Thursday Morning, November 6, 2003

Nanometer Structures
Room 308 - Session NS-ThM

Advances in Scanning Probes

Moderator: R. Bennewitz, University of Basel, Switzerland

8:20am **NS-ThM1 Autonomous Atom Assembly*, *R.J. Celotta,*** *J.A. Stroscio, A.P. Fein, S.R. Blankenship, A. Lacaze, J. Cugini,* National Institute of Standards and Technology

The ability to use an STM to move and position atoms with lattice site precision provides us with a quantum workbench to study the effects of quantum confinement and the electronic structure of perfect nanostructures. So far, atomic manipulation has been performed manually, or with rudimentary computer assistance. In this talk, we describe an Autonomous Atom Assembler (AAA), which is an instrument capable of assembling a desired nanostructure from an unknown random collection of atoms without human intervention. It is based on an existing low temperature STM system, with hardware and software extensions. In operation, a dilute coverage of adsorbate atoms is deposited on a clean, flat substrate. The AAA then instructs the STM to image the random adsorbate pattern, identifies the position of each adsorbate atom relative to the substrate lattice, and plans a series of moves to place the available atoms at positions specified in a previously entered diagram. The plan minimizes construction time, follows a set of extensible rules, and allows for error correction. The AAA then instructs the STM to execute the plan and, upon completion, provide an image of the final assembled nanostructure. In our initial trials, Co atoms were deposited to a coverage of 0.003 monolayer on a Cu(111) substrate initially held at 7 K in UHV. Subsequent STM measurements were performed at a 4.3 K sample temperature. Simple confinement structures were autonomously assembled involving tens of atoms. We will report our first results, including the accuracy of positioning, speed of operation, reliability, and scalability of our design. * Supported in part by the Office of Naval Research

8:40am **NS-ThM2 Combined Scanning Force Microscope and Mass Spectrometer*, A. Wetzel,*** University of Basel, Switzerland*; D.-W. Lee,* IBM Research, Zurich Research Laboratory, Switzerland*; R. Bennewitz,* University of Basel, Switzerland*; M. Despont, P. Vettiger,* IBM Research, Zurich Research Laboratory, Switzerland*; Ch. Gerber,* University of Basel, Swizterland*; E. Meyer,* University of Basel, Switzerland

We have constructed a scanning force microscope in ultrahigh-vacuum where the tip position can be switched between the sample surface and a local electrode that serves as entrance for a time-of-flight mass spectrometer. Material shall be picked up at the surface and chemically identified in the mass spectrometer after field- induced desorption from the tip. Tip, force sensor, switch, and local electrode are integrated in one silicon device. We will describe the technical details, proof the concept of the device, and discuss the important requirements to the tip quality. Finally, we will present first experimental results obtained with the new instrument.

9:00am **NS-ThM3 Imaging Semiconducting Samples by Scanning Capacitance Force Microscopy (SCFM) and Scanning Capacitance Microscopy (SCM)*, K. Kobayashi,*** *K. Kimura, H. Yamada, K. Matsushige,* Kyoto University, Japan

We recently developed scanning capacitance force microscopy (SCFM) based on electrical force detection which is capable of mapping local differential capacitance (dC/dV) without an ultrahigh frequency capacitance sensor. 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) as a differential capacitance (dC/dV) signal is detected by a lock-in technique. 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. In this paper, we investigate the imaging mechanisms of SCFM using a silicon test sample having several microfabricated patterns of p-type, n-type and heavily-doped n-type regions. Image contrast changes depending on the applied bias voltage to the sample and the laser light irradiation were discussed. Conventional scanning capacitance microscopy (SCM) imaging were also performed and the results obtained by both techniques are compared and their advantages and limitations are discussed.

9:20am **NS-ThM4 AFM Force Measurements: MEMS Devices for Easy and Accurate Cantilever Spring-Constant Calibration*, P.J. Cumpson,*** National Physical Laboratory, UK*; J. Hedley,* University of Newcastle, UK*; P. Zhdan,* University of Surrey, UK

A value for the spring-constant of Atomic Force Microscope (AFM) cantilevers@footnote 1@ is necessary for the measurement of nanonewton and piconewton forces, which are critical to analytical applications of AFM in the analysis of polymer surfaces, biological structures and organic molecules.@footnote 2@ We have developed compact and easy-to-use micromachined reference artefacts for this calibration. The principal device consists of an array of dual spiral-cantilever springs, each supporting a polycrystalline silicon disc of 170 micrometres in diameter. These were fabricated by a two-layer polysilicon surface micromachining method. Doppler velocimetry is used to measure the fundamental resonant frequency of each device accurately. We call such an array a Microfabricated Array of Reference Springs (MARS). These devices have a number of advantages. Firstly, modelling the fundamental resonant frequencies of the devices is much more straightforward than for AFM cantilevers,@footnote 3@ because the mass and spring functions are isolated in different parts of the structure. Secondly, the spring constant of each spring is in linear proportion to the mass of the device, given that the resonant frequency is measured accurately. The thickness and hence the mass can be measured accurately by AFM or interferometry. These factors lead to much lower uncertainty than previous methods in which spring constant is proportional to the cube of a critical dimensional measurement. The array spans the range from 0.1 N/m to 10 N/m important in AFM, allowing AFM cantilevers to be calibrated easily and rapidly. New devices that extend this range down to around 0.03 N/m, and up to around 80 N/m will also be discussed. @FootnoteText@ @footnote 1@ N A Burnham et al, Nanotechnology 14 (2003) 1-6 @footnote 2@ J Colchero in Procedures in Scanning Probe Microscopies, Ed. R J Colton et al (Wiley, Chichester, UK, 1998)@footnote 3@C T Gibson et al, Nanotechnology, 7 (1996) 259-262. .

9:40am **NS-ThM5 Thermal Approach to Cantilever Calibration over a 200 kHz Bandwidth*, G.A. Matei,*** Wayne State University*; E.J. Thoreson, **N.A. Burnham,*** Worcester Polytechnic Institute

A cantilever's stiffness can be determined from an analysis of its thermal distribution spectrum, making use of the equipartition theorem k = k@sub B@T, where k is the cantilever stiffness and k@sub B@T is the thermal energy. We recently showed that it is possible to obtain good values for k, even when the cantilever's resonance is close to the edge of the data acquisition bandwidth, with the formula k = k@sub B@TQ@DELTA@@nu@/(@pi@@nu@@sub k@).@footnote 1@ Here, Q is the quality factor, @DELTA@@nu@ the frequency resolution of the data acquisition, @nu@@sub k@ is the resonant frequency, and is the mean-square amplitude at resonance. The initial work was limited to a 30 kHz bandwidth, that is, to cantilevers of stiffness less than approximately 0.4 N/m. Thermal calibration methods are independent of materials properties, coating thickness, cantilever geometry, and the viscosity of the medium, so there is good motivation to extend the methodology to higher bandwidths and thus stiffer cantilevers. We have now expanded the frequency range to 200 kHz, the bandwidth of the preamplifier of our AFM. By monitoring the raw A-B signal with an oscilloscope, then downloading the signal to a computer in order to manipulate the data, we have determined the stiffness of a 180 kHz cantilever to be 3.9 ± 0.2 N/m. By comparing our results for "force-calibrated" cantilevers with the manufacturer's stiffness calculation, we estimate that the accuracy of our method is 5%. By inverting a coated cantilever and repeating the measurement on the uncoated side, we believe that the precision is a similar value. Moreover, consistent with finite-element analyses, we find that the coating can perturb the stiffness by twenty percent. @FootnoteText@@footnote 1@Comparison of Calibration Methods for Atomic-Force Microscopy Cantilevers," NA Burnham, X Chen, CS Hodges, GA Matei, EJ Thoreson, CJ Roberts, MC Davies, SJB Tendler, Nanotechnology 14 (2003) 1-6.

10:00am **NS-ThM6 Are V-shaped Atomic Force Microscope Cantilevers Obsolete?*, J.E. Sader,*** University of Melbourne, Australia

The performance of the atomic force microscope (AFM) is underpinned by the mechanical properties of its force-sensing microcantilever. Originally, the AFM cantilever was handcrafted from a thin film of gold, in the shape of a rectangular plate. Shortly after, microfabrication techniques were introduced to facilitate the construction and mass production of AFM cantilevers, and a number of different cantilever geometries were proposed. Of these, rectangular and V-shaped cantilevers have emerged as

the accepted standards for AFM applications. The V-shaped cantilever was proposed initially as an alternative to the rectangular cantilever, with the explicit aim of minimizing the effects of lateral forces on the deformation of the cantilever. However, the motivating premise that V-shaped cantilevers are more resistant to lateral forces than rectangular cantilevers was never examined. Consequently, in this talk I shall present a detailed comparison of the complementary performance of V-shaped and rectangular cantilevers, with regards to their stability to lateral forces. In so doing, I shall rigorously establish that contrary to accepted thinking and the original intent, use of V-shaped cantilevers will enhance the effect of lateral forces in comparison to rectangular cantilevers. This counterintuitive finding is independent of whether the cantilevers are in contact with a surface or not, and suggests that rectangular cantilevers should be used in preference to V-shaped cantilevers for applications where the effects of lateral forces are to be minimized. This finding strongly contradicts established operating principles of the AFM, which dictate that V-shaped cantilevers should be used to minimize the effects of lateral forces. Consequently, drawing on the findings of this study, a case will be presented for the universal use of rectangular cantilevers in the AFM, which in turn will improve the performance of the instrument while greatly simplifying its operation.

10:20am **NS-ThM7 Electronic Properties of Individual Defects in Carbon Nanotubes by Scanning Probe Microscopy**, ***V. Meunier***, *S.V. Kalinin, R.J. Harrison, A.P. Baddorf,* Oak Ridge National Laboratory

Electronic devices based on carbon nanotubes and semiconductor nanowires require development and understanding of quantitative tools for transport measurements at nanoscale dimensions. Scanning Gate Microscopy (SGM) and Scanning Impedance Microscopy (SIM) are both capable of detecting atomic-scale defects in carbon nanotubes. In SGM individual defects are visualized as a decrease in the current through the dc circuit, since defects are depleted for tip voltages that are related to the local electronic structure of the defect. In SIM the local ac potential amplitude and phase are recorded; the defects are manifest as potential drops (back gate regime) or potential minima (tip gate regime). Here, a method for quantitative characterization of the electronic structure of individual defects from SGM and SIM results is presented. The interaction between a carbon nanotube and a point charge is studied using both atomistic first principles calculations and continuum electrostatic methods. The results are compared and extrapolated to real tip geometries in order to simulate the interaction of the scanning tip with an adjacent nanotube. Comparison with experimental data suggests that the gate voltage dependence of the image contrast is a direct measure of the difference in Fermi energies at these defects. The potential of the present approach for the identification of individual defects is discussed.

10:40am **NS-ThM8 Real-Space Imaging of the Vortex Lattice in V@sub 3@Si Using Low Temperature Scanning Tunneling Microscopy***, ***J.A. Stroscio***, *C.E. Sosolik, M.D. Stiles,* National Institute of Standards and Technology*; E.W. Hudson,* Massachusetts Institute of Technology*; S.R. Blankenship, A.P. Fein, R.J. Celotta,* National Institute of Standards and Technology

In Type II superconductors, the expulsion of an applied magnetic field from the superconductor, the Meissner effect, is not complete for fields above the lower critical field. In this applied field regime, magnetic flux penetrates the superconductor as quantized vortices that interact through shielding currents. In equilibrium the vortices form a lattice with symmetry, and orientation relative to the crystallographic axes, determined by microscopic electronic properties. In this talk we present low temperature scanning tunneling microscopy measurements of the structural evolution of the vortex lattice in a single-crystal V@sub 3@Si sample. Large-scale conductance maps obtained at 2.3 K provide a real-space image of the vortex lattice formed with a magnetic field applied parallel to the [001] crystal axis. Atomic resolution topography of the V@sub 3@Si (001) surface shows the underlying Si sublattice and allows for a determination of the orientation of the measured vortex lattice relative to the underlying crystal axes. The conductance maps reveal a change in the symmetry of the vortex lattice from hexagonal to nearly square over the field range of 0 T to 4 T. A strong anisotropy in the long-range translational order of the vortex lattice is observed near the transition field of 4 T. These observations give evidence for nonlocal electrodynamics in the vortex-vortex interactions of this Type II superconductor. Calculations that account for the role of these nonlocal effects in determining the structure and symmetry of the vortex lattice are presented and compared to our experimental results. * This work is supported in part by the Office of Naval Research.

11:20am **NS-ThM10 Adatom Hopping Induced by Tunneling Electrons: Br on Si(100)-(2x1)**, ***K.S. Nakayama***, *E. Graugnard, J.H. Weaver,* University of Illinois at Urbana-Champaign

Tunneling electrons from the tip of a scanning tunneling microscope can be used to induce adatom hopping on Br-terminated Si(100)-(2x1) at low current and without voltage pulses. Hopping does not occur when electrons tunnel from a sample to a tip. The threshold energy is +0.8 V, and tunneling spectroscopy shows antibonding Si-Br states 0.8 eV above the Fermi level. Electron capture in these states is a necessary condition for hopping, but repulsive adsorbate interactions that lower the activation barrier are also required. Such interactions are strong near saturation for Br but are insufficient when the coverage is low or when Br is replaced by Cl.

11:40am **NS-ThM11 Elucidation of the Electronic Properties of Isolated Alkanethiolate-Passivated Undecagold Clusters by Low Temperature Scanning Tunneling Microscopy and Spectroscopy**, ***S.U. Nanayakkara***, *R.K. Smith, T.P. Pearl, B.A. Mantooth, P.S. Weiss,* The Pennsylvania State University*; G. Woehrle, J.E. Hutchison,* University of Oregon

We have studied the electronic properties of isolated, octanethiolate-stabilized undecagold clusters [Au@sub 11@(S(CH@sub 2@)@sub 7@CH@sub 3@)@sub 10@] using low temperature scanning tunneling microscopy (STM) and spectroscopy (STS). The clusters, d@sub CORE@ = 0.8 ± 0.2 nm, were immobilized by inserted dithiol molecules in an alkanethiolate self-assembled monolayer (SAM) on Au(111). The clusters were synthesized in solution by ligand exchange of Au@sub 11@(PPh@sub 3@)@sub 8@Cl@sub 3@ with octanethiol, resulting in a complete octanethiolate ligand shell, and were subsequently deposited upon a SAM. The geometry of the STM tip-vacuum-gold cluster-SAM-Au(111) assembly can be modeled as a double barrier tunnel junction, which may give insight into controlling the movement of single or small numbers of electrons. Discrete quantum energy levels are more evident in this cluster size range where the atomic character of the metal is prominent. We have observed Coulomb blockade of these clusters at 4 K. The current-voltage characteristics show uneven spacing between adjacent current steps, showing quantized energy states. The observed, large zero-conductance gaps result from quantum size effects, where the bound octanethiolate ligand shell further reduces the free volume in which the electrons can move. This study assesses the impact of sub-nanometer sized clusters on single electron transport properties, enlightening the future of nanoscale electronics.

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