub-10 nm resolution. The primary factors limiting the resolution and sensitivity of PFM are electrostatic contributions to the signal and capillary forces. Here, we performed PFM in an aqueous environment to simultaneously minimize both the electrostatic and capillary interactions. A resolution on the order of 1-3 nm, approaching the theoretical domain wall width, as compared to a resolution of ~30 nm in ambient, is reported. The dynamic behavior of the cantilever was analyzed using conventional amplitude-frequency and 2D amplitude-frequency-bias spectroscopy. It is shown that the cantilever dynamics in liquid are significantly different from ambient conditions due to the higher viscosity and added mass effects. Imaging at frequencies corresponding to high-order cantilever resonances is shown to minimize these effect thus allowing sensitivities comparable to ambient conditions. The absence of both long-range electrostatic forces and capillary interactions results in the localization of the ac field to the tip-surface junction and allows the tip-surface contact area to be controlled. This unusual mechanism enables spatial resolutions approaching the intrinsic domain wall width. PFM in liquids will provide novel opportunities for high-resolution studies of ferroelectric materials, imaging of soft polymer materials, and the study of biological systems in physiological environments on, ultimately, the molecular level.

11:20am **NS-MoM11 Spatial Resolution, Information Limit, and Contrast Transfer in Scanning Probe Microscopy***, S.V. Kalinin, **A.Y. Borisevich**, V. Meunier, S. Dag, S. Jesse, B.J. Rodriguez, S.J. Pennycook,* Oak Ridge National Laboratory

The development of scanning probe microscopy (SPM) techniques in the last two decades has provided a unique set of tools for high and ultimately atomic resolution imaging of surface structure and properties. Despite the two-decade long history of the field, there still exists no universally accepted definition of spatial resolution in SPM. Here we illustrate that for several broad classes of SPMs, including MFM, EFM and KPFM, and STM, the image can be represented as a linear convolution of an ideal image representing sample properties and a microscope resolution function. The Fourier transform of the latter, the object transfer function (OTF), describes the transmission of the frequency components of the object to the experimental image. For techniques with a monotonic OTF, two characteristic lengths can be unambiguously defined - the Raleigh resolution and the information limit. Raleigh two point resolution establishes the conditions necessary for quantitative measurements of local properties and is related to the full width of the OTF at half maximum. The information limit of the technique determines the minimal feature size that can be detected and is limited by the noise level of the system. These concepts are applied to piezoresponse force microscopy and STM. For PFM, the resolution and information limits are determined from the domain wall profiles and from written periodic domain patterns. For STM, several surfaces with well-known structures, including Si (111) with 7x7 reconstruction and In/Si, are investigated. The dependence of the information limit on tip bias and the effect of non-rotationally invariant tip states on imaging are demonstrated both experimentally and theoretically.

11:40am **NS-MoM12 Raman Spectroscopy of Strained Silicon Structures for CMOS Technology***, **M. Hecker**, C. Georgi, A. Mai, L. Zhu, E. Zschech,* AMD Saxony LLC & Co. KG Dresden, Germany

Straining the active regions in silicon CMOS devices is one of the key contributors to increase device performance in present and future technology nodes. Since dedicated strain on the transistor level with opposite sign is required for NMOS and PMOS transistors, the need to measure strain on a local scale has become a challenge for metrology. Raman spectroscopy has the potential to obtain strain information non-destructively on the sub-µm scale, and therefore, this technique is evaluated for process monitoring. In this paper it will be shown for silicon-germanium thin films, how the problem of the interfering influence of strain and composition on Raman peak shifts can be overcome by measuring independent phonon modes. The obtained strain and composition parameters will be compared with data from other techniques. The lateral resolution of the method can be improved by application of an appropriate aperture close to the sample surface, or by apertureless approaches utilizing tip-enhanced Raman scattering (TERS) at nanoparticles or metallized AFM tips brought into the laser beam. Results of both approaches are discussed and evaluated.

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