# Monday Morning, November 2, 1998

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS+SS-MoM

#### Tribology, Adhesion and Interfacial Forces Moderator: J.E. Houston, Sandia National Laboratories

8:20am NS+SS-MoM1 Nanotribological Interactions: Hard and Soft Interfacial Junctions, *U. Landman*, Georgia Institute of Technology INVITED Certain aspects of the dependence of materials properties on their size will be addressed, particularly at the nanometer length scale where new behaviour emerges when the physical dimensions of the system approach, or are reduced below, a characteristic length relevant for the phenomenon or physical process being probed. We focus on computer simulation studies of formation mechanisms, structural, mechanical, electronic, transport, dynamic and rheological properties of nanoscale hard (solid nanowires) and soft (confined liquid) interfacial junctions. These issues are central to research in nanotribology, that is explorations of the atomic and molecular scale origins and methods of control of friction and lubrication, and are relevant to modern miniaturization technologies. Work supported by DOE and AFOSR.

9:00am NS+SS-MoM3 Measuring Nanocontact Adhesion and Deformation, J.B. Pethica, University of Oxford, United Kingdom INVITED Nanoscale probe techniques such as nanoindentation and AFM have given new insight into contact mechanics, and hence into adhesion and tribology problems. Almost all local mechanical, and several electrical parameters of the component surface materials can be measured. The key is a reliable determination of the test probe displacement (lateral as well as normal) as a function of applied forces. Accurate determination of storage and loss moduli in thin polymer films, along with their frequency and temperature variation, will be described as one example of these capabilities, and of the associated stringent experimental requirements. During the approach to contact, sensitive force-distance spectroscopy, close to atomic scale spatial resolution, allows mapping of the interaction potential between the surfaces. Some of the outstanding problems will also be discussed. These include the effect of adsorbates, especially on the onset of irreversible or dissipative deformation, and the determination of contact area in systems which are anything other than clean and homogeneous.

# 9:40am NS+SS-MoM5 Adhesive Interactions and Damage Mechanisms in Scanning Probe Microscopy: A Study by Interfacial Force Microscopy, *J.F.*

Graham, O.L. Warren, P.R. Norton, University of Western Ontario, Canada It is often observed that contact mode scanning probe imaging with hard tips at loads of 10's of nN will damage soft surfaces, such as those of polymers. We have studied the origin of damage mechanisms on cellulose acetate (CA) calibration grids with the interfacial force microscope (IFM) using parabolic tungsten probes of 100 to 200 nm radius and forces of ~ 75 nN. The parabolic geometry of our W tips possesses a smooth profile at the apex with no sharp or discontinuous edges that can cause exceedingly high and damaging local stresses. The CA grid (elastic modulus ~ 2 GPa) can be imaged many times with a clean tungsten tip (modulus ~370 GPa) without visible damage, provided the maximum static force does not exceed that for plastic deformation of the CA. This force is readily and quantitatively determined by the IFM. After determination of a force (f) versus distance (d) curve (from which the nanomechanical properties are derived) during which the plastic limit of the CA was exceeded, contact mode imaging at the same force which had previously caused no damage, produced rapid, irreversible damage to the CA surface. It was also observed that there was a distinctive adhesion event (in the sense that it was not related to capillary forces) in the withdrawal curves of those f-d curves which preceeded damage. Further, occasionally we observed a sub-micron polymer particle on the surface after operation of the IFM in the tapping mode after an indentation experiment. This specific adhesive interaction (as well as the capillary forces) was eliminated by carrying out the measurements under hexadecane. The origin of the damage mechansim therefore appears to be: 1. the adhesive transfer of polymer to the tip; 2. the formation of adhesive contacts between this polymer "coated" tip and the CA surface; 3. repetitive formation and breaking through shear forces of polymerpolymer contacts at the interface.

#### 10:00am NS+SS-MoM6 Friction and Adhesion in the Attractive Regime, A.R. Burns, J.E. Houston, R.W. Carpick, Sandia National Laboratories

Recent molecular level measurements and simulations have shown a strong connection between adhesive bonding forces and energy dissipation

in sliding friction. In order to observe this directly, we have constructed a scanning force microscope with de-coupled lateral and normal force sensors to simultaneously observe the onset of both friction and chemical bond formation. Furthermore, by using a mechanically-stable interfacial force sensor,@footnote 1@ we are able to map the entire attractive interaction between the probe tip and the sample surface. Measurements made on self-assembling alkanethiol films with chemically different head groups show that friction can be directly attributed to bond formation and rupture well before repulsive contact. Thus we are able to separate chemical friction from more traditional mechanical sources of energy dissipation.@footnote 2@ @FootnoteText@ @footnote 1@ S. A. Joyce and J. E. Houston, Rev. Sci. Instrum. 62, 710 (1991). @footnote 2@ Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

#### 10:20am NS+SS-MoM7 Tribology and Mechanical Properties of Langmuir-Blodgett Monolayers, K.J. Wahl, W.R. Barger, S. Asif, Naval Research Laboratory

Mechanical properties of contacts with nanometer-scale dimensions are important in understanding the behavior of microscale sliding contacts. Monolayer films provide a model system to study fundamentals of relationships between adhesion, friction and mechanics. In this study, we deposited monolayers of distearoyl phosphatidyl ethanolamine (DSPE) and dioleoyl phosphatidyl ethanolamine (DOPE) (both mixed and single component) on freshly cleaved mica by the Langmuir-Blodgett technique. We use atomic force microscopy to examine morphology, adhesion and shear (sliding) behavior as well as modulation techniques to investigate mechanical properties of the monolayers as a function of deposition pressure. Measurements of film elastic/viscoelastic mechanical response via force modulation techniques are compared and contrasted with the surface compressional modulus determined during film deposition as well as to mechanical properties via nanoindentation.

### 10:40am NS+SS-MOM8 Deformation and Friction of Organic Monolayers, J.D. Kiely, J.E. Houston, Sandia National Laboratories

The use of organic monolayers as lubricating films has received considerable attention recently, especially with regard to their potential use in micromachine applications. We have used the interfacial force microscope (IFM) to characterize, on the nanometer scale, tribological properties of alkanethiol self-assembled monolayers on Au and monolayers of octadecyltrichlorosilane and perfluorodecyltrichlorosilane on Si. The IFM is similar to the atomic force microscope (AFM) but is distinguished by it use of a quantitative, mechanically stable, zero-compliance force sensor which allows us to measure both normal and frictional forces in a controlled fashion (i.e., without 'snap-to-contact'). We quantitatively relate monolayer deformation (chracterized by using the IFM in a nanoindentation mode) to friction, and find that, in the absence of appreciable adhesion/adhesion hysteresis, friction is very well correlated with deformation hysteresis. Additionally, we have identified the effects of wear, monolayer preparation procedure, and the environment (e.g., UV exposure and humidity) on tribological properties. This work was supported by the U.S. Department of Energy under Contract DEAC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the U.S. Department of Energy.

#### 11:00am NS+SS-MoM9 Structure-Dependent Viscoelasticity During Alkane Thiol Monolayer Growth, N.D. Shinn, T.M. Mayer, T.A. Michalske, Sandia National Laboratories

Organic monolayers adsorbed on contacting surfaces (e.g., as micromachine lubricants) modify both the chemistry and the mechanical properties of the interface. Whereas the chemistry can be predicted from the terminal functionality of the individual molecules, the viscoelastic properties reflect inadequately understood molecular ensemble dynamics. For example, the isomorphic self-assembled monolayers of methylterminated alkane thiol homologues [HS(CH@sub 2@)@sub n-1@CH@sub 3@ denoted as C@sub n@] have complex shear moduli that vary by orders of magnitude. We are using Acoustic Wave Damping (AWD) techniques and spectroscopic ellipsometry to elucidate the structure-dependent viscoelasticity of alkane thiols on polycrystalline Au(111) quartz crystal microbalance substrates. Multi-frequency analysis yields the complex shear modulus of equilibrium structures and a high-sensitivity oscillator circuit @footnote 1@ permits simultaneous measurement of the adsorption kinetics and energy dissipation during monolayer growth from the gas phase. Monolayer elasticity increases with alkane chain length. Co-

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adsorbed physisorbed molecules, chemisorbed two-dimensional fluid phases, and the nucleation and growth of condensed-phase islands each contribute to dissipation in the growing monolayers. Short chain (n < 10) thiol monolayers grow via Langmuir kinetics into a two-dimensional gas phase followed by slow condensation into ordered domains. However, the C@sub 12@ thiol exhibits surprising precursor-mediated kinetics and a highly viscous initial phase. Thiol dimerization is considered as a mechanistic explanation for the observed differences. Supported by DOE-BES Materials Sciences. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000. @FootnoteText@@footnote1@ K. Wessendorf, Sandia Nat. Labs. (patent pending).

# 11:20am NS+SS-MoM10 Lubrication Brought to Light: Optical Signatures of Nanomechanical Effects in Organic Thin Films, *R.W. Carpick*, *D.Y. Sasaki, S. Singh, A.R. Burns*, Sandia National Laboratories

The properties of organic lubricant films composed of hydrocarbon chain molecules is currently of great interest, as the detailed molecular response of these films to mechanical stress remains to be understood. Molecular films of polydiacetylenes are composed of ordered hydrocarbon chains attached to a chromophoric polymer backbone. These materials exhibit strong fluorescence emission and optical absorption which can be altered by mechanical stress (mechanochromism), providing the opporutunity to exploit the optical response as a signature of specific molecular behavior. Furthermore, structurally distinct chromatic phases ("blue" and "red" phases) of the film can be produced by a combination of Langmuir-Blodgett deposition and photopolymerization. We have studied the response of these films to mechanical stress at the nanometer level using a novel scanning force microscope. The instrument combines near-field optical detection with a displacement-controlled (non-compliant) normal force sensor and a decoupled lateral force sensor. Nanomechanical properties, including local elastic modulus, adhesion, and friction are compared for different chromatic phases of the film. We examine the modes of film deformation at the molecular level by monitoring the fluorescence emission while varying the mechanical stress applied to a nanometer-sized contact area. \* Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under Contract DE-AC04-94AL85000.

# 11:40am NS+SS-MOM11 In-situ Investigation of the Influence of a Mechanical Load on the Orientation of Organic Monolayers with Second Harmonic Generation, *M. Gurka, F. Eisert, M. Buck, M. Grunze,* University of Heidelberg, Germany

Understanding mechanical properties of ultrathin organic films is of fundamental importance due to their vital role in numerous technological applications such as hard disc drive lubrication or as moulde release agents in the injection moulding process. The relationship between macroscopically applied forces and processes taking place on the molecular scale can be studied in situ by photon-based techniques. Second harmonic generation (SHG) is a nonlinear optical technique which allows to investigate the effect of normal and shear forces on the order and orientation of confined monolayers. We have set up a model experiment consisting of an SHG-active silane film self-assembled onto a glas prism. A pressure in the range of 30 - 50 MPa is applied to the monolayer by pressing a quartz lens against the prism. Changes of the film structure are monitored by in situ polarization dependent SHG experiments. The SHG signal reflects the response of the SHG-active endgroup of the monolayer to the mechanical load and allows to determine the average tilt angle of the monolayer and also the in-plane symmetry of the molecular layer. During loading we observe only a minor change in the mean tilt angle of the endgroup whereas shear forces cause the molecules to align.

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS+EM+SS-MoA

#### Cross-sectional Scanning Tunneling Microscopy of Semiconductors

Moderator: M. Weimer, Texas A&M University

#### 2:00pm NS+EM+SS-MoA1 Scanning Tunneling Microscopy Studies of Atomic-Scale Structure In Semiconductor Heterostructures, E.T. Yu, S.L. Zuo, University of California, San Diego INVITED

Engineering of advanced heterostructure and nanoscale semiconductor devices requires a detailed understanding of the structure and properties of semiconductor materials and devices at the atomic to nanometer scale. Cross-sectional scanning tunneling microscopy provides unique and powerful capabilities for characterization of structural morphology and electronic properties in semiconductor epitaxial and device structures with spatial resolution at or near the atomic scale. In conjunction with results obtained using complementary characterization techniques, such studies can provide valuable insights into the relationships among epitaxial growth conditions, atomic-scale compositional structure, and various aspects of device behavior. We will discuss a number of recent applications of crosssectional scanning tunneling microscopy to the characterization of III-V compound semiconductor heterostructures. Studies of InAsP/InP heterostructures, currently of interest for optoelectronic devices operating at 1.3-1.55 microns, have revealed that extensive nanoscale compositional clustering occurs, with As-rich and P-rich clusters bounded preferentially by {111} planes forming in the InAsP alloys. Related studies of InNAsP/InP heterostructures, in which low concentrations (~1-2%) of N are incorporated, have provided information about the influence of N on heterojunction band alignments. And STM images of InAsP/InAsSb superlattices of interest for midwavelength infrared emitters have revealed nanoscale compositional fluctuations in these materials consistent with previously reported observations by electron diffraction of partial ordering in InAsSb alloys.

#### 2:40pm NS+EM+SS-MoA3 Growth Asymmetry in InGaAsP/InAsP Superlattices Studied by Scanning Tunneling Microscopy, B. Grandidier, H. Chen, R.M. Feenstra, Carnegie Mellon University; R.S. Goldman, University of Michigan; C. Silfvenius, G. Landgren, Royal Institute of Technology, Sweden

InGaAsP based multiple quantum well structures are increasingly used to fabricate optoelectronic devices. However the strain can lead to lattice relaxation processes during the growth which degrades the optical properties of these structures. To understand the differences in the photoluminescence efficiency of several superlattices composed of InGaAsP quaternary wells, we have investigated a series of InGaAsP/InGaP and InGaAsP/InAsP superlattices using cross-sectional scanning tunneling microscopy (xSTM). These superlattices were grown by metalorganic vapor phase epitaxy, with different number of periods and with or without InP interlayers inserted in the barrier. For InGaAsP/InGaP superlattices, the individual well and barrier layers are well resolved in the xSTM images. In contrast, for InGaAsP/InAsP superlattices, the InGaAsP quantum well and preceding InAsP barrier layers can be clearly seen, whereas the subsequent InAsP barriers are severely intermixed with the quantum wells. Possible mechanisms for this intermixing are described. In addition, the contrast observed in both types of superlattices has been related to the strain which exists in the layers; the compressively strained InAsP barrier protudes outwards from the (110) cleavage plane whereas the tensilely strained InGaP barrier contracts inwards. Finite element computations are used to quantify these elastic relaxation effects of the cleavage surface.

#### 3:00pm NS+EM+SS-MoA4 Microstructure of Mixed-Anion Interfaces Examined with XSTM@footnote 1@, J. Harper, M. Weimer, Texas A&M University; D. Zhang, C.H. Lin, S.S. Pei, University of Houston

The quality of the interfaces between the nearly-lattice-matched 6.1 Å materials (InAs, GaSb, and AlSb) is important for a number of applications, including the development of mid-IR lasers, long-wavelength photodetectors, and resonant-tunneling devices. Cross-sectional scanning tunneling microscopy (XSTM) is a powerful tool for characterizing the heterojunctions in these structures, which pose special challenges for molecular beam epitaxy (MBE) because of the mixed-anion nature of this material system. We have observed a white-noise component in the roughness spectrum of the GaSb-on-InAs interface with XSTM that is associated with the presence of interface point defects; these defects most

likely arise from thermodynamically favored anion exchange reactions that occur during the crossover from arsenide to antimonide growth. Abruptness of the InAs-on-GaSb interface, on the other hand, is limited by antimony segregation that causes compositional grading within the arsenic layers. We have quantitatively characterized the Sb fraction as a function of distance from the arsenide-on-antimonide heterojunction, and find this compositional grading is well described by an exponential profile. @FootnoteText@@footnote 1@ Work supported by the National Science Foundation (DMR-9633011).

# 3:20pm NS+EM+SS-MoA5 X-STM Study of InAs/In@sub 1-x@Ga@sub x@Sb/InAs/AISb Laser Structures@footnote 1@, W. Barvosa-Carter, M.J. Yang, L.J. Whitman, Naval Research Laboratory

Strained-layer heterostructures involving the 6.1 Å family of III-V semiconductors (including InAs, GaSb, and AlSb) are being investigated for use in a growing number of high-speed and opto-electronic devices. Recently it was shown in InAs/In@sub 0.73@Ga@sub 0.28@Sb/InAs/AISb mid-IR structures that the photoluminescence (PL) intensity and x-ray superlattice diffraction quality are strongly dependent on MBE growth temperature. These characteristics were shown to be optimized within a rather narrow growth temperature range (410-460°C) and much worse outside of that range. Although the quality of the interfaces in these structures is expected to play a crucial role in determining device performance, little is known about the actual atomic-scale structure of the interfaces. We present an atomic-resolution cross-sectional STM (X-STM) study of these laser structures in order to directly correlate atomic-scale features, such as interface roughness and layer intermixing, with material quality as measured by PL and x-ray measurements on the same samples. Two such laser structures have been examined, one grown at the optimum temperature and another grown at a higher temperature. Interface roughness appears to be larger in the higher temperature structure. In addition, intermixing occurs at the AlSb-on-InAs interfaces which results in electronic structure differences between the InAs-on-AISb and AISb-on-InAs interfaces as observed by X-STM. Based on our X-STM results, we will discuss the atomic-scale sources of device degradation, and present possible routes towards improvement of the growth of these laser structures. @FootnoteText@ @footnote 1@ Funded by the Office of Naval Research and the Air Force Research Laboratory.

#### 3:40pm NS+EM+SS-MoA6 Kinetics of Anion Cross Incorporation in Type-II Heterostructures Characterized with XSTM@footnote 1@, J. Steinshnider, J. Harper, M. Weimer, Texas A&M University; D. Zhang, C.H. Lin, S.S. Pei, University of Houston

We have used cross-sectional scanning tunneling microscopy (XSTM) to examine MBE material quality in the mixed-anion InAs/GaSb/AlSb system under growth conditions (including the use of cracked arsenic and antimony sources) similar to those presently employed for type-II quantum well and interband cascade lasers. Two apparently different anion defects are noted within the antimonide layers. The demonstration of a linear correlation between the defect densities observed with STM and the arsenic valve setting during antimonide-layer growth establishes background arsenic incorporation as the common origin for both of these defects.@footnote 2@ The distribution of As substitutional defects in a (110) cleavage plane is analyzed by way of the two-dimensional pair correlation function. We observe a pronounced attractive correlation in the [110] direction, parallel to the Sb dimer bonds of the (1x3) reconstructed growth surface, whereas the distribution in the orthogonal [001] direction is essentially random. This anisotropic correlation reflects the kinetics of arsenic dimer incorporation during growth and not the equilibrium distribution associated with strain-mediated repulsive interactions. @FootnoteText@ @footnote 1@ Work supported by the National Science Foundation (DMR-9633011). @footnote 2@ J. Harper, M. Weimer, D. Zhang, C.H. Lin, and S.S. Pei, JVST B 16, in press (1998).

#### 4:00pm NS+EM+SS-MoA7 Low Temperature Cross-Sectional Scanning Tunneling Microscope-Induced Luminescence of GaN, S. Evoy, C.K. Harnett, Cornell University; S. Keller, U.K. Mishra, S.P. DenBaars, University of California, Santa Barbara; H.G. Craighead, Cornell University

The GaN system is of interest for applications in the green, blue, and UV spectral regions. Advances in device development have been made in spite of issues such as dislocation densities and defect induced visible luminescence. These issues prompted interest in spatially resolved luminescence studies of the material. Scanning tunneling microscope-induced luminescence (STL) offers nanometer scale resolution and control of the injection bias. In-situ cleaving and cross-sectional imaging is of garticular interest for nanoscale luminescence studies of GaN

heterostructures and interfaces. We recently reported the first low temperature STL of GaN, and the first STL images of this material. We now report the low temperature cross-sectional STL of MOCVD-grown GaN. Optical interference filters are used for semiquantitative spectral analysis. Room temperature top-view experiments reveal faint visible emission at tip biases above 1.5 V, with no clear evidence of UV luminescence. However, a sharp increase of emission in the 350±35 nm range is observed under liquid He cooling at biases above 3 V. The room temperature visible emission may be related to surface issues, suggesting that low temperature is required for the analysis of intrinsic bulk luminescence. Cross-sectional experiments are performed on in-situ cleaved samples. Incompatible cleaving planes between the GaN and the sapphire produce 200-400 nm wide vertical features, yielding an edge roughness of 30-50 nm. Behavior of luminescence is similar to what was observed in top-view. However, close to the sapphire interface, the 350±35 nm band-edge emission is undetected even at low temperature. Images show strong correlation between the remaining visible emission and the cleaved-induced artifacts. We are currently working on our cleaving technique in order to improve the quality of the edge. The technique will also be applied to the study of GaN heterostructures such as InGaN/GaN quantum wells.

#### 4:20pm NS+EM+SS-MoA8 Cross Sectional STM Study on MBE-grown Si/Ge(111) Interface, *H. Hirayama*, *M. Ohmori, K. Takayanagi*, Tokyo Institute of Technology, Japan

We studied the (111) cross sectional surface of MBE grown Si/Ge(111) samples. Samples were cleaved in ultra-high vacuum, and their (111) cross s ection were investigated in-situ by using STM. On the as-cleaved surface, 2x1 reconstruction were observed at both Si and Ge side. After annealing, 2x1 reconstruction changed to 7x7 and c(2x8) on the Si and Ge layer, respec tively. At around the interface, 7x7 reconstruction changed to c(2x8) reconstruction in moving from Si to Ge side. But, the transition from 7x7 to c(2x8) was not abrupt. The transient region of the width of c.a.200nm was obs erved. In the transient region, adatoms arranged with 2x2 and c(2x4) shor t range orderings. Patchy domains of 7x7 reconstruction, which was accompa nied with (110)- oriented grooves and non-double layer height steps, were a lso observed in the sea of 2x2 and c(2x8) arrangement of adatoms. In a det ailed analysis of adatom arrangement, we found that the non-double layer hei ght step was caused by the glide in the (111) plane parallel to the substr ate. The groove was triggered by partial dislocations at the edge of the gild region. The strain field with the glide-induced step and grooves modifi ed the surface strain locally, and caused patchy 7x7 domains.

#### 4:40pm NS+EM+SS-MoA9 Scanning Tunneling Microscopy Characterization of the Depletion Zone of a Si Lateral pn Junction, *M.L. Hildner*, *R.J. Phaneuf*, *E.D. Williams*, University of Maryland, College Park

Scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) are used to characterize lateral pn junctions fabricated on silicon (100) surfaces. Two separate device structures , one with p@super +@-n and the other with n@super +@-p abrupt junctions, were examined. The STM images of the first set of devices show both an electronic feature and a structural groove on each side of the ion implanted p-type regions. The groove is an etching artifact of the implantation mask fabrication process and was easily avoided in making the second set of devices which show only a similar electronic feature. The electronic feature widens with applied reverse bias with a voltage dependence that closely matches that expected for the depletion zone. However, the width of the electronic feature is much smaller than that of the depletion zone. The STS measurements show that the tip-junction system can be modeled as a series of non-equilibrium metal-insulator-semiconductor (MIS) diodes formed with a semiconductor of spatially variable carrier density. From this model, we qualitatively describe the electronic feature as confined to that portion of the depletion region in which the biasing sense of the MIS junction is switched from the biasing sense when the junction is in the lightly doped neutral region. Thus, the electronic feature commences, as the tip is moved from the lightly doped neutral region into the depletion region, when the majority carrier changes (from electrons to holes for the lightly doped n devices). This work has been supported by the Laboratory for Physical Science, with partial support from the NSF-MRSEC.

Surface Science Division Room 308 - Session SS1+NS-MoA

#### **Novel Surface Probes**

Moderator: J.T. Yates, Jr., University of Pittsburgh

#### 2:00pm SS1+NS-MoA1 Momentum Resolved ESDIAD, A New Technique, Probing the Low Frequency Motion of Adsorbed Molecules on Single Crystal Surfaces@footnote 1@, J.W. Ahner, D. Mocuta, J.T. Yates, Jr., University of Pittsburgh

A new technique, Momentum Resolved ESDIAD (Electron Stimulated Desorption Ion Angular Distribution), provides a method for taking snapshots of the zero-point position and lateral momentum of particles adsorbed on crystalline surfaces. By employing state of the art electronics and computer technology it is possible to record for each desorbing particle the desorption direction together with the flight time. Highly momentum and directional resolved images are obtained, with time-offlight resolution in the picosecond range and data acquisition rates up to 100 kHz. This enables us to deconvolute spatial and momentum contributions to the ESDIAD pattern and to map the low frequency motion of the adsorbed particles. These maps reflect the adsorbate interactions with the substrate and with neighboring species on the substrate. For selected examples we will present data 'movies' demonstrating how these unique maps of the dynamical behavior of adsorbed species are used in several ways to probe the lowest energy states, as well as to measure the momentum distribution when the particle gains thermal energy. One major opportunity involves dissimilar chemisorbed species which, when imaged together in momentum and real space, give new insights into the first stages of interaction between the species, leading ultimately to a chemical reaction. In addition we present lateral momentum distribution studies for an adsorbed molecule with a rotational symmetry axis showing the rotation of the molecule on its adsorption site about this axis. Such information can be used as a basis for thinking about anisotropies in lateral motion of particles on surfaces. @FootnoteText@ @footnote 1@work supported by DOE/BES.

# 2:20pm SS1+NS-MoA2 UV Spectroscopy of CO and Benzene on Pt(110), N. Chen, I. Lee, R. Masel, University of Illinois, Urbana

Recently there has been some controversy about the role of d-backbonding in the adsorption of gases on transition metals. People have suggested that the antibonding orbitals should shift, but without any direct measurements, the theory remains controversial. In this paper we use a standard HREELS spectrometer, with modified electronics to measure the equivalent of a UV spectrum for two different systems: CO on Pt(110) and benzene on Pt(110). In the CO case, the UV spectrum shows peaks at 5.41, 5.58 and 7.91 eV independent of coverage. By comparison, gas phase CO shows peaks at 6.04, 6.92, 7.58, and 7.94 eV. The large shifts are indicative of the antibonding orbitals being stabilized, as one would expect from the Blyholder model and recent calculations of Ilias et al, Surface Science 376, (1997) 279 but not with the calculations of Ohsishi and Watarri Phys Rev B 49(1994)14619. In the benzene case we observe two different spectra: a first monolayer spectrum with a broad peak center at 4.71 eV, and a multilayer spectrum with peaks at 3.78, 4.73, 6.11 and 6.82 eV. The multilayer spectrum matches the spectrum of condensed benzene, but the first monolayer spectrum is quite different. Again these results suggest that there is a substantial stabilization of the antibonding orbitals of adsorbed benzene. Together these results show that UV spectroscopy provides useful information about adsorbates on surfaces.

#### 2:40pm SS1+NS-MoA3 Calorimetric Measurements of Metal Adsorption and Adhesion Energies on Clean, Single-Crystalline Surfaces, C.T. Campbell, J.T. Stuckless, D.J. Bald, D.E. Starr, J.E. Musgrove, University of Washington INVITED

The adsorption and adhesion energies of metals on solids are important in many materials and chemistry applications including oxide-supported metal catalysts, bimetallic catalysts, epitaxial thin film growth, metalceramic interfaces in microelectronics, metalization of polymers, composite materials and metal adsorption on minerals in soils. The heats of adsorption of metals have been measured calorimetrically for the first time on clean, single-crystalline surfaces. A pulse of metal vapor from a chopped atomic beam adsorbs onto an ultrathin single crystal's surface in ultrahigh vacuum, causing a transient temperature rise. This heat input is detected by a pyroelectric polymer ribbon, which is gently touched to the back of the crystal during calorimetry. The sticking probability is measured by detecting the reflected fraction mass spectrometrically with a line-of-sight modification of the King and Well's method. The differential heat of

adsorption is thus measured as a detailed function of coverage up through multilayer coverages. The integral heat of adsorption also provides the adhesion energy of the metal film, if the surface free energy of the clean metal surface is known. Adsorption and adhesion energies for metals (Pb or Cu) on the clean Mo(100) surface, on well-defined surface oxides of Mo(100) and W(100), and on clean and hydroxylated MgO(100) thin films will be reported. By comparing a variety of surfaces in Pb and Cu adsorption, an interesting correlation between the growth morphology of thin metal films and the initial heat of adsorption of the metal is revealed. The sticking probability also correlates with the heat of adsorption of the metal.

#### 3:20pm SS1+NS-MoA5 Multispectral Image Classifications of Si(001) Surface Electronic Structure, K.M. Horn, B.S. Swartzentruber, G.C. Osbourn, Sandia National Laboratories

We have imaged the electronic structure of Si(001) surfaces by applying multispectral image analysis techniques to multi-bias STM conductance data. Atomic surfaces are first characterized by recording conductance spectra, C(V), at each point in a 2D scan of the surface. The resulting 3D data set, (x, y, C(V) ), is then converted into a series of bias-dependent conductance images. These images are analyzed to produce a single, colorcoded, classed image that reflects the surface's electronic structure. The image analysis is performed by a computed grouping algorithm that identifies pixels sharing common conductance characteristics. The resulting classed images distinguish features not clearly resolved in a topographic image, and reveal stark electronic differences between topographically similar features. We first demonstrate the reliability of this classification technique on simple Si(001) features. Classed surfaces are then presented for various surface defects and Si and Ge structures that have been deposited on the Si(001) surface. These electronic structure images reveal features that are not readily visible or distinguished in a constant-current topograph. Direct comparison of the conductance spectra from these features confirms the classification result. This computer-based data reduction technique may prove useful in defect detection, validating surface models, and in understanding more complicated systems in which atomistic models are derived from a limited number of single-bias topographic images. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the U.S.DOE under contract DE-AC04-94AL85000.

#### 3:40pm SS1+NS-MoA6 A Simple Nulling Technique for Measuring Complex-Valued Nonlinear-Optical Susceptibilities of Interfaces, J.R. Dennis, V. Vogel, University of Washington

For studies of isotropic interfaces by nonlinear optics, a general technique is presented to measure the complete second-order surface susceptibility, up to an overall phase factor. The measurement and data analysis are simple and rapid, with no use of a variable reference phase, and the susceptibility is overdetermined, allowing consistency checks. The technique involves measuring the complete polarization state of the nonlinear-optical signal by nulling the signal, for several linear polarizations of the input beam. Mesurements of second harmonic generation from Langmuir monolayers of the liquid crystal 4'-n-octyl-4-cyano-biphenyl (8CB) have been made with this technique, giving results which agree with previous data, and also revealing a small phase shift between some susceptibility components. This phase shift may be explained by introducing a complex dielectric constant for the monolayer at the second harmonic frequency. Data for the free surface of isotropic 8CB have also been analyzed with this technique. The technique is particularly well-suited to testing or fitting different models of Fresnel factors and local-field factors.

#### 4:00pm SS1+NS-MoA7 Characterization of Near-Field Probes for Enhanced Raman Spectroscopy, *C.E. Jordan*, *L.J. Richter, R.R. Cavanagh, S.J. Stranick,* National Institute of Standards and Technology

Near-field Raman spectroscopy can potentially obtain chemical specificity with the subwavelength resolution of a near-field scanning optical microscope (NSOM). Signals from a single crystal diamond sample have been evaluated to assess the limits of this technique. Three different types of illumination mode fiber optic probes have been used in the near-field Raman experiments: an uncoated probe; a probe coated with a smooth layer of aluminum which has an aperture that is less than 100 nm in diameter; and a probe coated with a smooth layer of aluminum and then overcoated with a rough layer of silver. In order to discriminate between the enhanced contribution to the Raman signal observed in the near-field of the probe and bulk scattering, the Raman intensity from each type of probe is measured as a function of the probe sample separation. Very little change in the Raman intensity is observed as a function of probe sample separation when an uncoated fiber optic probe is used. For an optical probe coated with a smooth layer of aluminum the Raman intensity is about a factor of seven higher when the sample is in the near-field of the probe compared to the intensity measured when the sample is farther than 100 nm away from the probe. Probes that are coated with a rough silver film show a greater enhancement in the near-field Raman intensity than is observed for probes coated only with aluminum.

#### 4:20pm SS1+NS-MoA8 Element Specific Real-Space Imaging Surface Crystallography, *L. Houssiau*, *J. W. Rabalais*, University of Houston

Scattering and recoiling imaging spectrometry (SARIS) extends the technique of time-of-flight scattering and recoiling spectrometry (TOF-SARS) to include both spatial and time resolution of scattered and recoiled particles. SARIS uses a time-resolving, position sensitive, microchannel plate (MCP) detector, resistive anode encoder (RAE), time-to-digital converter (TDC), and a triple axis UHV goniometer to measure the velocityresolved spatial distribution patterns of scattered and recoiled particles produced by a keV beam of pulsed ions from on a crystalline surface. The images combine the advantage of atomic scale microscopy and spatial averaging simultaneously since they are created from a macroscopic surface area but they are directly related to the short-range (< 10 Å) atomic arrangements in the surface. The non-planar scattering features in the images are not normally observed in conventional ion scattering experiments using small-area detectors. The technique is applied to carry out real space imaging of Ni(110) and oxygen chemisorbed Ni(110) with 4 keV He@super +@ ions. A mapping of the entire hemisphere where the ions are reflected was made possible by collecting several images at different angles and merging them together. These maps reveal the blocking cones of surface atoms, which gives a real space image of the crystal surface. After oxygen exposure, the images are modified and reveal the O chemisorption sites. The features of these images can be accurately reproduced by classical ion trajectory simulations using the scattering and recoiling imaging code (SARIC).

#### 4:40pm SS1+NS-MoA9 An Axial Resonant Force Probe for Atomic Force Microscopy, J.A. Harley, T.W. Kenny, Stanford University

A resonant force probe has been constructed which exhibits high force sensitivity from a stiff transducer with a stationary tip. The resonant beam is mounted vertically relative to the surface, as in shear force microscopy. but a tether has been added near the tip. The tether forces the beam to oscillate in a pinned-pinned mode while the tip remains stationary, but does not interfere with axial forces. This configuration has several advantages over current force measurement techniques. First, since the beam is perpendicular to the surface, the probe is not susceptible to force gradient instabilities. Second, the stationary tip provides high spatial resolution in the force measurements. Typical oscillating cantilevers average forces over the oscillation amplitude. Third, since the oscillations can be large, the resonant detection method is not as demanding on the secondary detector, so the force sensitivity in a piezoresistive sensor could approach that of optical lever techniques. Finally, the oscillator could potentially be encapsulated, allowing a high Q resonator in a liquid environment. The beam was constructed out of single crystal silicon, and measures 0.2 x 3 x 200µm, with an implanted piezoresistor to detect the oscillations. The axial spring constant is over 200N/m. Resonant frequency shifts of  $30 \text{kHz}/\mu \text{N}$  are detected using a phase-lock loop circuit. In air, the oscillator is heavily damped (Q of 15) but still demonstrates 10nN force resolution in a 1kHz bandwidth. In a moderate vacuum the resonance quality improves to 1200, and10pN force resolution is expected. The design, analysis, and theoretical limitations of these sensors will be discussed.

5:00pm SS1+NS-MoA10 Super Transmission and Resolution Energy Analyzer and Mass-Analyzer System (STREAMS), K. Siegbahn, R. Maripuu, ESCA LASER Lab Institute for Materials Science, Sweden; N. Kholine, Russian Academy of Sciences, Russia; U. Golikov, State Technical University, Russia; M. Larin, Joint Stock Co. CRYOVACS, Russia

A new type of instrument for scientific and technological research is proposed. Its main peculiarity and advantage are the capability to separate charged particles in accordance with their energies and masses on high level of resolution and sensitivity. A basis of the spectrometer is electrostatic axially symmetrical field structure with matched radial and axial potential gradient. The electron optical system can function either in dispersion or time-of-flight mode of operation. The charge particles follow the same trajectories in this field independently of the mode of operation. Electron spectroscopy or mass-spectroscopy information from the same

point of the analyzed sample can be received by switching over to the appropriate potentials of the power source and the detector system. So one and the same instrument can function as a high performance electron spectrometer or a mass-spectrometer. Relative energy resolution better than 0.05% in the energy range of 20-3000 eV and mass resolution more than 5000 in the mass range 1-500 a.e.m. are easily realized for acceptance solid angle of the spectrometer equals to at least 30% out of hemisphere. The diameter of the analyzer is 200 mm, its length is 600 mm. An ultra high vacuum is ensured in the spectrometer at the level 10@super-11@ mbar by oil free pumping system with highly economical cryo condensation-absorption pump cooled by liquid helium. Almost all the spectrometer is made of nonmagnetic materials and first of all from titanium. The surfaces faced into vacuum have special plating with very low absorption-desorption capacity. So ultra high vacuum is achieved without baking out the spectrometer.

#### The Science of Micro-Electro-Mechanical Systems Topical Conference

#### Room 324/325 - Session MM+NS+SS-TuM

#### **Micro-Science and Tribology of MEMS**

Moderator: N.E. McGruer, Northeastern University

#### 8:20am MM+NS+SS-TuM1 Making a Bridge to the Nanoworld, S.R. Manalis, S.C. Minne, J.D. Adams, K.B. Crozier, H.T. Soh, T.A. Sulchek, K. Wilder, Stanford University; G.G. Yaralioglu, A. Atalar, Bilkent University, Turkey; C.F. Quate, Stanford University INVITED

Our vision for micro-electro-mechanical-systems (MEMS) is to provide a window to the microscopic world. Scanning probe microscopes with automated cantilever arrays now image surface areas in excess of one square millimeter with atomic resolution. We will present new types of cantilevers and transducers that improve the speed, sensitivity, and simplicity of scanning probe microscopes. Samples are imaged at video rates with an integrated piezoelectric actuator that bends the cantilever over surface topography at high speeds. The deflection sensor, which consists of a micromachined light modulator, monitors cantilever bending with a sensitivity near one percent of an atomic diameter. We also present approaches for microfabricated biological sensors based on mechanical, electrical, and optical methods of transduction.

#### 9:00am MM+NS+SS-TuM3 Nanotribology of Vapor-Phase Lubricants and Their Potential Applications to MEMS@footnote 1@, J. Krim, North Carolina State University INVITED

The concept of lubricating high temperature surfaces with organic vapors has existed for at least forty years, with substantial efforts beginning in the 1980's and continuing on to the present day. Vapor-phase lubricants are advantageous for use at high temperature, as well as in situations where the vapor can be used as a reservoir for replenishment of areas where the lubricant has been depleted in the course of device operation. While work in the area of vapor-phase lubrication has to date focussed on the lubrication of macroscopic systems, vapor lubrication mechanisms may ultimately prove to be of critical importance to sub-micron mechanical systems in cases where lubricant delivery and/or replenishment by other methods proves impractical. In order to examine the viability of vaporphase lubrication at length scales commensurate with submicron-scale machinery, we have constructed a Quartz Crystal Microbalance which operates in combination with a Scanning Probe Microscope so as to form a simple nanometer-scale mechanical system whose response to a number of vapor-phase lubricants can be monitored for nanotribological performance. Our observations of organic and water-vapor films recorded with this device will be discussed. @FootnoteText@ @footnote 1@Work supported by NSF and AFOSR

#### 10:20am MM+NS+SS-TuM7 Vacuum Deposited Fluorinated Alkyl Siloxane Films for Adhesion Control in MEMS Devices, T.M. Mayer, M.P. de Boer, N.D. Shinn, P.J. Clews, T.A. Michalske, Sandia National Laboratories

Monolayer films of polymerized alkyl siloxanes have been employed for surface passivation and adhesion control in MEMS devices. However, reproducible film formation and properties have been difficult to achieve due to process sensitivity to substrate preparation conditions, presence of small quantities of adsorbed water, and the high aspect ratio structures typical of MEMS devices. In contrast to the normal solution coating process using alkyl trichlorosilane precursors, we have developed a vacuum-based film deposition process, using volatile fluorinated alkyl trichloro silane precursors. Reproducible substrate conditions are obtained by UV-ozone oxidation followed by sequential or simultaneous exposure to the chlorosilane precursor and water vapor. Efficient transport of reactants into high aspect ratio structures is accomplished by maintaining Knudsen flow conditions at low pressures. We measure kinetics of film growth by insitu ellipsometric and quartz-crystal microbalance techniques, and evaluate film composition and structure by XPS and IR spectroscopies. We also measure the work of adhesion and surface energy of coated cantilever beams under equilibrium fracture mechanics conditions. We compare results to uncoated structures, and to structures coated from solution with alkyl and fluoro-alkyl siloxane films. Sandia is a multiprogram laboratory operated by Sandia Corp., a Lockheed Martin Company, for the U. S. Dept. of Energy under contract DE-AC04-94AL85000.

11:00am MM+NS+SS-TuM9 Adhesion Hysteresis of Polysilicon Beams in Controlled Humidity Ambients, *M.P. de Boer, T.A. Michalske, M.R. Tabbara,* Sandia National Laboratories; *R. Maboudian,* University of California, Berkeley; *T.M. Mayer,* Sandia National Laboratories

Auto-adhesion, or spontaneous sticking between MEMS structures, is currently a major limitation in bringing this new class of engineering devices to the broader market. MEMS are particularly susceptible to autoadhesion because the structural members: 1) are constructed in close proximity to each other, 2) are highly compliant due to their extreme length to thickness aspect ratio and, 3) have large surface to volume ratios which increase the relative importance of adhesive surface forces. If the miniature structural members are brought together by surface (capillary, electrostatic) or inertial (shock, rapid air flow) forces, they may remain adhered after the external force is removed. If the structures remain adhered, bonding may increase over time, giving rise to the phenomena known as adhesion hysteresis. In this work we develop mechanical analysis for and report on measurements of adhesion hysteresis in surface micromachined polysilicon beams subject to dry and wet ambients. The electrostatically activated beams used in this study were tested directly after supercritical drying or after the application of hydrophobic molecular octadecyltrichlorosilane (ODTS) coatings such as or perfluorodecyltrichlorosilane (FDTS). Results indicate that both uncoated and coated beams show strong increase in adhesion after an incubation period in humid environments. This incubation time is shorter and occurrs at lower RH for uncoated beams than coated beams. For the case of uncoated beams, we are able to show that a model based on individual asperity contact forces can be used to predict the overall adhesion behavior in micromachined beams. The behavior of coated beams is compared with ellipsometric measurements indicating water adsorption on these nominaly hydrophobic surfaces after extended exposure at high RH conditions.

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS-TuM

Quantum Structures and Molecular Electronics Moderator: C.R.K. Marrian, Naval Research Laboratory

8:20am NS-TuM1 Quantum-Dot Cellular Automata, G.L. Snider, A.O. Orlov, I. Amlani, G.H. Bernstein, C.S. Lent, J.L. Merz, W. Porod, University of Notre Dame INVITED

Quantum-dot Cellular Automata (QCA) is a promising architecture which employs quantum dots for digital computation. It is a revolutionary approach which addresses the issues of device density and power dissipation. It represents a concrete device design, scalable down to atomic dimensions, with possible implementations in both metals and semiconductors. A basic QCA cell consists of four quantum dots coupled capacitively and by tunnel barriers. Two excess electrons within the four dots are forced to opposite "corners" of the four-dot system by Coulomb repulsion. These two possible polarization states of the system represent logic "0" and "1". Properly arranged, arrays of these basic cells can implement the Boolean logic functions and memory needed for general purpose computation. An introduction to the QCA architecture will be presented, along experimental results from a functional QCA cell built of nanoscale metal dots defined by tunnel barriers. The QCA cell to be presented consists of two Al double-dot islands defined by tunnel junctions, capacitively coupled to each other. Al/AlO@sub x@/Al tunnel junctions are fabricated using a standard e-beam lithography/shadow evaporation technique. In addition to the QCA cell, two single-dot Al islands are capacitively coupled to the QCA cell to act as electrometers. Direct measurements of the charging diagram of QCA cell, combined with electrometer measurements of the cell, show a controlled polarization switch of the QCA cell. These and additional results confirm the control of the switching of a single electron by a single electron, and demonstrates a non-linear, bistable response in the QCA cell. There is excellent agreement between the experimental results and theory.

#### 9:00am NS-TuM3 A Proposal of Atom/Molecule Switching Devices, Y. Wada, Hitachi Ltd., Japan INVITED

This paper describes the possibility of atom/molecule switching devices, Atom Relay Transistor (ART)@footnote 1@ and MOlecular Single Electron Switching transistor (MOSES),@footnote 2@ which would supersede present semiconductor devices beyond their ultimate limitations. ART consists of an atom wire, a switching atom, a switching gate and a reset

gate. MOSES devices consist of a conducting molecule and an insulating molecule, the former being the quantum dot and the latter the tunnel barrier. ART and MOSES devices are evaluated on the basis of the five major characteristics necessary for information processing integrated circuit devices,@footnote 1@ and indicated that they are the most promising candidates for the future information processing. Scanning Tunneling Microscope (STM) should be the most probable tool to fabricate these devices. Technology development to realize these atom/molecule devices are described, including Beam Assisted STM (BASTM)@footnote 3@ which enables insulator observation, Needle Formation and Tip Imaging (NFTI)@footnote 4@ which directly evaluates STM tip apex for reliable atom/molecule manipulation, micromachine STM@footnote 5@ which makes possible the direct observation of vacuum tunneling gap. Gallium (Ga) atom wire was successfully fabricated on Si (100)-H surface by removing hydrogen atoms by STM and filling the dangling bond by Ga atoms,@footnote 6@ which is theoretically predicted to be conductive.@footnote 7@ Those technologies should lead to a successful ART/MOSES demonstration. @FootnoteText@ @footnote 1@Y.Wada, et al., J. Appl. Phys., 74, 7321 (1993). @footnote 2@Y.Wada, Trans. IEICEJ, OME 93-54, 31 (1994). @footnote 3@S.Heike, et al., Appl. Phys. Lett., 64, 1100 (1994). @footnote 4@S.Heike, et al., Japan. J. Appl. Phys., 34, L1061 (1995). @footnote 5@M.I.Lutwyche et al., Appl. Phys. Lett., 66, 2807 (1995). @footnote 6@T.Hashizume, et al., Japan. J. Appl. Phys., 35, L1085 (1996). @footnote 7@S.Watanabe, et al., Phys. Rev. B, 54, 17308 (1997).

#### 9:40am NS-TuM5 Self-Assembled Single Electron Tunneling Devices, S.H.M. Persson, L.K. Hedberg, L.G.M. Olofsson, B. Kasemo, Chalmers Univ. of Technology and Univ. of Gothenborg, Sweden

Single electron tunneling effects were studied in self-assembled devices, by contacting a nanoscale gold cluster to two gold electrodes. The size of the gold cluster was around 5 nm, which is controlled by the chemical synthesis. Coulomb blockade of tunnelling was observed at room temperature and Coulomb staircase at 4.2 K. With a third gate terminal it was possible to modulate the tunneling characteristic by electric field effect at 4.2 K. Nanoscale electronic devices can be made with refined lithographical techniques, e.g. using scanning probe instruments, but these methods are very slow and not practical for large scale fabrication. The electrodes were made by electron beam lithography and angled evaporation to control the gap between the electrodes to distances smaller than 10 nm. The surface of the gold electrodes were modified by a selfassembled monolayer of 1,8-octanedithiol and gold clusters were found to be captured in the electrode gap after immersion of the sample in a hexane solution of clusters. The characteristic feature of single electron tunneling can be seen in the current voltage characteristic as a number of steps, that is named the Coulomb staircase. The Coulomb blockade is observed at room temperature with a blockade voltage of the order 0.2 V. Recent theoretical results@footnote 1@ predict a new "electron shuttle" mechanism for systems similar to ours, where the middle electrode is softly coupled to the outer ones via organic molecules. The softness of the molecular links implies that charge transfer could give rise to deformation of these structures. Under certain conditions this would result in oscillation of the gold nanoparticle and a current through the structure that is proportional to the cluster vibration frequency. One aim of our work is to verify these predictions experimentally. @FootnoteText@ @footnote 1@L.Y.Gorelik, A.Isacsson, M.V.Voinova, B.Kasemo, R.I.Shekter and M.Jonson, Phys. Rev. Lett., 80 (1998), 4526

#### 10:00am NS-TuM6 Quantum Transport in Metallic Nanowires Fabricated by Electrochemical Deposition/Dissolution, *N.J. Tao*, *C.Z. Li*, *A. Bogozi*, *J. D'Agnese, B. Duong*, Florida International University

A non-mechanical method for fabricating a metallic narrow constriction between two electrodes using electrochemical deposition is described. The width of the constriction can be adjusted by slowly dissolving metal atoms away or re-depositing atoms onto the constriction which can be controlled flexibly by the electrodes' potentials. Well-defined plateaus near the integer numbers of the conductance quantum have been observed in these constrictions at room temperature. Since no mechanical movements are involved, nano-constrictions with long term stability have been fabricated. @FootnoteText@ Financial support is acknowledged through grants from AFSOR (F49620-96-1-0346) and NIH (GM-08205).

# 10:20am NS-TuM7 Conductance of Molecular Junctions, M.A. Reed, Yale University INVITED

The charge transport and conductance measurement of a single atom or moiety, is an intriguing, experimentally challenging, and long sought goal. We have developed a number of techniques for the electrical measurement of single and/or few molecule systems. First, we have measured the electrical transport properties of a single molecule selfassembled onto the electrodes of a mechanically controllable break junction, allowing for direct observation of charge transport through the molecules. Current voltage I(V) measurements at room temperature demonstrate a highly reproducible apparent gap at about 0.7 V and a corresponding resistance of 22 MOhms. Second, we have developed a nanostructure device technique called a "nanopore" approach to measure the electronic transport of a class of stable self-assembled conjugated oligomers. This has allowed us to determine the barrier heights and transport mechanisms in these conjugated oligomer systems. We have also measured for the first time the molecular equivalent of a resonant tunneling device, and experimental results on a new molecular switching mechanism.

# 11:00am NS-TuM9 Carbon Nanotubes: Manipulation, Properties and Functional Electronic Devices, *R. Martel, T. Hertel, T. Schmidt, H. Shea, Ph. Avouris,* IBM T.J. Watson Research Center

Carbon nanotubes (CNT) are materials with unique properties. Depending on their atomic structure, their electronic structure can be that of a metal or semiconductor, and this coupled with their extreme mechanical strength and high thermal conductivity makes them ideal candidates for novel nanoelectronic devices. While discussions of the CNT properties are usually confined to isolated perfectly symmetric tubes, CNT are supported on a solid substrate in actual applications. We will first explore the changes in atomic structure (axial and radial deformations) that result from the adhesion forces between the CNT and the substrate. For this we employee AFM measurements, molecular mechanics and electronic structure calculations. We will show that the deforma tions are significant and can have important consequences for the electrical transport properties of CNT. Next, we will demonstrate that by using the AFM we can manipulate not only the position but also the shape of individual CNTs and in this way fabricate model nano-electronic devices. We will demonstrate a field effect transistor based on a single nanotube (CNT-FET) connected via Schottky barriers to gold electrodes. The resulting band-bending can be controlled by a gate to change the source-drain current by four orders of magnitude at 300 K. Nanotube-based single electron transistors (CNT-SET) will also be demonstrated.

#### 11:20am NS-TuM10 Simultaneous Study of the Formation and Conductance of Single Wall Carbon Nanotube at STM Tunnelling Gap by Transmission Electron Microscopy, J. Yamashita, H. Hirayama, Y. Oshima, K. Takayanagi, Tokyo Institute of Technology, Japan

It is theoretically predicted that the single wall carbon nanotube (S WNT) has metallic and semiconducting property depending on its helicity, or diameter. Little experimental study has been done on the electric properties of the SWNT, although several works reported those of the carbon nanotubes. To study the structure and conductance of the SWNT simultaneously, we devised a miniaturized scanning tunnelling microscope (STM) in a UHV transmission elecron microscope (UHV-TEM). We observed formation process of SWNT and measured its conductance and I-V characteristics. The miniaturized STM had two tungsten tips, and graphitized carbon layers adhered to the surfaces of the both tips. The STM tip was touched to other tip and withdrawn from. At the moment of the touch and withdrawal, a SWNT with diameters 1~5nm was formed to bridge the both graphitized layers. The bridge of the SWNT grew and its conductance decreased as the withdrawal of the STM tip. The I-V characteristics were measured(-1.5~1.5V) in the course of the withdrawal of each SWNT. These I-V characteristics were found to fit with a formula, I=@alpha@V(1+ßV@super 2@). We calculated the resistivity for each SWNT by @pi@dt/l@alpha@. Here, l, d and t=0.17nm are the length, diameter, and thickness of each SWNT, which were measured from TEM images. The diameter is an average value for a SWNT, because each SWNT has shapes like coca-cola bottle. We found that the resistivities were from 0.0001 to 0.01(@OMEGA@cm). The magnitude of the resistivity is of high doped semiconductors. This result suggests that the coca-cola bottle like SWNT has metallic part and semiconducting part which are mixed alternately along the SWNT axis.

# **Tuesday Afternoon, November 3, 1998**

Biomaterial Interfaces Group Room 326 - Session BI+AS+MM+NS+SS-TuA

#### Nanoscale to Mesocale Biomaterial Structures

Moderator: M.J. Tarlov, National Institute of Standards and Technology

#### 2:00pm BI+AS+MM+NS+SS-TuA1 Self-Assembly of a Multidomain Protein: Fibronectin at Lipid Model Interfaces, V. Vogel, G. Baneyx, University of Washington INVITED

Fibronectin, an adhesion protein with multiple recognition sites, mediates cell attachment to synthetic and biological surfaces. In solution, fibronectin exists in a globular state where most of its recognition sites are buried in the protein core. Surface adsorption induces conformational changes in the protein that expose many of these sites. Furthermore, it is known that on the surface of cells fibronectin assembles into detergent insoluble fibers, which are considered to be the main functional form of the protein. Fibronectin is hence a prime example of a protein with multiple recognition sites that can be regulated through environmental control. Unfortunately, the molecular pathways of activation and self-assembly are still poorly understood. We have recently found that fibronectin can self-assemble into fibrillar networks at receptor-free phospholipid monolaver interfaces under physiological conditions. This is a crucial observation since the paradigm in biology is that fibril assembly of fibronectin is mediated by membrane-bound receptor molecules. Availability of a simplified model system allows investigation of the molecular pathways by which appropriate surfaces can activate fibronectin and facilitate self-assembly.

#### 2:40pm BI+AS+MM+NS+SS-TuA3 Nanofabricated Substrates for Probing Single Biomolecules by Surface Enhanced Raman Scattering, *S. Petronis, L.K. Hedberg, H. Xu, M. Käll, B. Kasemo,* Chalmers Univ. of Technology and Univ. of Gothenborg, Sweden

The effect of Raman scattering enhancement when coherent laser light interacts with molecules attached to rough surfaces and microscopic metal domains has been known for more than two decades and is called Surface Enhanced Raman Scattering (SERS). The intensity of the Raman signals for such molecules is frequently enhanced by a factor 10@super 5@-10@super 6@ at best.@footnote 1,2@ However recently much larger enhancement factors, in the range 10@super 14@-10@super 15@, have been observed for molecules adsorbed on colloidal silver particles of specific dimensions.@footnote 3,4@ This giant enhancement allows the recording of vibrational spectra from a single molecule for the first time, instead of the ensemble averaged spectra from many molecules, which are normally obtained in optical spectroscopies. Here we report on an attempt to use nanolithography to fabricate structures of silver in the size range 100 - 200 nm and having different shapes in order to explore the size and geometry dependence of the SERS effect. Microfabricated structures which give the highest enhancement could be used for probing different biomolecules and perhaps designing a biosensor. SERS active substrates were prepared as arrays of silver particles on a Si wafer. Within each array the silver particles had a constant shape, size and separation. Three particle shapes (circular, triangular and square), two particle sizes (100 nm and 200 nm), and five different particle separations (10, 50, 100, 150 and 200 nm) were produced by electron beam lithography with a double-layer resist system and "lift-off" procedure. A reference area of uniformly deposited Ag film mimicked an infinite silver surface. The final structures and the chemical composition of the silver particles were characterized by Scanning Electron Microscopy (SEM) and Auger electron spectroscopy (AES), respectively. Preliminary Raman scattering experiments have been performed on the dye-molecule Rhodamin 6G adsorbed on the nanofabricated substrates. A giant enhancement of the Raman signal was observed on all patterns, but not on the Ag film or the Si surface. @FootnoteText@ @footnote 1@M.Moskovits, Rev. of Mod. Phys., vol. 57, No 3, 1985, pp 783-826 @footnote 2@A.G.Mal'shukov, Phys. Rep., vol 194, Nos 5&6, 1990, pp 343-349 @footnote 3@K.Kneip et al., Phys. Rev. Lett., vol. 78, No 9, 1997, pp1667-1670 @footnote 4@S.Nie, S.R. Emory, Science, vol. 275, No 21, 1997, pp 1102-1106

#### 3:00pm BI+AS+MM+NS+SS-TuA4 Nanostructured Surfaces for Biorecognition - A Novel Templating Approach, H. Shi, B.D. Ratner, University of Washington

Materials that specifically recognize proteins may find a variety of applications in separations, sensors and medical materials. Molecular imprinting provides an intriguing approach to plastic antibodies against small molecules, but the use of proteins as templates has been less successful in making protein recognition materials. In this study, nanostructured surfaces with tailored protein-binding cavities are prepared

by an imprinting technique based on RF-plasma deposition of organic thin films. A polysaccharide-like surface with protein-imprinted nanopits allows only the template protein to fill the pits, and to bind strongly, because the nanopits are complementary to the template protein in shape and in the distribution of functional groups. The bound protein in its pit is prevented from exchange with protein in the solution due to a strong binding and steric hindrance, while the non-template protein that is weakly adsorbed on the surface is displaceable. Atomic force microscopy (AFM) and transmission electron microscopy (TEM) showed that nanometer-sized pits, in the shape of imprinted proteins, were created on the surfaces of our protein-imprinted polymer films. Imprinting fidelity was confirmed by AFM analysis of imprints of monodisperse colloidal gold nanoparticles. Electron spectroscopy for chemical analysis (ESCA) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) indicated that template proteins were washed off the surfaces of protein imprints while sugar molecules were covalently incorporated. Radiolabeled -protein adsorption showed that a protein imprint recognized its template protein from a binary mixture with a high specificity. This study illustrates a novel templating strategy for biological molecules that can be exploited for fabrication of biorecognition materials.

#### 3:20pm BI+AS+MM+NS+SS-TuA5 Sensing and Analyzing Single Molecular Interactions with Microfabricated Devices@footnote 1@, J.-B.D. Green, G.U. Lee, Naval Research Laboratory INVITED

There is an intense effort to create new tools for manipulating and characterizing single macromolecules because of the power that these techniques can bring to the analysis of biological macromolecules. Due to the high force and displacement sensitivity of the atomic force microscope (AFM) it has been used to measure inter- and intramolecular forces between model ligand-receptors, i.e., streptavidin-biotin, complimentary strands of DNA, and biologically relevant supra-molecular structures, i.e. titin. With the success of these measurements, there are efforts to obtain even more detailed force measurements and to establish these techniques in the biotechnology laboratory. Our efforts focus on: 1. Designing force transducers with force (10@super -12@N), time (10@super -5@s) and spatial (10@super -9@m) resolutions that push the thermal noise envelope. 2. Developing immobilization strategies that produce more reliable force measurements. We will discuss two new microfabricated devices under development in our laboratory. The first microfabricated apparatus offers an excellent platform for detailed measurements of intermolecular interactions and possibly even analysis of combinatorial arrays. The second is an ultra-sensitive detector based on piezoresistive force transduction and magnetic microparticles. The future of these and similar devices will be considered. @FootnoteText@ @footnote 1@This work has been conducted in collaboration with Alexey Novoradovsky, Jonah Harley, Mohan Natesan, Steven Metzger, David Baselt, and Richard Colton.

4:00pm BI+AS+MM+NS+SS-TuA7 Nanomechanical Properties of Cellular Components Determined by Interfacial Force Microscopy, P.R. Norton, K de Jong, J.F. Graham, N.O. Petersen, University of Western Ontario, Canada The cell membrane is the contact surface between the cell's internal environment and the outside world. Increasingly it is recognized the there is strong active coupling between mechanical properties and cellular functions in properties such as locomotion and adhesion and in cytoskeletal diseases such as muscular dystrophy.@footnote1@ There is therefore an urgent need to understand the mechanical properties of cells and cellular subcomponents at length scales << 1µm. We will describe our initial experiments to achieve this goal. We have used three different imaging techniques in our investigation of the nanomechanical properties of larynx cells. First, immunofluorescent labelling was used to permit visualization of specific cell components in the confocal microscope, for example to determine whether the cell nucleus was removed in a shearing process. The same cell was then imaged in the atomic force microscope (AFM), permitting identification of components involved in motion such as microspikes. The nanomechanical properties of cells were then studied by using nanoindentation the interfacial force microscope (IFM).@footnote2@ While we have not yet succeeded in imaging and measuring the same cell used in the confocal and atomic force microscopies, we have demonstrated the feasibility of our approach and have obtained quantitative force-distance curves on different regions of a single cell fixed in paraformaldehyde, sodium periodate and lysine, which cross-links the proteins. From these data we can derive the elastic modulus, hardness etc of the specific region of the cell. The modulus of such a cell was ~ 3GPa, comparable to a soft polymer. Similar measurements are planned on unfixed cells. @FootnoteText@

### **Tuesday Afternoon, November 3, 1998**

@footnote1@Chen, C.S., et al. Science 276, 1425 (1997) @footnote2@Warren, O.L., et al. Physics in Canada 54, 122 (1998)

#### 4:20pm BI+AS+MM+NS+SS-TuA8 Unbinding Force of NTA-M@super 2+@--Histidine Complexes. The His-Tag Immobilization Force, J.G. Forbes, P. Yim, University of Maryland, College Park

A sequence of six or more histidines will bind tightly to a Cu, Ni, or Co complex. The compound typically used to immobilized the metal is N-(5amino-1-carboxypentyl)iminodiacetic acid (NTA). Most proteins will not bind to the complex unless there is a sequence of histidines, which is readily added using recombinant DNA techniques. The histidine tag may be removed from the metal complex with a high concentration of imidazole or by protonating the histidines at a pH below 6. We have studied the the unbinding strength of this interaction with the atomic force microscope (AFM). To perform this measurement, we have functionalized silicon nitride AFM tips with NTA-M@super 2+@. A glass slide was coated with recombinant DNAse I with a his-tag on the C-terminus. Unbinding force measurements were made in phosphate buffered saline (PBS) to reduce electrostatic interactions. We find that the unbinding force for the NTA-M@super 2+@/His-tag interaction to be ca. 85~pN for each of the metal complexes. Interestingly, 0.5~M imidazole does not remove the interaction, but only changes the distribution of the measured forces. This is a result of the non-equilibrium condition of the tip being forced into the protein coated surface. The interaction is almost completely removed by lowering the pH to 5.0 where the histidines are protonated and can no longer coordinate with the nickel. The remaining interaction forces are due to the histidines which are exposed when the tip presses into the surface. These results provide a quantitative measurement of mechanical strength of binding of proteins to surfaces functionalized with NTA-M@super 2+@.

#### 4:40pm BI+AS+MM+NS+SS-TuA9 Sieving of DNA Molecules in Nanofluidic Channel, J. Han, H.G. Craighead, Cornell University

Entropic trapping and sieving effect of long DNA molecules was studied in variable thickness nanofluidic channels. We used photolithography and etching techniques to define fluid channels on Si wafers, and anodic bonding method to seal the channel with a thin pyrex glass coverslip. The channel consists of alternating regions with two different channel thicknesses(~100nm and 1.6µm). We studied electrophoretic motion of lambda phage DNA in this channel by epi-fluorescence microscopy. Since the radius of gyration of a typical long DNA molecule is larger than the smaller gap of the channel, the shallow part of the channel can be an entropic barrier for DNA motion. Therefore, DNA molecules were retarded when they entered into the thin region from the thick region. We measured the mobility of DNA molecules in these channels and observed that below a certain electric field, mobility of DNA molecule decreased to near zero drastically, showing that DNA molecules be entropically trapped and sieved. The threshold electric field was mainly dependent on the geometry of channel(e.g. gap size) and the length of DNA driven. This suggests a new type of separation device for DNA and other polymers.

#### 5:00pm BI+AS+MM+NS+SS-TuA10 Detection of Molecular Ion and Quantification of Pentapeptide on Plasma Hydroxylated Fluoropolymer by Time of Flight Secondary Ion Mass Spectrometry, J.A. Gardella, L.M. Sun, State University of New York, Buffalo

Abstract: Poly(hexafluoropropylene-co-tetrafluoroethylene) (FEP) was modified by a hydrogen/methanol radio frequency glow discharge plasma. Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) was employed to characterize the modified FEP surface and three pentapetides (YGGFM, YGGFL, YIGSR) which were microsyringe deposited on the modified FEP film. New fragments of OH (CF@sub 2@)n in negative ion SIMS of the modified FEP film indicated that -OH functional group had been incorporated on the FEP surface after plasma treatment. In the positive ion SIMS of three pentapeptides on the hydroxylated FEP film, protonated molecular ions were dominant signals from the peptides whereas not many fragments were observed either from the peptides or the impurity. Sodium and potassium adduct molecular ions were detected as well as oxidized protonated molecular ion of YGGFM in the positive ion SIMS spectrum. Negative ion SIMS of YGGFL yielded a deprotonated molecular ion. The mixture of these three pentapetides was also studied by TOF-SIMS. The relative intensity of protonated molecular ions of YGGFL, YGGFM and YIGSR showed the possibility of quantification on the hydroxylated fluoropolymer by TOF-SIMS. As a study of substrate effects, TOF-SIMS spectra of these peptides on oxidized Ag substrate were recorded. Comparing SIMS results of pentapetides on Ag and on modified FEP film, fewer fragments occurred from the FEP film than that from the Ag substrate. A substrate like the FEP

fluoropolymer might be beneficial for the quantification of peptides because of the intensity of parentlike species in SIMS measurement.

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS-TuA

#### **Quantum Wires and Quantum Dots**

Moderator: Ph. Avouris, IBM T.J. Watson Research Center

#### 2:00pm NS-TuA1 Self-organized Ge Quantum Wires on Si (111) Substrate,

G. Jin, Y.S. Tang, J.L. Liu, K.L. Wang, University of California, Los Angeles Self-organized nanostructures have been of considerable interest recently due to the fact that self-organization provides a possible way to realize nanostructures without process-induced damage as frequently seen in those defined by electron beam lithography and reactive ion etching. However, very few work has been reported on self-organized quantum wires. In this work, self-organized Ge quantum wires on Si (111) substrate grown by MBE have been realized and studied. Regular surface steps were formed on cleaned Si (111) substrate after annealing at 870°C in a UHV MBE system, then selforganized Ge quantum wires were formed on the substrate after growing 300 nm Si buffer layer. Atomic force microscopy (AFM) studies showed that regular surface steps were formed along [11 -2] direction and the terrace width was about 120 nm and the step height was about 0.6 nm. The self-organized Ge quantum wires with the width of about 65 nm and the height of about 5.5 nm were parallel to the direction of [11 -2] with the pitch close to the terrace width, or about 120 nm. The uniformity of the quantum wires was found to be reasonably good. Raman studies indicated that the peak of Ge-Ge mode shifted to a higher energy. This suggests that the Ge quantum wires are tensilely strained on the edge sites of the steps. The result of polarized Raman studies confirmed the existence of the wires and the wire orientation which is consistent with the AFM result. The size distribution and the optical properties of the Ge quantum wires are also under study. @FootnoteText@ The work was supported in past by National Science Foundation (DMR-9520893)

#### 2:20pm NS-TuA2 Formation and Characterization of Metal Atom Nanostructures on Si(112) Facet Surfaces, S.M. Prokes, O.J. Glembocki, Naval Research Laboratory

Facet semiconductor surfaces have been suggested for use as templates in the formation of ordered dots and lines. A bulk-terminated Si(112) surface is of particular interest since it consists of distinguishable (111) terraces and (001) steps which can serve as a template for the formation of wellordered nanostructures, such as metallic wires or dots, with the aim of producing electron gratings and magnetic nanostructures. We have studied the formation of Ga, Al and Sc nanostructures on facet Si(112) surfaces, which were investigated using LEED, Auger spectroscopy and Reflectance Difference Anisotropy (RDA). Although the clean Si(112) surface exhibits (1x2) reconstruction, we found that the deposition of Ga or Al above 300°C removes this reconstruction and leads to a periodic stepped structure of alternating (111) terraces and (001) steps. Ga or Al chains then form by a self-limiting process, which we can track from the rapid change of the (2x1)Si(112) reconstruction under sub-critical coverage, to chain formation leading to a 5x1 reconstruction followed by a 6x1 reconstruction, using RDA. Furthermore, AES and RDA results show the replacement of Ga atoms by Al atoms at the step edges during sequential deposition of Ga and Al, indicating a stronger Al-Si bond. Using RDA, we have also observed that depositions at lower temperatures can lead to the formation of Ga metallic wires on the Si(111) terraces. For Sc, we find that its higher surface energy precludes the formation of wires but leads to the formation of nanometersize Sc islands, which may exhibit enhanced magnetic moments. Using Monte Carlo techniques to model the time evolution of the deposition at various temperatures, we are also able to extract highly accurate values for the surface kinetic parameters involved in the formation of these nanostructures.

#### 2:40pm NS-TuA3 Nanotubes and Nanowires: Physics, Chemistry and Applications, C.M. Lieber, Harvard University INVITED

One-dimensional nanostructures, nanowires and nanotubes, represent an exciting and intellectually challenging area of research that crosses the borders between many areas of the physical sciences and engineering. Interest in these structures has been driven by fascinating issues in chemistry and physics, and the potential to impact science and technology. For example, it remains a great challenge to understand the intrinsic and potentially unique properties of nanowires and nanotubes, and thereby define new applications. This presentation will focus on addressing these

# **Tuesday Afternoon, November 3, 1998**

critical issues. First, STM studies of the atomic structure and tunneling density of states of single-wall carbon nanotubes (SWNTs) will be described. Measurements show that SWNTs exhibit semiconducting and metallic behavior that depends predictably on helicity and diameter, and also exhibit well-defined 1D van Hove singularities. These results are compared and contrasted with theoretical calculations, and their implications discussed. Second, atomic force microscopy studies of the bending and stretching of individual nanowires and nanotubes will be discussed. The implications of these results on potential structural applications will be discussed. Lastly, the application of nanowires and nanotubes as molecular resolution, functionally-sensitive probes for chemistry and biology will be described.

3:20pm NS-TuA5 Self-Assembled Nanostripes on Silicon, D.Y. Petrovykh, University of Wisconsin, Madison; J. Viernow, Universität Hannover, Germany; J.-L. Lin, University of Wisconsin, Madison; F.M. Leibsle, University of Missouri, Kansas City; F.-K. Men, National Chung Cheng University,Taiwan, R.O.C., Republic of China; A. Kirakosian, F.J. Himpsel, University of Wisconsin, Madison

We report on the successful fabrication of one-dimensional structures on silicon with sizes of a few nanometers. As templates we use stepped Si(111)7x7 surfaces, which can be prepared with high precision (only one kink in 20,000 edge sites).@footnote 1@ On top of such a template, CaF@sub 2@ stripes are produced by step flow growth. They play the role of a photoresist in nanolithography. Various growth modes of CaF@sub 2@ are found by chemically-sensitive scanning tunneling microscopy, including a regime at 600-650@super o@C where regular, 10 nm wide stripes are formed and a second regime at 700-750@super o@C where the stripes break apart spontaneously into strings of 10 nm diameter dots. Chemical sensitivity to CaF@sub 2@ is achieved in STM via current images at a bias voltage where electrons from the tip tunnel into the band gap of CaF@sub 2@. After producing passivating CaF@sub 2@ stripes, metallic wires are to be deposited on the remaining reactive silicon, e.g., by selective CVD, electroplating or evaporation and diffusion off the CaF@sub 2@. Test experiments on these processes will be reported. @FootnoteText@ @footnote 1@J. Viernow, J.-L. Lin, D. Y. Petrovykh, F. M. Leibsle, F. K. Men, and F.J. Himpsel, Appl. Phys. Lett. 72, 948 (1998) @footnote 2@J.-L. Lin, D. Y. Petrovykh, J. Viernow, F. K. Men, D. J. Seo, and F.J. Himpsel, J. Appl. Phys. 84, July 1 (1998)

3:40pm NS-TuA6 Fabrication of Metallic Nanowires via UHV-STM Lithography and Thermal CVD, M.C. Hersam, G.C. Abeln, D.S. Thompson, J.S. Moore, H. Choi, S.-T. Hwang, J.W. Lyding, University of Illinois, Urbana-Champaign

The selective removal of hydrogen from a passivated Si(100) surface with an ultrahigh vacuum (UHV) scanning tunneling microscope (STM) allows nanometer-sized "templates" of clean Si(100) to be defined on an otherwise unreactive surface. By delivering chemically reactive species to the surface in the gas phase, different materials can be selectively deposited on the unpassivated Si(100) areas. In particular, nanopatterned metallization is achieved through selective thermal chemical vapor deposition (CVD) of organometallic precursor molecules. This paper systematically analyzes such precursor molecules to determine their suitability for selective CVD of metal on Si(100) in UHV. Initially, a novel aminoalane precursor was employed for CVD of aluminum at ~200°C. STM images of the surface after exposure to this precursor suggest monolayer coverage and evidence of a 2X2 reconstruction. However, variation of the dose and deposition conditions did not lead to the growth of a thicker film. Hence, in an effort to produce a more receptive surface for the growth of metallic thin films, CVD of nucleating agents (e.g., TiCl@sub 4@) was also studied. XPS and STM data show the selective deposition of Ti on clean versus H-passivated Si(100) after TiCl@sub 4@ exposure at room temperature. In an effort to grow TiN, the TiCl@sub 4@ experiments were repeated on an ammonia coated Si(100) surface. Again, XPS and STM data show the selective deposition of Ti. Finally, multiple precursor molecules were integrated for the growth of multi-layer structures. As a footnote, our efforts for interfacing these STM patterned nanowires with macroscopic external electronics will be updated.

#### 4:00pm NS-TuA7 STM/AFM Nanofabrication Process on Atomically Flat Substrate for Single Electron Device, K. Matsumoto, Electrotechnical Laboratory, Japan INVITED

Planar type single electron transistor(SET) and SET memory are proposed and realized on the atomically flat @alpha@-Al@sub 2@O@sub 3@ substrate using a STM/AFM nanofabrication process. Using STM tip/AFM cantilever as a cathode, the surface of the titanium(Ti) metal which was on an atomically flat @alpha@-Al@sub 2@O@sub 3@ substrate is selectively oxidized to form a few tens of nanometer wide oxidized titanium(TiO@sub x@) line just under the tip. The surface roughness of the 2.5nm thick Ti metal is less than 0.15nm and retains the atomically flat condition. The surface roughness of TiO@sub x@ is also less than 0.15nm. The TiO@sub x@ works as an energy barrier for an electron, and the barrier height between Ti and TiO@sub x@ is 468meV. Therefore, the narrow TiO@sub x@ line could be used for the tunneling junction for SET. The size of the SET island is 8nm x 26nm square. The width, the thickness, and the length of the two tunnel junctions are 19nm, 2nm, and 26nm, respectively. The tunnel junction capacitance calculated from these structure parameters is C@sub t@=0.12aF. The gate electrode is set 964nm away from the island. The SET operates even at room temperature and shows the Coulomb oscillation with the periods of ~1.8V at the drain bias of -0.3V. At the different drain bias from -0.2V to -0.7V, the drain current shows the same oscillation periods of ~1.8V against the gate bias change. From this periods of Coulomb oscillation, the gate capacitance is estimated to be C@sub G@=0.1aF. Owing to the atomically flat @alpha@-Al@sub 2@O@sub 3@ substrate, the uniformity and reproducibility of the TiO@sub x@ line improves drastically, and it makes possible to fabricate the SET memory with complicated multi-tunnel junction structure.

#### 4:40pm NS-TuA9 Raman Scattering Studies of Multiple Ge Dots on Si (100) By Solid Source Molecular Beam Epitaxy, J.L. Liu, Y.S. Tang, G. Jin, K.L. Wang, University of California, Los Angeles

Recently, the growth of Ge dots on Si substrate has attracted much attention due to its potential applications in Si-based optoelectronics and its possible contribution to the scaling-down devices. The optical properties of these dots are particular interest in the investigation. In this work, we report the Raman scattering studies of multiple Ge dots on Si (100) substrate grown by solid source molecular beam epitaxy. The sample contains 20 periods of boron-doped Ge dots with 6 nm Si as barriers. The cross-sectional transmission electron microscopic observations illustrate that the size and height uniformities of the Ge dots are not worse that 7 percent. Raman spectrum shows the upper shift of Si-Ge mode and downward shift of Ge-Ge mode which are attributed to the alloying of the wetting layers and the phonon confinement in the Ge dots, respectively. From the polarization dependence Raman spectrum, we find the possibility of the strong intersubband absorption in the dots.

5:00pm NS-TuA10 Surface Oxidation of Germanium Quantum Dots Produced by a Laser Vaporization-Controlled Condensation Technique, S. Li, S. Wen, M. Wiess, J.A. Carlisle, M.S. El-Shall, Virginia Commonwealth University

Weblike aggregates of coalesced Ge quantum dots are produced by a laser vaporization-controlled condensation technique. The surface oxidation of Ge guantum dots is studied with Fourier-Transform Infrared Spectroscopy (FTIR), core-level X-ray photoelectron spectroscopy (XPS), and x-ray diffraction (XRD). The freshly prepared Ge particles possess the bulk Ge crystal lattice. After the particles are removed from the reaction chamber for 10 min, the surface of the particles becomes oxidized as seen by XPS and FTIR. However, the FTIR peaks are very weak. Further oxidation of the Ge core, by exposure of the Ge quantum dots to air, results in the epitaxial growth of surface oxidation layers, as confirmed from XRD and XPS. After the particles are stored in air for two months, sharp features corresponding to crystalline GeO@sub 2@ are observed in the XRD spectrum. The surface-oxidized Ge quantum dots do not show photoluminescence from the core Ge particles, but rather show the emission characteristics of GeO@sub 2@, with photoluminescence lifetimes less than 20 ns. The photoluminescence is thus attributed to defect states in GeO@sub 2@.

### Wednesday Morning, November 4, 1998

#### 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 forcecompensated 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 $\mu$ m long and 0.1 $\mu$ m 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  $\mu$ m 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

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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  $\mu$ m. Factors influencing the optical and topographic resolution characteristics 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)

### Wednesday Afternoon, November 4, 1998

#### Magnetic Interfaces and Nanostructures Technical Group Room 324/325 - Session MI+NS-WeA

#### Nanoscale Magnetics: Imaging and Fabrication Moderator: S. Foss, Seagate Technology

#### 2:00pm MI+NS-WeA1 Using the Magnetic Force Microscope as a Quantitative Micromagnetic Probe, *R. Proksch*, Digital Instruments INVITED

The Magnetic Force Microscope (MFM) has developed into a popular tool for nanometer scale resolution imaging of a wide variety of magnetic samples. The routine <50nm spatial resolution rivals and sometimes exceeds electron based microscopies while not requiring operation in a vacuum or special sample preparation. Since the MFM is sensitive to the external magnetic field gradients of a sample, however, it does not directly yield quantitative values of either the external field or a sample's magnetization. A recent advance@footnote 1@ has allowed guantitative imaging of the localized field from a sample. It based on a magnetically soft tip that acts as a fluxgate sensor. An external field is applied to the MFM tip and sample until the response of the MFM is zeroed. This zeroing occurs when the external field cancels the local field at the MFM tip. The resulting quantitative images have the same spatial resolution of the MFM. Another recent development in MFM was the realization that the energy dissipated by an oscillating cantilever was quantifiable.@footnote 2,3@ Measurements of the energy dissipated by the MFM tip have been quantitatively compared to micromagnetic models.@footnote 4@ Measurements of other fundamental quantities such as the moment of a single magnetic particle and nucleation volumes in relaxing domain structures through dissipation observations will be presented. @FootnoteText@ @footnote 1@R. Proksch, G. Skidmore et al., Appl. Phys. Lett. 69, 2599 (1996). @footnote 2@P. Grutter, Y. Liu, P. LeBlanc, and U. Durig, Appl. Phys. Lett. 71, 279 (1997). @footnote 3@J. P. Cleveland et al., Appl. Phys. Lett. in press (1998). @footnote 4@Y. Liu, B. Ellman and P. Grutter, Appl. Phys. Lett. 71, 1418 (1997).

#### 2:40pm MI+NS-WeA3 Imaging Current Flow in Polycrystalline Bi2Sr2CaCu2Ox Superconductors by Magnetic Force Microscopy, F. Král, D. Perednis, ETH Zürich, Switzerland; D.A. Bonnell, The University of Pennsylvania, US; G. Kostorz, L.J. Gauckler, ETH Zürich, Switzerland

The measurement of magnetic fields induced by current flow can be used to visualize current transport paths in complex microstructures. Magnitudes of fields induced by currents typical of metallic conductors and of superconductors are within the range accessible by magnetic force microscopy. Finite element calculations indicate that conducting grains separated by as little as a hundred nm will be distinguished. The fields emanating from current in the complex textured microstructure of a Bi2Sr2CaCu2Ox based thick film in the superconducting state at temperatures below 60 K were clearly delineated. Magnetic field variations with the size and orientation of the textured grains that carry current were quantified. Obstructions to current flow are imaged.These measurements were accomplished on a commercial instrument modified to connect to a He cryostat and operate in medium vacuum.

# 3:00pm MI+NS-WeA4 Imaging Magnetic Domains by Spin-Polarized Scanning Tunneling Spectroscopy, *M. Bode*, *M. Getzlaff*, *R. Wiesendanger*, University of Hamburg, Germany

The concept of spin-polarized scanning tunneling spectroscopy (SP-STS) promises the unique capability of magnetic imaging with a resolution down to atomic scales. We will show that the (0001)-surface of Gadolinium, which has a bulk Curie-temperature T@sub C@ = 293K, is ideally suited for the realization of SP-STS since Gd(0001) exhibits a d@sub z@@super 2@like surface state. This surface state is exchange split in an occupied majority (spin-up) and an empty minority (spin-down) spin-part below T@sub C@. Already in a previous publication we have shown that both spin-parts appear as a double-peak structure in the tunneling spectra.@footnote 1@ Here we report on our experiments with magnetic thin film probe tips. In accordance with the spin-valve effect@footnote 2@ we found characteristic variations in the tunneling spectra which correlate with the direction of the external field, i.e. the differential conductivity of the particular spin-part of the surface state being parallel with the tip is enhanced on the expense of the counterpart being antiparallel. This allows the imaging of magnetic domains with the STM. The resolution obtained so far is approximately 20nm. The measured spin-asymmetry of approximately 40% (20%) at the majority (minority) part of the surface

state is in good agreement with former spin-resolved (inverse) photoemission experiments. We will show that the application of thick Fecoatings on the tip leads to a sudden contrast reversal probably caused by a switching of sample domains due to the strong magnetic interaction between tip and sample. @FootnoteText@ @footnote 1@R. Pascal, Ch. Zarnitz, M. Bode, M. Bode, and R. Wiesendanger, Appl. Phys A 65, 603 (1997). @footnote 2@M. Julliere, Phys. Lett. A 54, 225 (1975).

#### 3:20pm MI+NS-WeA5 Imaging Magnetization in Fe and Layered Fe/Co Films Using an Element-Specific Scanning Transmission X-Ray Microscope, J.B. Kortright, S.-K. Kim, T. Warwick, G. Meigs, Lawrence Berkeley National Laboratory

Magnetization distributions in demagnetized polycrystalline Fe films and in the individual Fe and Co layers of layered films were imaged with a scanning transmission x-ray microscope and circular polarizing filters using the strong magnetic circular dichroism at the Fe and Co 2p3/2 levels. Transmission images were obtained at roughly 200 nm resolution with high contrast that was reversed by reversing the saturated magnetization in the polarizing filters. Large, regular 180 degree domains dominate Fe films 20-30 nm thick. Smaller magnetization features (swirls, ripples, etc.) are observed at grain boundaries and near the tip of needle-shaped domains growing into or being consumed by larger domains. In layered films consisting of Fe and Co layers separated by a 2 nm SiC spacer the magnetization in each layer is entirely different from the single Fe film, revealing significant interaction between the two different layers in the demagnetizing process. Large 180 degree domains are absent, and are replaced by much smaller, more irregular magnetization distributions having characteristic dimensions of several microns and somewhat resembling stripe domains. The domains in the Fe and Co layers show some degree of spatial correlation, and some degree of antiferromagnetic alignment. These first imaging studies using a scanning transmission x-ray microscope in conjunction with a high resolution grating monochromator complement other recently demonstrated imaging techniques using x-rays, and point to new opportunities to quantitatively study magnetization distributions in a variety of samples. Technical aspects underlying these new capabilities will be reviewed. This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Division of Materials Science, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

3:40pm MI+NS-WeA6 Substrate and Growth Related Nanostructural and Magnetic Properties in La@sub 0.67@SR@sub 0.33@MnO@sub 3@ Thin Films, *M.E. Hawley*, *G.W. Brown*, *C. Kwon*, *Q.X. Jia*, Los Alamos National Laboratory

Beyond achieving a target chemical composition, optimization of metal oxide thin film properties depends on a number of growth-determined factors: microstructure, defects, and stress. For CMR materials, these factors can lead to low Curie temperature, non-ideal temperaturedependent magnetization, undesirable domain structures, higher coercivity, and magnetic anisotropy. In particular, growth of these materials, which possess fairly large positive magnetostrictive constants, on lattice-mismatched substrates can result in residual stress-induced mazelike domains. This type of domain was observed by magnetic force microscopy (MFM) for some La@sub 0.67@Sr@sub 0.33@MnO@sub 3@ films grown on LaAlO@sub 3@ (compressive mismatch) and tied to substrate-induced stress and film thickness. Stress-induced elongation of the out-of-plane lattice parameter may be necessary but is not sufficient to produce these domains. Their existence has also not been correlated with processing parameters. To address some of these issues, we have grown films over a range of temperatures by pulsed-laser deposition on LaAlO@sub 3@ and SrTiO@sub 3@ (tensile mismatch) to determine the correspondence of lattice-induced strain and degree of granularity to magnetic properties. Nanostructure characterization (STM, AFM, and MFM) magnetization, and coercivity will be presented to show the relationship between growth and properties. Maze-like domain structures, with 150 to 200 nm separations, were observed for thicker films grown at 800@degree@C on LaAlO@sub 3@ versus weak diffuse domains for thin films and all films grown on SrTiO@sub 3@. Application of an increasing inplane external magnetic field converted the maze-domains first into stripe domains with decreased spacing (with reduced out-of-plane magnetization) and then into diffuse in-plane structures. Field orientation versus magnetic structures will be included.

### Wednesday Afternoon, November 4, 1998

4:00pm MI+NS-WeA7 Monodisperse Cobalt Nanocrystals and Their Assembly into Nanocrystal Superlattices: Building with Magnetic Artificial Atoms, C.B. Murray, S. Sun, IBM T.J. Watson Research Center INVITED We present chemical methods which yield cobalt nanocrystals uniform in size to + or - one lattice constant while simultaneously controlling crystal shape, structure and surface passivation. We use high temperature (200 -300° C solution phase synthesis and size selective processing to produce organically passivated nanocrystals with size distributions less than 5%. These monodisperse transition metal nanocrystals self-organize during controlled evaporation to produce three dimensional superlattices (colloidal crystals, opals). The cobalt nanocrystals resemble "artificial atoms" sitting on regular close-packed superlattice sites, each separated by a selected organic spacer. The superlattices retain and enhance many of the desirable mesoscopic properties of individual cobalt nanocrystals and provide a model system for studies the electronic coupling of neighboring particles. The inter-particle spacing can be varied from intimate contact up to 40 Å separation. Superlattices can be prepared as either faceted colloidal crystals or as ordered nanocrystal thin films on a variety of optically and electronic addressable substrates (sapphire, silicon, etc.). Structural and magentic investigations of both dispersed and assembled nanocrystal systems will be presented.

#### 4:40pm MI+NS-WeA9 Fabrication and Characterisation of Micron Scale Magnetic Features, C.N. Borca, P.A. Dowben, University of Nebraska, Lincoln

Different methods can be adopted to fabricate patterned thin films with features spatially restricted in the micron-scale regime. We are studying ferromagnetic films of cobalt and cobalt - palladium heterostructures fabricated by selective area deposition from organometalic compounds. We have developed this one-step deposition technique sufficiently to deposit pure metal features with excellent spatial resolution and in multilayer geometries. From the comparison between the continuous and patterned films we can conclude that the patterning of the films into arrays of discrete micron-scale features has a greater influence on the magnetic properties of the films than changes microstructure and film growth. We propose that this organometallic chemical vapor deposition (CVD) method represent a new approach for novel devices fabrication.

#### 5:00pm MI+NS-WeA10 Domain Behavior in Magnetic Nanostructures as Revealed by MOIF Observations, *R.D. Shull*, *A.J. Shapiro*, National Institute of Standards and Technology; *V.I. Nikitenko, V.S. Gornakov*, Institute of Solid State Physics RAS, Russia

A magneto-optical indicator film (MOIF) technique has been used for imaging magnetic domains and applied to magnetic nanostructures, including granular metals, magnetic multilayers, and antiferromagnet (AF)/ferromagnet (FM) bilayers. In this technique, the sample domains are imaged by their effect on a garnet film with in-plane magnetization located immediately above the sample. In addition to static domain structures, dynamic information has been obtained by monitoring the domain pattern evolution upon the application of an external magnetic field. Fractal type domain walls were observed in Co/Ag granular metals with a two-step remagnetization process, non-homogeneous nucleation processes were observed in AF/FM bilayers with remagnetization behavior dependent upon field direction, and non-collinear spin configurations were detected in Cu/Co multilayers (electrodeposited on Si substrates) displaying giant magnetoresistance (GMR) effects during the remagnetization process. In these latter samples, the GMR magnitude was correlated with the spin reorientation mechanism. In all samples the effects of crystal lattice defects on the remagnetization process was documented, and found to be significant. The MOIF technique was also found to be capable of detecting not only the domain structure of the surface layer, but also that of subsurface layers in a multilayer morphology. In this presentation, a review of the domain statics and dynamics which have been observed in a variety of nanostructured material types will be discussed. Particular attention will be given to the origin of enhanced coercivity in a bilayer system with unidirectional anisotropy.

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS-ThM

#### Nanoscale Patterning and Modification

Moderator: J.F. Wendelken, Oak Ridge National Laboratory

8:20am NS-ThM1 Fabrication of Nanometer Size Photoresist Wire Patterns with a Silver Nanocrystal Shadowmask, S. Choi, K.L. Wang, University of California, Los Angeles; M. Leung, G. Stupian, N. Presser, The Aerospace Corporation; S. Chung, G. Markovich, S. Kim, J.R. Heath, University of California, Los Angeles

We propose a new method of fabricating precisely defined nanometer size photoresist wire patterns by exposing electron beams onto silver(Ag) nanocrystal wires which have already been deposited on photoresist film. Ag nanocrystals linked with organic ligands were fabricated first using an organically functionalized Ag nanocrystal technique. Arrays of high aspect ratio wires formed spontaneously at the air/water interface when these Ag nanocrystals were dropped onto water. Ag nanocrystal wire structures were then transferred onto the polymethyl methacrylate(PMMA) coated substrates by the Langmuir-Blodgett lift-off process. To prepare a thick Ag nanocrystal shadowmask, a second monolayer of Ag nanocrystal wires was added to the previously deposited monolayer. The occupied areas by organic ligands between the nanocrystals in the wire decreased through a metallization process in air. Monte Carlo simulation was done to estimate the electron stopping effectiveness for the Ag nanocrystal shadowmask at low voltages. Low energy electron beam exposure resulted in numerous 50nm wide wire patterns on photoresist film. The wire patterns were defined in the photoresist material by spatially selective electron beam exposure on the Ag nanocrystal wire shadowmask. The wire patterns on photoresist film can be used for pattern transfer to many different substrates by subsequent selective etching for use in device fabrication. Our new method therefore enables rapid and low cost fabrication of quantum wire structures.

#### 8:40am NS-ThM2 Fabrication and Structuring of Ordered Two-Dimensional Nanopore Arrays in Anodic Alumina, A.P. Li, F. Mueller, A. Birner, K. Nielsch, U.M. Goesele, Max-Planck-Institute of Microstructure Physics, Germany

We will describe the fabrication of nonlithographic nanopore arrays in anodic alumina with areal pore densities in the 6\*10@super 8@ - 5\*10 @super 10@ cm @super -2@ range, and the lithographic structuring of the arrays for potential applications in photonic crystal. A two-step anodization process was used to oxidize aluminum in oxalic, sulfuric, and phosphoric acid solutions. Self-organized hexagonal pore arrangements were formed in the end of the first anodization process. After removing the irregular upper part, the densely ordered pits in the bottom of anodic layer act as natural masks, and the nanopore arrays were fabricated by the second anodization process. Perfect ordered pore arrays were obtained within domains of a few micrometers, which are separated from neighboring domains with different orientation of the pore lattice by grain boundaries. i.e., the nanopore arrays show polycrystalline structure. The pore distance can be controlled by changing the anodic electrolyte and voltage. The ratio of pore diameter and wall thickness can be adjusted by chemical etching after anodization. The structural characteristics make the ordered porous alumina a potential two-dimensional photonic crystal material for the visible to ultra-violet light range. Although they are polycrystalline, these structures are expected to exhibit interesting photonic crystal properties analogous to electronic properties of polycrystalline semiconductors. For optical transmission measurements, light has to be coupled in perpendicular to the pore arrays and to traverse a well defined number of pore layers. For this purpose we have developed a lateral structuring technique that allows to remove the porous alumina precisely yielding vertical walls. Bars of porous alumina which are 100-400  $\mu$ m wide, 100-300 µm high and several mm long have been prepared. The achieved samples are well suited to investigate the optical properties of these structures with light traveling perpendicular to the pore arrays.

9:00am NS-ThM3 Chemically Assisted Ion-Beam Etching of Submicrometer Features in GaSb-based Quantum Wells, G. Nagy, R.U. Ahmad, M. Levy, R.M. Osgood, Jr., Columbia University; M.J. Manfra, G.W. Turner, Massachusetts Institute of Technology

GaSb-based semiconductor systems are of interest because of their potential applications in advanced microelectronic and optical devices. For example, GaSb-based materials have been used for the fabrication of resonant interband tunneling devices, as well as high performance laser diodes, and midinfrared photodetectors. Anisotropic, high-resolution dry etching is a desirable processing technology for device fabrication in this semiconductor system. Here, we have used electron beam patterning and chemically assisted ion beam etching to fabricate structures down to 200 nm in diameter in GaSb and in GaInAsSb/AlGaAsSb multiple quantum well material. The chemically assisted ion beam etching was performed with chlorine as the reactive gas and Ar@super +@ ions of 400-900 eV energy. With the GaSb substrate, the Cr masks used to fabricate the features exhibited good etch selectivity and smooth, highly anisotropic structures were realized. The measured etch rates of GaSb were successfully fitted to a model of chlorine-based chemically assisted ion beam etching that assumes the formation and desorption of trichloride etch product species. With the GaInAsSb/AlGaAsSb multiple quantum well material, the chemically assisted ion beam etching provided highly anisotropic pattern transfer using Cr as the mask material. Efforts are underway to examine the dependence of etch damage on incident ion energy for 200-1000 nm diameter features in the multiple quantum well material using photoluminescence spectroscopy at 4K.

9:20am NS-ThM4 Nanometer-scale Sputter-Induced Rippling of the SiO@sub2@ Surface Characterized with Real-Time X-ray Scattering, C.C. Umbach, Cornell University; R.L. Headrick, Cornell High Energy Synchrotron Source; B.H. Cooper, J.M. Blakely, Cornell University; E. Chason, Sandia National Laboratories

Certain advanced technologies (quantum wires, dots, etc.) may require surfaces with periodic topographic modifications to produce desirable length scales during fabrication. The formation of ripples on the surfaces of materials during ion sputtering has been known for decades and represents a potentially useful type of periodically modified substrate. In this paper we report on forming ripples on thermally-grown SiO@sub 2@ while the structure of the ripples is monitored in real-time with x-ray scattering. Ripples were formed in UHV using argon ions with energies between 0.5 and 2 keV. Ripple wavelengths between ~150 and ~600 Å were formed, with the wavelength increasing linearly with the ion energy. During sputtering, both the specular and diffuse intensity of 11 keV x-rays scattered from the surface were measured. Well-defined peaks in the diffuse scattering allow the determination of the ripple morphology. The effects of annealing during or after sputtering can also be monitored. Realtime monitoring of the ripples may permit more precise control of aspects of the ripple structure. The mechanism of ripple formation will be discussed in the context of theories of surface instabilities associated with ion bombardment.

# 9:40am NS-ThM5 Materials Considerations for Optical Lithography at the Nanometer Scale, F.A. Houle, W.D. Hinsberg, M.I. Sanchez, J. Hoffnagle, M. Morrison, C. Nguyen, IBM Almaden Research Center

The extendability of polymeric photoresist systems to the fabrication of nanostructures is a critical issue for both conventional microelectronics and for new device concepts. We have developed techniques using deep-UV interferometric lithography to evaluate quantitatively the relationship between polymer properties and reactivity and structure formation, with a particular focus on chemically amplified resist systems. Extended grating structures ranging from 50 to 500 nm linewidth and millimeters of line length are readily produced using 257 nm light, enabling systematic studies of the scaling of both chemical and physical phenomena. The apparatus and experimental methods will be described. We will present data using real-time spectroscopy and AFM analyses that probe the role of acid diffusion and aerial image definition in determining dimensional control and line-edge roughness at the nanometer scale. Implications of the data for x-ray and electron-beam resists will be discussed.

# 10:00am NS-ThM6 Nanofabrication of Organic Thin Film Materials Using Scanning Probe Lithography, G. Liu, S. Xu, K. Wadu-Mesthrige, Y. Qian, Wayne State University

Scanning probe microscopy(SPM) allows the surface structure to be visualized with unprecedented spatial resolution. Under carefully chosen conditions, SPM tips or probes can be used as a manipulation tool to fabricate nanoscopic patterns on surfaces. Recent work in our laboratory focuses on using SPM tips to fabricate nanopatterns on the surfaces of organic thin films. The talk will describe two new techniques developed in our lab. First method is nanografting which utilizes atomic force microscopy (AFM) tips as a nanoshaver. Such a shaver is operated on a self-assembled monolayer matrix in a solution containing adsorbate molecules. New molecules adsorb onto the shaved area following the plowing track of the AFM tip. Second methods utilizes scanning tunneling microscopy (STM)

tips which operates under a high tunneling current. Single layers of metals can be removed from desired locations. The advantage and potential applications of the two techniques will also be discussed.

#### 10:20am NS-ThM7 Proximity X-ray Lithography of Siloxane and Polymer Films Containing Benzyl Chloride Functional Groups, *S.L. Brandow*, *W.J. Dressick, C.S. Dulcey*, Naval Research Laboratory; *H. Witschi, P.F. Nealey*, University of Wisconsin

Siloxane and polymer films containing benzyl chloride functional groups were exposed to patterned proximity x-rays. Silicon wafers coated with films of (p-chloromethyl)phenyl-trichlorosilane (CMPTS) or spun coated poly-vinyl benzyl chloride (PVBC) were exposed at the University of Wisconsin synchrotron x-ray source using 0.9385 nm radiation (800 MeV) at doses ranging from 50-1500 mJ/cm@super2@. Exposure resulted in changes to the surface energy and chemical reactivity of the imaging layers. The loss of chlorine and formation of oxidized carbon photoproducts upon exposure was followed as a function of dose using x-ray photoelectron spectroscopy. A corresponding change in surface energy, as monitored by static water contact angle, was also observed. The successful chemical grafting of amine ligands to the portions of the siloxane and polymer films which have been exposed to proximity x-rays definitively establishes the formation of surface aldehyde or ketone groups as a major photochemical pathway. The resulting surface amine was used to covalently bind either a fluorescent tag or a colloidal Pd(II) nanoparticle capable of initiating the deposition of electroless Ni. Pattern formation is demonstrated and the mechanistic differences of photoproduct formation on siloxane and polymer films will be discussed.

#### 10:40am NS-ThM8 Nanofabrication of Apertures for Single Quantum Dot Spectroscopy, D. Park, C.R.K. Marrian, D. Gammon, R. Bass, P. Isaacson, E. Snow, Naval Research Laboratory

Optical spectroscopy of quantum dots can be performed by probing sub micron lateral regions of a quantum well through apertures in an opaque metallic film on the surface of a sample. The apertures must be small, smooth to avoid scattering the incident light and fabricated without inducing damage in the quantum well, i.e. without an etch process. We have developed a technique based on a metal lift-off using electron beam nanolithography in a negative resist. (The resist is patterned, developed, coated with metal film and then treated with a solvent to remove the remaining resist to lift-off the metal in areas where it covers the resist.) The use of a positive resist (the preferred choice for lift-off) would require prohibitively long write times as the entire sample except the apertures would have to be exposed. Obtaining the undercut (e.g. re-entrant) profile required to form a clean edged lift-off is difficult with negative beam resists. However, using 20 kV electrons, an undercut profile can be obtained by using a sufficiently thick (>1 µm) resist layer. To obtain sub 200 nm apertures, we have used our 50 kV e-beam tool. However, it is not possible to obtain an undercut profile directly with 50 kV electrons as even a thick resist film does not stop a sufficient number of the incident electrons as occurs at 20 kV. By changing to an area, as opposed to a point. exposure high aspect ratio (~10:1) resist features with vertical sidewalls can be obtained at 50 kV. An oxygen plasma treatment has been found to smooth the sidewalls of the resist features and to provide a slight undercut sufficient to give a clean aperture following metal lift-off. These lithographic results will be compared to, and shown to be in quantitative agreement with, the predictions of our simulations of electron elastic and inelastic scattering. Finally, an example of the spectroscopy will be presented along with a discussion of the relative advantages of this technique over spectroscopy with a near field probe.

### 11:00am NS-ThM9 Nanolithography and Macromolecular PMMA, E.A. Dobisz, S.L. Brandow, R. Bass, L.M. Shirey, Naval Research Laboratory

Polymethylmethacrylate (PMMA) has been the standard high resolution resist for over 20 years. The limits to its resolution has been the subject of many controversies that center upon our understanding of e-beam interactions with materials over 10 nm length scales, resist development, and the utility of macromolecular resists for very high resolution lithography. In this work nanolithographic pattern development is examined from the latent image formation through the evolution of surface morphology as patterns develop. Two molecular weights of PMMA, 950K and 50K were spun onto Si wafers as 50 nm thick films. Lithographic patterns consisted of: (1) large (1-20 mm) pads with a 50 nm gap of exposure in the center and (2) grating patterns of 10 nm lines on periods from 40-100 nm to examine the limit of the resist material on pattern density. Exposures were made by 50 kV e-beam lithography system with a Gaussian probe standard deviation of 8 nm. The resist patterns were examined by AFM operated in tapping and contact modes. Examination of as-baked PMMA showed a nodular structure, with average particle diameters of 50 nm. Morphological changes during development will be discussed. AFM latent images of detected e-beam exposure show a 0.2-2.9 nm depression in the resist. Latent images are observed over a much larger dose latitude than observed in developed patterns. In developed grating patterns the 60 nm period is critical. The granular structure of 50K resist prevented the development of the 60 nm grating. In the 950K resist, the 60 nm grating developed readily, but the 40 nm period grating is problematic. AFM images show etching of the particle boundaries across the resist between the lines to prevent formation of the 40 nm grating. AFM images are compared and contrasted to SEM micrographs. The results are discussed in terms of electron scattering during exposure, resist contrast, stress, and resist structure.

#### 11:20am NS-ThM10 Indium Phosphide Nanocrystals formed by Sequential Ion Implantation into Fused Silica, D.O. Henderson, R. Mu, A. Ueda, M.H. Wu, D. Denmark, Fisk University; C.W. White, A. Meldrum, R.A. Zuhr, Oak Ridge National Laboratory

Indium followed by phosphorous were implanted into optical grade fused silica at energies of 320 and 120 keV, respectively and at doses ranging from 1x10@super 16@ ions/cm@super 2@ to 1x10@super 17@ ions/cm@super 2@. The implanted substrates were annealed at 800°C for 1 h in a reducing atmosphere (5% H@sub 2@ +95% Ar). Vibrational and electronic spectra were recorded before and after annealing the samples. The vibrational spectra revealed a peak at 320 cm@super -1@ after annealing at 800°C. The intensity of this peak increased with ion dose and is assigned to the surface phonon of InP nanocrystals. XRD measurements confirmed the presence of crystalline InP and TEM showed particles with radii ranging from 4.6 to 11.6 nm. Electronic spectra of the annealed samples indicated that the energy of the band edge absorption is well below the bulk value of 969 nm. The band gap energies increased with decreasing ion dose and is attributed to quantum confinement of the exciton. The quantum confinement of the exciton is supported by the TEM measurements which demonstrated that the nanocrystals are nearly equal to or smaller than the InP exciton radius of 10.7 nm.

#### Surface Science Division Room 308 - Session SS1+NS-ThM

#### **Growth and Thin Films**

Moderator: J.B. Hannon, Sandia National Laboratories

8:20am SS1+NS-ThM1 Peter Mark Memorial Award Address - Morphology of Epitaxial Films during Low Temperature Growth, D.G. Cahill<sup>1</sup>, University of Illinois, Urbana-Champaign INVITED

# 9:00am SS1+NS-ThM3 The Influence of Dislocations on the Intermixing Kinetics of Pd-Au Monolayer Films, O. Schaff, A.K. Schmid, M.C. Bartelt, R.Q. Hwang, Sandia National Laboratories

The kinetics of surface alloying differ dramatically from the bulk counterparts. This is due to the fact that the dominant diffusion mechanisms are fundamentally different on surfaces. In this work, we used classical bulk studies as a guide to construct an effective experimental approach to directly determine such atomic mechanisms controlling intermixing kinetics in two-dimensional samples. On a clean Ru(0001) substrate, we prepared prototypical two-dimensional diffusion couples, consisting of monolayer regions of Au adjacent to monolayer regions of Pd. Between carefully controlled annealing steps, atomic resolution scanningtunneling microscopy was used to image changes in the adlayer structure, and thereby identify and characterize the phenomena leading to the formation of a Pd-Au surface alloy. We report several striking observations: (I) The Pd-Au surface alloy forms only on the Au-rich side of the diffusion couple, reflecting strong asymmetry in the diffusion constants of the two metals across the Pd-Au seam. (II) Diffusion constants, and thus alloying, in this system are strongly anisotropic. Specifically, the rate of interdiffusion at a given Pd-Au boundary, measured from changes in the average position of the alloy interface, depends strongly on the orientation of the boundary with respect to the underlying Ru lattice. We relate these observations to the energetics of exchange of surface atoms with the "gas" of Au and Pd adatoms, as well as to the dynamical properties of the dislocation network present in the Au side of the diffusion couple.

<sup>1</sup> Peter Mark Memorial Award Winner

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9:20am SS1+NS-ThM4 The Kinetic Nature of Slope Selection during Unstable Growth, *S. van Dijken*, *L.C. Jorritsma*, *B. Poelsema*, University of Twente, The Netherlands

At temperatures below 320 K, the Cu(001) transforms into an arrangement of facets upon epitaxial growth, resulting in a pyramidlike surface morphology. The sides of the pyramids all have the same facet orientation, selected by the growth temperature. With increasing growth temperature, [113], [115] and [117] facet faces are obtained.@footnote 1,2@ Up to now, it is not clear whether this slope selection is determined by local equilibrium or by the kinetics of the growth process. We present evidence for the kinetic origin of this phenomenon. At the same temperature various facet orientations can be obtained, depending on the angle of incidence of the impinging adatoms. We will show that the observed slope selection can be explained by the refraction of atoms above the surface. @FootnoteText@ @footnote 1@H.J. Ernst, F.Fabre, R. Folkerts and J. Lapujoulade, Phys. Rev. Lett. 72, 112 (1994) @footnote 2@L.C. Jorritsma, M. Bijnagte, G. Rosenfeld and B. Poelsema, Phys. Rev. Lett. 78, 911 (1997)

9:40am SS1+NS-ThM5 Growth on Cu(100) Using Improved Simulation Algorithm@footnote 1@, J.G. Amar, University of Toledo; M.R. D'Orsogna, T.L. Einstein, University of Maryland, College Park; I. Beichl, National Institute of Standards and Technology; F. Sullivan, Center for Computing Sciences

We have developed a novel Monte Carlo scheme to simulate homoepitaxial growth on (100) surfaces of sc and fcc crystals, using tree and list structures. We have applied it to the specific case of Cu, in both the submonolayer and multilayer growth regimes. Energy barriers were calculated using Effective Medium Theory, and diffusive processes were grouped into 4 classes. The effect of an Ehrlich-Schwoebel barrier was also considered. For the submonolayer regime at 213 K, we find reasonable agreement with experimental results@footnote 2@ for the scaling of the island density as a function of the ratio of diffusion and deposition rates. For multilayer growth at 160 K we find good quantitative and qualitative agreement with experimental results@footnote 3@ for the width as a function of coverage. In particular, the width exponent  $\beta$  agrees with the experimentally reported value. At higher temperatures (T=200 K), our simulations underestimate the exponent ß. Presumably a new diffusion channel becomes important. We have tried several single-atom processes, e.g. up-stairs climbing and biased upward and downward funneling, but none improve agreement significantly. The initial surface morphology may also exert an important influence on ß at high coverages. We are currently investigating the effects of different initial growth conditions such as a slightly rough or stepped substrate. @FootnoteText@ @footnote 1@Work partly supported by DoD; MRD and TLE primarily supported by NSF MRSEC grant DMR 96-32521. @footnote 2@A. Swan, Z.P. Shi, J.F. Wendelken, and Z. Zhang, Surface Sci. 391, L1205 (1997). @footnote 3@H.J. Ernst, F. Fabre, R. Folkerts, and J. Lapujoulade, Phys. Rev. Lett. 72, (1994); JVSTA 12, 1809 (1994)

# 10:00am SS1+NS-ThM6 The Atomistics of Homoepitaxial Growth on bcc(110)-Surfaces, U.K. Koehler, C. Jensen, A. v. Stockhausen, Ruhr-Universitaet Bochum, Germany

Time lapsed STM-movies, which allow a direct observation of the kinetics of growth processes on an atomic level up to 500°C, SPA-LEED and Monte-Carlo simulations are used to study the nucleation and growth behavior of homoepitaxy on the bcc(110)-surface. For Fe on Fe(110) and W on W(110) a strongly anisotropic growth with islands elongated in [001]-direction is found. A very effective Schwoebel-barrier leads to a nearly perfect statistical growth at RT with increasing island anisotropy in upper layers. At higher coverage a complete facetting of the surface is found and analyzed with SPA-LEED. A quantitative analysis of the STM-movies together with a kinetic Monte-Carlo simulation, which includes the full crystallographic symmetry of the bcc(110)surface, is used to extract information on the atomic diffusion behavior governing growth. A variety of growth features like the rugged appearance of the island edges and changes in the island shape with temperature are correctly reproduced in the simulation and can be assigned to the local diffusion energetics at step edges. Especially an anisotropic diffusion, which strongly suppresses diffusion along [001], is needed to reproduce the observed island anisotropies. A comparison of the layer distribution in the simulation with the one found with STM is used to determine the Schwoebel-barrier. Lateral island coarsening and an atom flux across the step edge following a temperature increase after growth is directly observed in STM.

10:20am SS1+NS-ThM7 Probing the Forces Stabilizing Self-Assembled Structures: Dynamics of Vacancy Island Lattices in Ag films on Ru(0001), K. Pohl, M.C. Bartelt, J. de la Figuera, N.C. Bartelt, Sandia National Laboratories; J. Hrbek, Brookhaven National Laboratory; R.Q. Hwang, Sandia National Laboratories INVITED

Nature exhibits processes that rival our most advanced patterning technologies used to create ordered lattices of nanoscale structures. Such self-organized phenomena have the potential to revolutionize materials performance, leading to higher density information storage and high-speed nanoscale electronics. Though many observations of self-organization have been reported, the fundamental mechanisms underlying such behavior remain unclear. The commonly accepted source of such mesoscopic-scale forces is the stress field mediated by the substrate which supports the grown structures. This, however, has not been confirmed, nor have such interactions been directly measured. In our work we have taken the approach of using observations of thermal fluctuations of an ordered array of surface defects to probe the interactions between the defects. In particular, we have used STM to study the array of vacancy lattice islands which forms upon exposure of a monolayer of Ag on Ru(0001) to sulfur. This is an extremely well-defined example of an ordered "mesoscopic" surface structure. At room temperature, each island is observed to vibrate around its equilibrium lattice postion. These vibrations appear to be harmonic and by performing a normal mode (phonon) analysis of the vibrations we can determine the elastic constants of the island array. The magnitude of the interactions is consistent with theories of elastic stepstep interactions in strained films. This work was supported by the Office of Basic Energy Sciences of the U.S. DOE, Division of Materials Science (Contract No. DE-AC04-94AL85000).

# 11:00am SS1+NS-ThM9 STM Study of Ultrathin NaCl(111) Layers on Aluminum, W. Hebenstreit, J. Redinger, TU Vienna, Austria; R. Podloucky, University Vienna, Austria; M. Schmid, P. Varga, TU Vienna, Austria

Polar surfaces like NaCl(111) are electrostatically unstable and cannot be found as terminating surfaces of crystals. But we can grow NaCl islands with (111) surface orientation on Al(111) and Al(100) single crystals. The (111) structure is revealed by atomically resolved Scanning Tunneling Microscopy (STM). The maxium coverage we could achive was 0.3 monolayer. The islands are triangular shaped, located at the lower side of substrate step edges and in the case of the Al(111) substrate alinged with the closed packed directions of the substrate. The islands consist of two Na layers with one Cl layer in between. We performed ab initio calculations with the FLAPW (full potential lineraized augumented plane waves) method of the electronic structure of a free standing Na-Cl-Na sandwich structure. These calculations reveal that the Na 3s level is filled with half an electron. The sandwich consists of two +0.5 charged Na metallic layers with a -1 charged ionic Cl layer in between, so the whole film ist neutral, free of dipoles, and electrostatically stable. The film is 4.6% laterally contracted and 5.3% expanded in vertical direction with respect to NaCl bulk distances.

# 11:20am SS1+NS-ThM10 Three-Dimensional SiGe Island Density on Si(001) and Morphology After Si Overgrowth@footnote 1@, J.S. Sullivan, E. Mateeva, H. Evans, D.E. Savage, M.G. Lagally, University of Wisconsin, Madison

Thin films of SiGe deposited on Si(001) can form three-dimensional (3D), coherently strained islands via a modified Stranski-Krastanov growth mode. Single films as well as highly ordered 3D superlattices with specific island sizes and densities may exhibit unique electronic and optoelectronic properties. In order to investigate how common process variables in epitaxial multiple-layer film growth influence 3D island density and morphology, we deposited SiGe films on Si(001) using low-pressure chemical vapor deposition and varied alloy composition, substrate temperature, and deposition rate. Films containing {105} faceted SiGe islands were overgrown with and embedded in Si at various substrate temperatures. Film growth and morphological evolution were monitored with in-situ, real-time reflection high-energy electron diffraction. Atomic force microscopy was performed ex-situ to characterize film morphology, and buried-island morphology was determined with cross-sectional transmission electron microscopy. The 3D island number density exhibits an Arrhenius-type dependence on substrate temperature, a power law relationship with deposition rate, and an inverse proportionality to Ge mole fraction in the alloy. Islands broaden during overgrowth and embedding due to thermally activated mass transport and Si interdiffusion; such that the {105} facets grow outward producing a (001) mesa at the apex. We will discuss our results in the context of simple thermodynamic and kinetic models and describe possible methods of obtaining and

maintaining a specific size and size distribution of 3D islands. @FootnoteText@ @footnote 1@Supported by the NSF.

11:40am SS1+NS-ThM11 Effects of Ion Pretreatments on the Nucleation of Silicon on Silicon Dioxide, *C Basa*, University of North Carolina, Chapel Hill; *Y.Z. Hu*, AG Associates Inc.; *M.T. Tinani, E.A. Irene*, University of North Carolina, Chapel Hill

It is well known that the silicon (Si) surface condition is crucial for low temperature Si expitaxy.@footnote 1@ In particular, hydrogen has been implicated as an important factor inhibiting Si nucleation on Si,@footnote 2@ and SiO@sub 2@ surfaces.@footnote 3@ In addition, nuclei densities can change depending upon the preparation of the surface film.@footnote 3@ We previously studied the effects of pretreatments of various forms of hydrogen (molecular and ionic) on nucleation parameters (nuclei density, and incubation time (t@sub inc@)).@footnote 4@ We found that H@sub 2@ pretreatments passivated the surface causing longer t@sub inc@, lower nuclei density, larger nuclei, and rougher final films. H@super +@ pretreatments increased the number of nucleation sites resulting in shorter t@sub inc@, higher nuclei density, smaller nuclei, and smoother final films. However, the mechanism for H@super +@ effects on nucleation was not elucidated in that study. Therefore, the study was extended to include the effects of (1) ion dose and energy, (2) other ions (Ar@super +@, He@super +@, N@super +@) and (3) temperature of the pretreatment on the nucleation of poly-Si on SiO@sub 2@. There are three major results from the surface pretreatment experiments. First, in the range tested, ion dose has more influence on nucleation parameters than ion beam energy. Second, results with different ionic species indicate a physical, rather than chemical mechanism, for creating nucleation sites. Third, high temperature ionic pretreatments damage the surface less than room temperature treatments. @FootnoteText@ @footnote 1@T. Yamazaki, M. Miyata, T. Aoyama, and T. Ito, J. Electrochem. Soc., 139, p. 1175 (1992). @footnote 2@K. Tsubochi and K. Masu, Mat. Res. Soc. Symp. Proc., 315, p. 59 (1993). @footnote 3@J. T. Fitch, J. Electrochem. Soc., 141, p. 1046 (1994). @footnote 4@Y. Z. Hu, C. Y. Zhao, C. Basa, W. X. Gao, and E. A. Irene, Appl. Phys. Lett., 69, p. 485 (1996).

## Thursday Afternoon, November 5, 1998

#### Nanometer-scale Science and Technology Division Room 321/322/323 - Session NS-ThA

#### Nanoscale Manipulation and Chemical Modification Moderator: R.J. Hamers, University of Wisconsin, Madison

2:00pm NS-ThA1 Nanomanipulation for Material Properties, Interactions and Devices, R. Superfine, University of North Carolina, Chapel Hill INVITED We have pursued the study of nanometer-scale materials, including colloids, viruses, DNA and nanotubes, through the use and development of an advanced interface for scanning probe microscopy (SPM). The ability to manipulate objects efficiently on surfaces makes available a wide variety of experiments on the interactions between the sample and substrate, on the physical properties of individual objects and on the creation of unusual devices incorporating the nanometer objects. For example, the simple pushing of an object with the AFM tip, with the measurement of the applied lateral force, measures the surface adhesion and friction. Dynamical phenomena such as stick/slip, sliding and rolling has been observed. Manipulation can be used to deform objects, and the response of the objects to large strain reveals phenomena such as buckling and fracture. Finally, manipulation allows us to begin the study of unusual device structures incorporating nanometer samples such as nanotubes. Making these experiments possible is the nanoManipulator, an interface for SPM's. The interface allows for real time control of the AFM tip while simultaneously recording topography, lateral forces, as well as device features such as conductivity. In addition, the data is rendered as 3-d, directionally illuminated surfaces providing immediate, intuitive interpretation of the SPM data.

#### 2:40pm NS-ThA3 Monitored Mechanical Nano-Manipulation, B.E. Koel, C. Baur, A. Bugacov, A. Madhukar, N. Montoya, T.R. Ramachandran, A.A.G. Requicha, R. Resch, P. Will, University of Southern California

We have investigated the mechanical positioning of colloid particles deposited on a flat substrate. The positioning is achieved by pushing the particles with the tip of an Atomic Force Microscope (AFM). We gained fundamental insight into this manipulation process by monitoring the tip deflection and other signals during the pushing event. We also developed a procedure that allows the operator to compensate for instrument errors such as creep, hysteresis and thermal drift, especially important for operation in ambient air and at room temperature. This has lead to manipulation with high spatial certainty and increased the reliability considerably. We demonstrate precise positioning of arrays of colloidal particles and building and manipulating 3-D and linked 2-D structures of nanoparticles. It is possible to adapt this generic type of manipulation to a variety of environments and materials promising a high potential for fabrication of nano-scale devices such as those required in applications ranging from high-density data storage to single-electron electronics and nanoelectromechanical systems (NEMS) prototyping and fabrication.

# 3:00pm NS-ThA4 Field-Induced Manipulation of Ag Clusters for Tailoring of Nano-Structures on Silicon Surface, K.-H. Park, J.S. Ha, W.S. Yun, E.-H. Lee, ETRI, Republic of Korea

A precise nano-fabrication method was devised by using field-induced manipulation of Ag clusters with a tip of scanning tunneling microscope(STM). After deposition of Ag on Sb-terminated Si(100) surface, we were able to selectively desorb(redeposit) Ag clusters from(to) the surface by applying a voltage pulse. The manipulation of metal clusters was found to be precisely controlled due to the weak bonding strength between the clusters and an Sb-terminated Si(100) surface. We investigated those field-induced manipulation by varying bias voltage, pulse duration, and distance between tip and sample. Under suitable conditions for manipulation, we could fabricated various kinds of metallic nanostructures on the Sb-passivated silicon surface. Of interest, single electron charging and tunneling behaviors were observed in the local current-voltage (I-V) measurements on top of Ag clusters at room temperature. We have found that Coulomb staircases in tunneling spectroscopy at Ag nano-clusters sensitively depended on the distribution of surrounding clusters, indicating that the lateral conduction channel through neighboring Ag clusters was very important for the tunneling I-V characteristics. This result can be further utilized for the construction of single electron devices operating at room temperature by means of lateral conduction channels.

3:20pm NS-ThA5 Probe Induced Manipulation of Bromine, Iodine, and Sulfur on Si (100), C.F. Herrmann, J.J. Boland, University of North Carolina, Chapel Hill

Scanning tunneling microscopy was used to study probe induced manipulation of the chemisorption sites of molecules on the Si (100) 2x1 surface. Specifically, the behaviors of bromine, iodine, and sulfur were studied. These adsorbates initially adsorb to the room temperature surface on adjacent dimers of the same row. Using the probe tip, one of the atoms can then be shifted to another dimer in this row, leaving one or more bare Si dimers in between them. This relocation occurs due to an increase in the field between the probe tip. The separation efficiency as a function of probe tip voltage was measured for each adsorbate and it was found that each adsorbate exhibited a different threshold voltage. The diatomic molecules can also adsorb to the surface along a single dimer unit. A comparison study of the behaviors of the two different adsorption configurations was also conducted.

#### 3:40pm NS-ThA6 Two Mechanisms of Nanostructure Growth for STM Assisted CVD@footnote 1@, I. Lyubinetsky, S. Mezhenny, University of Pittsburgh, U.S.; J.T. Yates, Jr., University of Pittsburgh

A large Cu-containing organometallic molecule, [Cul (hfac) (vtms)],@footnote 2@ has been employed to produce nanostructures under the action of the STM tip on Si(111) exposed to the molecular flux in a UHV system at room temperature. The nanostructure growth mechanism, induced by dissociative electron attachment, exhibits an electron energy threshold of 4.5 eV. A second growth mechanism is related to an electric field-assisted activation process, which takes place in a narrow sample voltage bias range from +6 to +10 V. The topology of the deposited structure produced by the two mechanisms is strikingly different. Electron-induced growth leads to flat and broad structures which extend up to 100 nm laterally with an increase of electron energy to +15 eV. Field-induced growth leads to column-like structures with a HWHM down to 4 nm and a high aspect ratio up to 3. Comparative studies of the electron attachment mechanism have been carried out using a broad beam electron gun and x-ray photoelectron spectroscopy of the deposit on Si(111) to confirm the 4.5 eV energy threshold observed under the STM tip, and to measure elemental composition of the deposit. @FootnoteText@ @footnote 1@Work supported by the Office of Naval Research; @footnote 2@Hexafluoroacetylacetonate Cu (I) vinyltrimethylsilane, a CVD precursor for Cu deposition.

#### 4:00pm NS-ThA7 Nano Scale Selective Al Growth on the Si(001)-H Surface using Dimethylethyamine Hydride, *T. Mitsui*, *E. Hill*, University of Minnesota, US; *E. Ganz*, University of Minnesota

I will discuss nano-scale selective growth of Al on a hydrogen patterned Si(001) surface. We have studied Al growth on the clean and mono-hydride terminated Si(001) surface over a range of temperatures using Dimethylethyamine Hydride (DMAH). We have found that Al growth occurs upon dosing the clean Si(001) surface heated above 150 °C but does not occur on a mono-hydride Si(001) surface up to 300 °C. Patterning a monohydride terminated Si(001) surface with the STM tip,@footnote 1@ we have created nano-scale regions of bare Si(001). Heating this surface to 200 °C and dosing with DMAH, we observe the selective growth of Al from DMAH on the clean silicon region. The mechanism for selectivity will be discussed. @FootnoteText@ @footnote 1@Lyding et al, Science. 268 (1995) 1590

#### 4:20pm NS-ThA8 Modifications of Thioaromatic Monolayers by Low Energy Electrons, W. Geyer, V. Stadler, W. Eck, M. Zharnikov, A. Gölzhäuser, M. Grunze, Universität Heidelberg, Germany

Electron induced modifications of aromatic self-assembled monolayers (SAMs) were investigated by x-ray photoelectron (XPS) and near edge x-ray absorption fine structure (NEXAFS) spectroscopy. SAMs of 1,1'-biphenyl-4-thiol (BP), 4'-nitro-1,1'-biphenyl-4-thiol (NBP) and 4'-amino-1,1'-biphenyl-4-thiol (ABP) were prepared on (111) textured gold surfaces. The subsequent characterization indicated that the films were well ordered. The films were then irradiated with low energy electrons (20 - 300 eV) and changes were observed in situ. Biphenyl SAMs are generally more stable to low energy electrons than alkanethiols exposed under similar conditions. The functionalized biphenyls (NBP, ABP) showed distinct differences in the reactions of their end groups with the incident electrons. Mechanisms of interactions between the electrons and the SAMs as well as possible applications in the tailoring of surfaces for electron beam lithography will be discussed.

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4:40pm NS-ThA9 Selective Etching of the SiO@sub 2@/Si Surface with Low Energy Electron Stimulated Reaction by using STM, N. Li, T. Yoshinobu, H. Iwasaki, Osaka University, Japan

Nano-fabrication on the SiO@sub 2@/Si surface is such an important subject that it may relate directly to the silicon industry. It has been shown that with focused high energy (tens to a few hundreds keV) electron beam, such as employed in TEM or SEM, nanometer scale windows can be cut through the SiO@sub 2@ overlayer and nano-structures can be formed on the exposed Si surface in a subsequent growth.@footnote 1,2@ However, large scale applications of this technique has been prevented by the relatively low efficiency of the high energy e-beam in etching the SiO@sub 2@ layer, particularly compared to the cost to build and maintain such a high energy accelerating system. Here we report an experimental study on selective etching of the SiO@sub 2@/Si surface with a low energy electron stimulated surface reaction by using STM. The investigation was carried out on Si(001) surface either with a thin native oxide overlayer or with a thermal oxide overlayer prepared in the UHV chamber. By applying a voltage of ~70 to 150 V across the tip-surface vacuum gap in the STM setup, while the tip-surface separation was adjusted to ~100 to 200 nm, a field emission current of ~5 to several hundred nA can be obtained, with which the SiO@sub 2@/Si surface was irradiated. Subsequent STM imaging shows that within the exposed area, the SiO@sub 2@ overlayer can be effectively evacuated by a thermal annealing of the surface at a temperature of ~650 to 700 °C. This can be shown evidently in the STM images by the obvious contrast change and clear atomic steps appeared within the exposed areas. By adjusting the beam energy, beam current and tip-surface separation, windows down to tens of nanometers in diameter can be etched through the SiO@sub 2@ overlayer. This result demonstrates the possibility of fabricating the SiO@sub 2@/Si surface with a low energy electron stimulated surface etching, and also presents another possibility of conducting such a nano-fabrication in a well controlled way with the STM. @FootnoteText@ @footnote 1@D.R. Allee, C.P. Umbach, and A.N. Broers, J. Vac. Sci. Technol. B9, 2838, (1991) @footnote 2@S. Fujita, S. Maruno, H. Watanabe, and M. Ichikawa, J. Vac. Sci. Technol. A15, 1493, (1997)

#### 5:00pm NS-ThA10 Current-Induced Local Oxidation: Mechanism, Quantum-Size Effects, and Applications, *R. Martel*, *T. Schmidt*, *Ph. Avouris*, IBM T.J. Watson Research Center

A novel method is introduced for locally oxidizing thin metal films with nanometer-scale resolution. Simply by subjecting Ti and Nb films in air to local current densities of 10@super 7@ A/cm@super 2@, metal-oxide tunneling barriers of 10-50 nm width can be fabricated in a self-limiting fashion. The high spatial resolution of the process results from its strongly non-linear dependence on the current density. Our experiments suggest that the oxidation involves current-induced atomic rearrangements and local heating. At the final stages of the barrier formation, when only atomic-scale channels remain unoxidized, the oxidation rate decreases drastically while the conductance drops in steps of about 2e@super 2@/h. This behavior gives evidence of conductance quantization and a superior stability of such metallic nanowires against current-induced forces compared with the bulk metal. This current induced local oxidation (CILO) process can be used in nanofabrication. Single electron transistor structures exemplifying Coulomb staircases at room temperature were fabricated in this manner.

#### Nanometer-scale Science and Technology Division Room Hall A - Session NS-ThP

# Nanometer-Scale Science and Technology Division Poster Session

# NS-ThP1 A Low Temperature STM System for the Study of Quantum Electronic Systems@footnote 1@, J.A. Stroscio, R.J. Celotta, National Institute of Standards and Technology

We describe a new experimental system with the goal of providing new measurement capabilities for the study of quantum electronic systems. Several experimental challenges are posed in the study of electronic systems confined to nanoscale dimensions. The physical information desired in such systems includes: the quantized electron energy distributions arising from spatial or magnetic confinement, the spatial extent of electronic wavefunctions, the role of electron-electron interactions and electron interactions with the confining boundaries, the exact physical structure of the system, the shape of the confining potentials, and finally, the physics of the electron transport. To meet these measurement challenges, we have designed a scanning tunneling microscope (STM) that will operate in the temperature range from 2-150 K with the capability of applying magnetic fields up to 10 Tesla. Equally challenging to the measurement methodology is the fabrication of quantum electronic systems. The STM is part of a facility that includes separate MBE fabrication systems for III-V semiconductor growth and thin metal film growth with in-situ transfer of samples to the STM system. In addition to these traditional fabrication techniques we are developing an autonomous atom assembler to fabricate quantum structures atom-byatom on a large scale. In this poster we will describe the design of the overall system, its components, and performance to date. @FootnoteText@ @footnote 1@ This work is supported in part by the Office of Naval Research.

#### NS-ThP2 Development of Low-Temperature Ultrahigh-Vacuum Atomic Force Microscope / Scanning Tunneling Microscope (LT-UHV-AFM/STM) Using Two-Stage Coil-Spring Suspension Isolator, *N. Suehira*, *K. Sugiyama*, *Y. Sugawara*, *S. Morita*, Osaka University, Japan

Recently, true atomic resolution imaging of The Noncontact Atomic Force Microscope (NC-AFM) was demonstrated, and the NC-AFM is expected as powerful tool to investigate the surface structure including insulators and the force acting on the surface. In such measurements, low temperature (LT) is one of the best environment, because it can reduce thermal noise in the force signal measured by using AFM cantilever and thermal drift between tip and surface. However, there is a few report on NC-AFM operating under low temperature and ultrahigh vacuum (UHV) condition. Here, we describe a new LT-NC-AFM/STM design. The most serious problem in the LT Scanning Probe Microscope (SPM) design is the vibration which influences the resolution of the images and the sensitivity of the signals such as tunneling current and the force. This is due to that, in the conventional LT-SPM design, the SPM body is mechanically connected with the bottom of the helium dewar, and hence the sufficient isolation of the various vibrations such as the building vibration, the acoustic noise and bubbling of the liquid nitrogen is difficult. In our design, such problem of the vibration is solved by using two stage coil-spring suspension isolation system with eddy current damper. Using a special designed gear mechanism, the SPM body is connected with the bottom of the helium dewar during cooling down, then it is mechanically isolated from the bottom of the helium dewar and suspended by the springs during measurement. The inertial translational mechanism is used for cryogenic coarse approach between tip and surface, because its compactness, rigidity and reliability. In the AFM measurement, the cantilever is scanned by the tube scanner, and its deflection can be detected by the fiber-optic interferometer inside the tube scanner. In the STM measurement, the tunneling tip is scanned instead of the AFM cantilever. Preliminary LT-STM imaging was demonstrated on Si(111)7x7 surface with atomic resolution and the LT-AFM measurement is under way.

NS-ThP3 Characterization of Various SiO@sub2@ by Scanning Capacitance Microscopy, G.H. Buh, C.J. Kang, Seoul National University, Korea; K. Mang, Samsung Electronics, Korea; S. Lee, C.K. Kim, C. Im, Y. Kuk, Seoul National University, Korea

Although electrical charge in SiO@sub2@ system can cause an adverse effect on the device performance, little is known about its spatially resolved characteristics. By using scanning capacitance microscopy (SCM),

dynamics of spatially distributed trapped charge in a SiO@sub2@ film on Si can be imaged with spatial resolution of ~20nm, which is determined by tip diameter. As in the case of macroscopic capacitance-voltage (C-V), trapped charge results in VT shift. The VT shift can be measured from C-V curves at various positions with good spatial resolution. Experiments were carried out with various kinds of SiO@sub2@ processed by thermal oxidation (wet, dry), plasma enhanced chemical vapor deposition(PECVD), and atmospheric pressure chemical vapor deposition(APCVD). The VT measured on PECVD oxide was found to be lower than that of thermal oxide. This result indicates the existence of positive fixed charges in the PECVD oxide. On the contrary to the VT shift in a thermal oxide with induced traps, the shift was not observed even after 4MV/cm stress. It is believed the traps in PECVD oxide is nearly saturated.

# NS-ThP4 Automated, High Precision Measurement of Critical Dimensions using the Atomic Force Microscope, D.A. Chernoff, D.L. Burkhead, Advanced Surface Microscopy, Inc.

Atomic Force Microscopes are used in many industries for research, engineering and process control. Until now, AFM operators have usually made dimensional measurements of sub-micron features by manually placing cursors on images or cross-section plots. Time constraints and operator fatigue limit the number of measurements. This in turn limits the extent of statistical analysis. We have developed a high accuracy measurement process which overcomes these limitations. On DVDs (Digital Versatile Discs), the smallest features are about 400 nm long, 320 nm wide, 120 nm high, with a track pitch of 740 nm. We use a specific data capture protocol and automated image analysis to measure the following parameters: track pitch@footnote 1,2,3@, bump height, bump width (at various threshold levels), bump length, and four sidewall slope angles. In a single 10x10 micron image of a DVD stamper, containing about 100 bumps, we tabulate about 1000 values. It is useful to pool the data from several images. In a plot of bump width vs. bump length, we see that width at half height increases from 315 nm for the shortest bumps (420 nm long) to about 380 nm for bumps longer than 1100 nm; this matches the increase seen for corresponding optical signals produced when a finished disc is played. Where sidewall angle deviates from the norm, we are able to review the image data to identify the specific nature of the defect. @FootnoteText@ @footnote 1@D.A. Chernoff, "Nano-metrology for the data storage industry", abstract of paper presented at AVS National Meeting 10/97, p.113 @footnote 2@US Patent # 5,644,512 and other patents pending @footnote 3@see also www.a1.com/asm

#### NS-ThP5 Use of Phase Imaging Tapping Mode AFM to Spatially Resolve Areas of Different Doping Densities on Patterned Si Wafers, *M.W. Nelson*, *P.G. Schroeder*, *R. Schlaf*, *B.A. Parkinson*, Colorado State University

The continuously shrinking dimensions of integrated circuits demand new methods for spatially resolved characterization of doping profiles in patterned semiconductor structures. Scanning probe microscopies offer the lateral resolution required to characterize structures down to atomic dimensions. Among these methods atomic force microscopy (AFM) has the advantage that the imaging process does not depend on the conductivity of the sample surface which offers easy characterization of oxidized samples in ambient conditions. In our experiments we demonstrate the use of tapping mode AFM (TMAFM) with additionally applied bias to spatially resolve areas of different doping densities and types on Si wafers. We observed changes in the phase contrast in regions of different doping densities as a function of the applied bias. Additional measurements on metallic substrates revealed that the cantilever phase and resonance frequency are strongly influenced by the bias induced Coulomb force between cantilever and sample. This allowed the discussion of the observed phase contrast variations in terms of semiconductor surface potential changes depending on the applied bias and the doping type of the various areas.

NS-ThP6 Metallic Adhesion at the Atomic Scale, A. Schirmeisen, G. Cross, P. Grütter, McGill University, Canada; U. Dürig, IBM Research Division, Switzerland

A unique AFM / STM / FIM system operating in UHV has been shown to measure directly force interactions of an atomically defined tip-sample junction. A W(111) tip, terminated by only three atoms, approaching an atomically flat Au(111) surface showed structural stability even upon touching the surface. The measured force distance curves reveal adhesion peaks of 5 nN and a contact stiffness of about 40 N/m. Moreover, there are no indications of a jump to contact. The yield strength of the junction (maximum contact pressure divided by the tip radius) reaches values of up to 25 GPa. The observed metallic short range adhesion forces show

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substantial contributions over an unexpectedly large distance range, which can not be explained within in the standard model for metallic adhesion by Ferrante and Smith.@footnote 1@ This study is being extended to different tip materials. First results of an Ir tip approaching a Au surface will be presented. Furthermore, to complement this study, MD simulations were performed at Tampere University by J. Nieminen. @FootnoteText@ @footnote 1@ J.H.Rose, J.R.Smith, J.Ferrante, Phys.Rev.B 28, 1835 (1983)

## NS-ThP7 Tribological Properties of Crystalline Surfaces, A.J. Gellman, J.S. Ko, Carnegie Mellon University

Atomic scale influences on the macroscopic tribological properties of surfaces have been explored through studies of friction between single crystalline metallic surfaces prepared and characterized under ultra-high vacuum conditions. Our experiment allows two single crystalline surfaces to be brought into contact under an applied load and then sheared at constant velocity while measuring friction forces developed at the interface. The presentation will describe observations made using Cu(111) surfaces that have been modified by adsorption of atomic and molecular species. These studies show quite clearly that in order to observe lubricating effects between Cu(111) surfaces it is necessary to adsorb at least one complete monolayer of adsorbed species. A second set of measurements have explored the frictional properties of single grain quasicrystals. Quasicrystals nominally possess low friction surfaces, however, all prior measurements have made use of surfaces contaminated by exposure to air. Under vacuum conditions the Al@sub 70@Pd@sub 21@Mn@sub 9@ quasicrystal surfaces have been cleaned prior to making friction measurements. While these yield coefficients of friction for perfectly clean surfaces that are significantly lower than those observed with clean metal surfaces they are much higher than those measured with quasicrystals in air. Controlled oxidation of the Al@sub 70@Pd@sub 21@Mn@sub 9@ guasicrystal surfaces has been shown to lower friction coefficients but not to the point observed in air. While surface contamination must be responsible, at least in part, for the low friction measurements reported in the past, oxidation alone cannot account fully for the properties of these surfaces.

# NS-ThP8 Frictional Force Microscopy Study of Discrete Surface Functional Group Assembled by Langmuir-Blodgett Technique, *M. Nakamura, A.*

Shimizu, Y. Nakayama, Y. Nagasawa, Toray Research Center, Inc., Japan Obtaining a flat surface which contains discrete functional groups with aimed density is doubtless valuable for nanometer science and technology. The functional groups can be used as nucleation sites for vacuum deposition or as adsorption sites for biomolecules. Furthermore, such a surface can be used as a test structure for scanning probe microscopy in terms of chemical identification. For this purpose, we have studied the Langmuir-Blodgett (LB) films formed with the mixture of stearic acid (SA) and stearyl mercaptan (SM) on atomically flat silicon substrates. The length of SM is approximately same as that of SA, but SM has a -SH group instead of a hydrophilic -COOH group. We therefore expect some part of the -SH groups in the bilayer film of the mixture to protrude from the surface. It was confirmed with atomic force microscopy (AFM) that flat and tight films were obtained with the mixtures which contain less than 10% of SM. Topographic and frictional force images showed that the surfaces contained number of protrusions exhibiting higher frictional force. The diameter of the higher friction spots was more than a few nanometer and varied with the AFM tip. Their density was nearly proportional to the concentration of SM. The distribution of the topographic height of these spots clearly exhibited two peaks around 1.6 and 2.6 Å, which can be explained by the models where the -SH shifts outward for one and two -(CH@sub 2@)- units of an alkyl chain. These results imply that the expected surface was successfully obtained. An interesting point is that the distribution of the maximum friction at these spots has only single peak in contrast to the height distribution.

#### NS-ThP9 Quantitave Surface Force Gradient Measurements Using Atomic Force Microscopy, *L.A.W. Sanderson*, *M.A. George*, *J.J. Weimer*, University of Alabama, Huntsville

The accepted theory that reconciles attractive and repulsive forces has become known as the Deryaguin-Landau-Verwey-Overbeek (DLVO) theory and may be simply stated as V@sub S@ = V@sub C@ + V@sub A@ + V@sub R@ , where V@sub S@ is the total potential, V@sub C@ is the core repulsive potential due to Pauli exclusion, V@sub A@ is the Van der Waals attractive potential and V-@sub R@ is the double layer repulsive potential. The DLVO theory is widely regarded as a cornerstone for understanding colloidal systems and forces on the molecular scale. The objective of this study is to characterize the forces between two surfaces at the molecular scale using an atomic force microscope (AFM) and to relate the results quantitatively to parameters in DLVO theory. Investigations have been made using an AFM of surface forces present between a standard Si@sub 3@N@sub 4@ AFM tip and mica substrates for water, ethanol, and carbon tetrachloride. Results agree with those previously reported in the literature. Colloidal probes over mica and silica substrates are being used in on going research to provide easier geometries for comparison to the DLVO theory. Variations in surface forces as a function of pH and salt concentrations are being examined. The goal is to obtain a means of characterizing molecular scale forces over thin films such as aminopropyltriethoxysilane and polyethyleneglycol anchored to substrates. From a fundamental side, understanding of these forces is also important in analyzing the behavior of such molecules in solution, and the results can be used to select solutions for improving image resolution with the AFM.

#### NS-ThP10 The Durability of Optical Fiber Probe Tips for Surface Profilometry, J.E. Griffith, R. Raghunathan, L.E. Plew, J.B. Bindell, Bell Laboratories, Lucent Technologies; J. Carlson, A. Berghaus, J.J. Plombon, C.E. Bryson, Surface/Interface, Inc.

The performance of a surface profiler strongly depends on the shape of the stylus or probe tip. Changes in the shape of the probe, caused by erosion or contamination during scanning, can be especially troublesome. The durability of the stylus depends on its shape, its composition, the composition of the sample, and the force sensor employed. We use cylindrical, silica glass probes etched from a specially chosen optical fiber. The glass probes are used in conjunction with a balance beam force sensor with sensitivity of approximately 10 nN. These probe tips have been used for extended periods on samples ranging from photoresist to silicon nitride with very little change in their shape from either erosion or contamination.

#### NS-ThP11 Force Measurement of Optical Evanescent Field using Kelvin-Null Method, K. Sawada, M. Abe, Y. Sugawara, Y. Andoh, S. Morita, Osaka University, Japan

We have measured an optical evanescent field using a noncontact mode atomic force microscope (AFM) combined with a frequency modulation detection method.@footnote 1@ Using a semiconductor AFM tip, the surface photo voltage (@delta@@phi@) is induced by the optical evanescent field. It causes the electrostatic force acting on the tip. This force (F) can be expressed by F=(V-@phi@)(@delta@C/@delta@z)@delta@@phi@. Here, V, @phi@, C, and z are the bias voltage, the contact potential difference, the capacitance and the distance between the tip and the sample, respectively. In this method, the electrostatic force is affected not only by the potential change @delta@@phi@ due to the evanescent field but also by the contact potential difference @phi@ between tip and sample which is not uniform on the surface. In this paper, we propose a novel method to detect only the variation of the surface photo voltage due to the optical evanescent field without the influence of the contact potential difference. Applying Kelvin-Null method,@footnote 2@ bias voltage V is controlled so that V-@phi@ is kept constant. Simultaneously, the incident beam is modulated at a frequency w, and the w component of the force gradient is measured which is proportional to the optical evanescent field. @FootnoteText@ @footnote 1@M. Abe, Y. Sugawara, Y. Hara, K. Sawada and S. Morita, Jpn. J. Appl.Phys. Vol. 37 (1998) pp. L167-L169 @footnote 2@M. Nonnenmacher, M. P. O'Boyle and H. K. Wickramasinghe, Appl. Phys. Lett. Vol. 58, No. 25, (1991) pp. 2921-2923

#### NS-ThP12 Tunneling and Photon Emission of Colloidal Particles, G.S. McCarty, C.D. Keating, P.S. Weiss, M.J. Natan, Pennsylvania State University

Binding of molecules of interest to colloidal particles allows the optical properties of the molecules to be studied using techniques such as surface enhanced Raman spectroscopy. By binding these colloidal particles to a conducting surface the electronic properties of the molecules can be probed using scanning tunneling microscopy. We have imaged gold and silver colloids bound to Au[111] coated with 2-mercaptoethylamine. The particles were then studied using photon emission scanning tunneling microscopy to probe the electronic and optical properties of single particles.

NS-ThP13 Temperature Dependence of the Raman Scattering Spectra in Zn/ZnO Nanoparticles, J. Xu, National University of Singapore, Republic of Singapore; W. Ji, Z.X. Shen, S.-H. Tang, National University of Singapore, Republic of Singapore

By using the gas evaporation technique with induction heating method, Zn nanoparticles coated with ZnO were prepared in Ar. The Raman spectra of

the Zn/ZnO nanoparticles have been studied over a wide range from room temperature through liquid nitrogen temperature and up to 873 K. Heating in air, we have, for the first time, successfully observed the transformations from the surface phonon mode to bulk vibrational mode in Zn/ZnO nanoparticles.

#### NS-ThP14 Electromagnetic Coupling Efficiency of a Metal Coated Optical Fiber Tip, *L. Alvarez*, CICESE, Mexico; *M. Xiao*, UNAM, Mexico

The optical coupling of a metal coated optical fiber tip is calculated by using the direct moment method. The purpose of the calculation is to study the transmission efficiency of the near field probe tips widely used in the scanning near field optical microscopy. In the scanning near field optical microscopy, the near field probe is often made of tapped optical fiber tip which is coated with a metallic thin layer to form a subwavelength aperture at the very end of the tip. It is of great importance to study the transmission efficiency of the tip as functions of the size of the aperture, the thickness of the metal coat as well as the shape and optical characters of the fiber tip. One wants to know that for a given incident light, how much light would effectively contribute to the final readout of the microscope. In the literature in near field optics, the tip transmission was studied with various two dimensional simulations and with microscopic discretional theories. In the present work, a general three dimensional electromagnetic theory is however proposed. As examples, numerical results of the coupled electromagnetic waves as functions of the aperture size, the coat thickness and the materials properties of the fiber tip are presented for the simplified case where the incident light is assumed to be a plane wave, and the results are discussed with regards to the imaging of the scanning near field optical microscope. Finally, it is pointed out that the proposed model calculation would be useful to provide guidances for the manufacture of the probe tip in the scanning near field optical microscopy.

#### NS-ThP15 Annealing Atmosphere and Electron Irradiation Effects on Gold Nanocrystals Buried in MgO, A. Ueda, R. Mu, M.H. Wu, D.O. Henderson, Fisk University; R.M. Uribe, Kent State University; A.F. Hepp, E.M. Gordon, NASA Lewis Research Center; C.W. White, J. Budai, A. Meldrum, R.A. Zuhr, Oak Ridge National Laboratory; P. Wang, University of Texas, El Paso

We have reported previously annealing effects on the surface plasmon (SP) of gold nanocrystals (NCs) formed by Au ion implantation in MgO. Annealing the samples after implantation promotes the diffusion of gold atoms, nucleation and growth of the NCs. The SP absorption for the Au/MgO system annealed in an oxidizing atmosphere (OA) is observed at ~560 nm, while annealing the same sample in a reducing atmosphere (RA) shifts the SP to ~524 nm. The process is entirely reversible. We propose that the SP shift originates from the creation of F@sub n@-centers when the samples are annealed in a RA, while they are annihilated in an OA. The F@sub n@ center acts as an e@super -@donor to the Au NCs that causes a blue shift of the SP. Subsequent annealing in an OA annihilates the F@sub n@ centers and the SP shifts back to 560 nm. TEM studies on Au NCs in MgO indicate the crystals are cubic and are aligned along the direction of the MgO lattice. Maxwell-Garnet effective medium theory was used to simulate the absorption spectra of the Au NCs formed in MgO under RA and OA. A good fit was obtained for sample annealed in an OA, but the fit for the annealed sample in a RA deviated from the experimental results. This is attributed to a change in the dielectric function of the Au NC that was caused by electron transfer from the F@sub n@ center. Electron beam irradiation of the Au/MgO samples was also investigated as an alternative method to study F@sub n@center creation and their interaction with the gold NCs. The optical spectra in the SP region are presented for the electron irradiated Au/MgO samples and are compared to those annealed in a reducing atmosphere.

#### NS-ThP18 The Shape Evolution of Patterned Submicron Structures under Thermal and Chemical Activation, K.C. Lin, University of Maryland, College Park, U.S.A; D. Kohn, K. Thuermer, J.E. Reutt-Robey, E.D. Williams, University of Maryland, College Park

Lithographic techniques developed extensively for microelectronics applications provide new opportunities to design experiments to explore the nanoscale realm, where traditional continuum based descriptions of morphology are likely to fail. In particular, the preparation of defined surface patterns with crystalline subfeatures, e.g. facets and steps, allows physically-based studies of mass transfer processes in response to the changes of physical and chemical environments. We utilize electron beam lithography and lift-off techniques to pattern submicron lines, squares and dots of noble metals on silicon oxide and silicon substrates, and use STM and AFM to characterize their structure and evolution. These structures are chosen to mimic the microelectronic device contact lines and model heterogeneous catalysts. We find, for example, the surfaces of submicron Au lines, as prepared, exhibit a uniform surface texture consisting of 3-D islands of ca. 50 nm size. Thermal activation for 20 hrs at 200 ° C breaks the lines into grains with ~0.2  $\mu$ m size. Structures with initial size less than 0.2  $\mu$ m are thermally stable and develop crystalline features. The sensitivity of these features to the carbonaceous impurities introduced during fabrication are currently being tested with the aid of an in situ plasma.@footnote 1@ @FootnoteText@ @footnote 1@work supported by UMD, NSF-MRSEC

#### NS-ThP19 Processing and Characterization of Nanometer Sized Copper Sulfide Particles, S. Seal, L. Bracho, C. Urbanik, M. Hampton, University of Central Florida; J. Morgiel, Polish Academy of Science, Poland

Nanomaterials, commonly characterized by their size smaller than 100 nm, have attracted a scant attention in the modern-day technology. These materials provide unique physical, mechanical and chemical properties in the nano-crystalline state. In this paper, sulfide nanoparticles of industrial interest are formed by sol-gel process using metal chloride precursors dissolved in a mixture of silica gel and organic cellulose network followed by a reaction with hydrogen sulfide gas. Particles are then heated in a vacuum oven. Variations in the sol-gel composition are used to study the particle growth rate and fractal density resulting from the reactivity of the large surface area nano particles. While scanning and transmission electron microscopy and x-ray diffraction are used to study their morphology and structure in the nanometer scale, x-ray photoelectron spectroscopy (XPS) is employed to understand the bonding chemistry and the stoichiometry of the sulfide particles. XPS results show a change in the binding energy of the sulfide particles with various temperature treatments. Secondary ion mass spectrometry is also used to show the distribution of precursor elements with depth, i.e. the chemical reactivity from surface to bulk. Nanometer sized gold particles are also produced using this method. The results from this study are expected to show promising applications and production of other oxides, sulfides and their compounds using this modified sol-gel synthesis.

#### NS-ThP20 Film Formation and the Onset of Multilayer Growth in Chloromethylphenylsilane Films as Determined by Atomic Force Microscopy, W.J. Dressick, Naval Research Laboratory; J.M. Calvert, Shipley Co.; M.-S. Chen, S.L. Brandow, Naval Research Laboratory

Organosilane films containing benzyl chloride functional groups are of interest for use as imaging layers in high resolution lithography due to the extreme sensitivity of the carbon-chlorine bond to cleavage by either photon or electron radiation. For applications where feature sizes are on the nanometer scale it is important to have an understanding of the surface coverage and intermolecular order of these films. We have developed protocols for the reproducible deposition of two organosilanes, p-chloromethylphenyltrichlorosilane (CMPTS) and 1-(dimethylchlorosilyl)-2-(p,m-chloromethylphenyl)ethane (CMPEMS), on native oxide silicon and fused silica substrates. Film growth was characterized using several techniques including UV absorbance, ellipsometry, and contact angle measurements. Atomic force microscopy was found to be a powerful tool for monitoring the onset of oligomer formation in the CMPTS films, ultimately allowing the macroscopic properties of the films to be correlated with their nanometer scale morphologies. Results indicate that film growth which is limited to direct chemisorption at the surface results in low coverage, disordered films. In the CMPTS system multilayer growth was found to be a predominant pathway even at sub-monolayer coverages. The significance of these observations on the design of imaging layers capable of molecular scale resolution in nanolithographic applications is discussed.

#### NS-ThP21 Room Temperature Fabrication of Transparent ZrO@sub 2@/Polymer Nanocomposite Thin-Films with Controlled Thickness by the Ionic Self-Assembled Monolayer (ISAM) Method, A. Rosidian, Y. Liu, R. Claus, Virginia Polytechnic Institute and State University

Nanocomposites of transparent multilayer structures of ZrO@sub 2@/polymer thin-films have been fabricated on silicon and quartz substrates utilizing the Ionic Self-Assembled Monolayer (ISAM) method. This method is based on the alternating adsorption of anionic and cationic polyelectrolytes in the aqueous forms. The deposition process was monitored by UV/Vis spectroscopy and ellipsometry. A linear behavior of both optical absorption and film thickness as the number of bilayers increases was observed, which indicated the formation of homogeneous and uniform thin-films on both substrates. The study also showed the control of film thickness of each bilayer of the thin-films increased with the ZrO@sub 2@ concentration. For the films with ZrO@sub 2@

concentrations of 10 and 30 mg/ml, the thickness of each adsorbed bilayer were 18 and 24 Ångstroms, respectively.

NS-ThP22 Nanowire Formation Using a Resistively-Heated Piezoresistive Cantilever, *T. Uchihashi, U. Ramsperger, H. Nejoh*, National Research Institute for Metals, Japan

The main aim of our research is to fabricate atomically thin metal wires on a clean sample surface in UHV, and to investigate the electronic transport properties of such systems. For that system, it is expected that new phenomena, e.g., single electron tunneling effect, quantization of conductance, will be observed, and that relation between a structure of the wire and transport properties will be clarified. The procedures required to reach this aim would be as follows. 1) Evaporate a defined electrode pattern on a clean sample using a through-hole mask in UHV. This pattern has macroscopic electrodes, on which the four-point-probe leads is pressed, and microscopic electrodes for which a gap distance is a few micrometers. 2) Draw atomically thin metal wires in the gap between the electrodes in UHV using a scanning tunneling microscope (STM) or an atomic force microscope (AFM). 3) Measure electronic transport properties of the wire in UHV, and at low temperatures if necessary. We succeeded in fabricating a through-hole mask using a discharge cutter machine and focused ion beam (FIB) machining. The discharge cutter machine is used for building the macroscopic pattern, and FIB for the microscopic structure of the through hole mask. Further we also succeeded in drawing thin gold wires with a width of a few tens of nanometer using an AFM cantilever. A piezoresistive cantilever (provided by Park Scientific Instruments) was first coated with gold by thermal evaporation. This cantilever can be heated up resistively with a power of about 30 mW by current running through it.@footnote 1@ The gold on the cantilever tip was transferred onto the surface of both a silicon and sapphire substrate. The minimum width of gold wires fabricated thus far is around 50nm. The fabrication method using a heated AFM cantilever can be applied not only to a conductive sample but also to a insulating sample like sapphire. This fact will eliminate the difficulty in measuring conductivity of !!! nano-scale wire, especially at room temperature. @FootnoteText@ @footnote 1@ H. J. Mamin, Appl. Phys. Lett. 69, 433 (1996)

NS-ThP23 Adsorbate Effect on Conductance Quantization in Metallic Nanowires, C.Z. Li, H. Sha, M. Adam, N.J. Tao, Florida International University

We have studied conductance quantization in metallic nanowires upon adsorption of molecules with different adsorption strengths. The conductance still changes in a stepwise fashion even in the presence of strong adsorption, and the average sharpness, length and number of the conductance steps remain unchanged. However, the step positions deviate significantly from the integer values of the conductance quantum, 2e2/h. While the deviation may be attributed to the scattering of the ballistic electrons by the adsorbates, evidence shows that the adsorbates also affect the conductance by changing the atomic configurations of the nanowires. @FootnoteText@ Financial support is acknowledged through grants from AFSOR (F49620-96-1-0346) and NIH (GM-08205).

### NS-ThP24 Substrate Effects on Electronic Properties of Atomic Chains, *T. Yamada*, MRJ, NASA Ames Research Center

Atomic chains, precise structures of adatoms created on an atomically regulated surface, are candidates for constituent elements in future electronics. It was predicted that Si chains were metallic and Mg chains were semiconducting, and a doping method was also discussed.@foot 1@ The substrate was assumed to work as a noninteracting template holding the adatoms. However, this scheme requires a low-temperature environment so that the adatoms will not displace from their ideal positions due to unwanted thermal agitation. For better structural stability, we may seek a scheme to allow the adatoms to form chemical bonding with the substrate atoms and secure their positions. The chemical bonding has two major effects on the chain electronic properties. First, only the remaining s and p orbitals in an adatom not used for the chemical bonding can decide chain band structures, rather than the full set of orbitals previously assumed.@foot 1@ Second, because of a possible HOMO energy difference between adatom and substrate atom, semiconducting chains are unintentionally doped. These effects are studied with a selfconsistent tight-binding method with universal parameters. With one adatom per unit cell, adatom chains are semiconducting (1) if adatoms are of group III and form one chemical bond per adatom, or (2) if adatoms are of group IV and form two chemical bonds. The previous result@foot 1@ of realizing semiconducting chains by group II adatoms without chemical bonds is consistent with this picture. When the HOMO energy in an adatom

is shallower than that in a substrate atom, the entire chain is positively polarized, unintentionally achieving p-type doping. When deeper, the chain is negatively charged, achieving n-type doping. Specific examples will be discussed. @FootnoteText@ @footnote 1@T. Yamada, Y. Yamamoto, and W. A. Harrison, J. Vac. Sci. Technol. B 14, 1243 (1996); T. Yamada, to appear in J. Vac. Sci. Technol. A 16 (1998).

NS-ThP25 Designing New Materials at the Molecular Scale - An Example in Etching and Deposition, J.A. Gurney, McGill University, Canada; E.A. Rietman, Bell Laboratories, Lucent Technologies; M.A. Marcus, KLA Instruments; M.P. Andrews, McGill University, Canada

We desire the ability to design molecular-scale components and new materials using a heuristic programming technique such as genetic algorithms. To this end we have been investigating the possibility of designing new materials in the space of cellular automata. Molecules interact, more or less, only with their nearest neighbors. This suggests that cellular automata (arrays of nearest neighbor interacting finite state machines) may be used for modeling the dynamics of molecules. Since it is well known that structures can be "grown" in the space of the cellular automata (CA) we conjecture that by manipulation of the rule table or rule vector describing the CA dynamics we can evolve desired structures in the CA space. To support this supposition we present work on modeling the etching and deposition (dissolution and growth) of crystals. We empirically find a mapping between the automaton rule table and the surface physics of the crystal. @FootnoteText@ @footnote 1@ J.A. Gurney, E.A. Rietman, M.A. Marcus, and M.P. Andrews, "Mapping the Rule Table of a 2-D Probabilistic Cellular Automaton to the Chemical Physics of Etching and Deposition", Submitted, 1998

#### NS-ThP26 Spectroscopic Studies of Carbon Nanotube by Ballistic Electron Projection Microscopy, J.-Y. Park, S.-H. Kim, Y.D. Suh, W.-G. Park, Y. Kuk, Seoul National University, Korea

Geometric and electronic properties of carbon nanotubes have been studied by ballistic electron projection microscopy(BEPM). An interference pattern between the scattered and transmitted e-beam was observed using coherent electron source from an atomically sharp emitter.@footnote 1@ In this work, a microcolumn in an SAFE(STM Aligned Field Emission) microcolumn system was replaced by carbon nanotubes or self-sustaining single crystal films on TEM grid.@footnote 2@ From images of single-walled carbon nanotubes, the performances of BEPM were tested. By adding an electrostatic energy analyzer, the electron energy loss of the nanotubes was measured. When the tip is positioned at < 2 nm, the tunneling I-V could be measured using BEPM. In addition to the nanotube. the results of free-standing single crystal metal and semiconductor will be presented. It was found that the Fourier transformation of inelastic scattering pattern gives information on scattering in the sample. @FootnoteText@ @footnote 1@H. -W. Fink, W. Stocker, H. Schmid, Phys. Rev. Lett. 65, 1204(1990). @footnote 2@J. -Y. Park et al., J. Vac. Sci. Technol. A 15, 1499(1997).

#### NS-ThP27 Synchrotron-Radiation-Induced Deposition of Nanocrystalline Particles, *R.A. Rosenberg*, *Q. Ma, B. Lai, D.C. Mancini,* Argonne National Laboratory

The high-intensity, high-energy x-rays produced by third-generation synchrotron radiation sources have made possible many new applications, such as deep x-ray lithography, that take advantage of the long penetration lengths of the x-rays in lower-Z materials. Recently, we have initiated a program to evaluate the prospects for using x-rays for materials processing by performing Surface Photochemistry Induced by X-ray Irradiation (SPIXI). X-rays have significant advantages over more conventional sources. The high energy x-rays produced by the Advanced Photon Source have deep penetration lengths for low-Z materials. Therefore, they can be used to induce chemical reactions on surfaces of solids immersed in liquids containing low-Z molecules. The most likely mechanism by which these reactions proceed is through the production of electrons caused by corelevel excitation of the substrate atoms. Therefore, the reaction rate should be both energy dependent and site specific. If the liquid contains a metal salt, then x-ray irradiation should induce deposition of metallic films or particles that can be in either polycrystalline or nanocrystalline forms. We present preliminary results which demonstrate the feasibility of the SPIXI approach for deposition of nanocrystalline particles. In particular we have deposited both gold and silver nanoparticles in liquids containing salts of the appropriate ion. In addition thin films have been formed on Mo substrates. In this paper we discuss the experiments, their results and prospects for future development. The submitted manuscript has been created by the University of Chicago as Operator of Argonne National

Laboratory ("Argonne") under Contract No. W-31-109-ENG-38 with the U.S. Department of Energy. The U.S. Government retains for itself, and others acting on its behalf, a paid-up, nonexclusive, irrevocable worldwide license in said article to reproduce, prepare derivative works, distribute copies to the public, and perform publicly and display publicly, by or on behalf of the Government

# Friday Morning, November 6, 1998

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

#### **Innovative Nanoscale Measurements**

Moderator: S. Semancik, National Institute of Standards and Technology

### 8:20am NS+AS-FrM1 Nanoscale Variations in Surface Potentials at Interfaces, B.D. Huey, D.A. Bonnell, University of Pennsylvania, US

Nanoscale variations in the electronic properties of individual oxide grain boundaries have been directly measured. In-situ application of atomic force microscopy used as a Kelvin probe maps local field variations in the presence of lateral applied bias and current flow within an oxide grain boundary device, providing measurements while the device is in operation. The in-situ experiment involves: 1) applying a lateral bias across microfabricated contacts on varistor and SrTiO@sub 3@ bicrystals, and 2) simultaneously mapping the surface potential in the vicinity of grain boundaries. Voltage dependent properties of both multiple boundaries as well as carefully characterized individual interfaces are thus measured. The non-linear voltage dependence of potential barriers at the grain boundaries is additionally obtained at the nanoscale as a function of local orientation, chemical content, and position.

#### 8:40am NS+AS-FrM2 Noncontact Measurement of Electrical Dissipation using Ultrasensitive Cantilevers, *T.D. Stowe*, Stanford University; *D. Rugar*, IBM Almaden Research Center; *D.J. Thomson*, University of Manitoba, Canada; *T.W. Kenny*, Stanford University

We have used ultralow loss 0.17 µmm thick silicon cantilevers to measure electrical dissipation in insulators and doped silicon samples using a technique similar to the one originally developed by Denk and Pohl.@footnote 1@ Images were taken by recording the mechanical Q of a self-oscillating cantilever as it was scanned 10-100 nm above the sample surface in a perpendicular orientation. All experiments were performed at room temperature in vacuum with cantilevers having 10@super -4@-10@super-3@ N/m spring constants and 10-100 kHz resonant frequencies. Electrical dissipation was measured as function of applied voltage, tipsample distance, and resistivity. We were able to measure electrical dissipation as small as 10@super -14@ N-s/m and ohmic losses as small as 10@super -18@ Watts. Using tips with 50 nm radii, we were able to simultaneously image permanent charge in the surface oxide and doping levels between 10@super 15@-10@super 19@ /cm@super 3@ in silicon with 200 nm spatial resolution. Possibilities for improving the spatial resolution and doping sensitivity of this technique will be discussed. @FootnoteText@ @footnote 1@W. Denk and D. W. Pohl, Appl. Phys. Lett. 59, 2171 (1991).

#### 9:00am NS+AS-FrM3 Nanocalorimetry for Thermodynamic Measurements of Nanostructures, L.H. Allen, University of Illinois, Urbana-Champaign INVITED

This talk will focus on a novel calorimetry technique@footnote 1,2,3@ for measuring the thermodynamic properties of nanometer size material. The thermodynamic properties of material having small nanometer dimensions can be considerably different as compared to material in bulk form (e.g., the reduction of melting point). This occurs because of the tremendous influence of the surface energy. Conventional differential scanning calorimetry (DSC) techniques are extremely difficult to apply to the study of small structures because the total amount of heat generated during the transformation is too small as compared with the background heat capacity of the calorimeter. The new nanocalorimeter is fabricated using standard MEMS thin film techniques and it has the capability of measuring the dynamics of the energy exchange at the level of 0.2 nanojoule. This technique is so sensitive that it can easily measure the melting process of 1 Angstrom of Sn, which has been deposited on a Si-N surface. Results of specific materials studies will be discussed including the size-dependent melting point depression of small particles of Sn and Al and preliminary work on the coalescence of small clusters. @FootnoteText@ @footnote 1@S.L. Lai, J.Y. Guo, V. Petrova, G. Ramanath and L.H. Allen, "Size-Dependent Melting Properties of Small Tin Particles: Nanocalorimetric Measurements", Phys. Rev. Lett. 77, 99-103 (1996) @footnote 2@S.-L. Lai, P. Infante and L. H. Allen, "Heat capacity of Sn nanostructures via Thin Film Differential Scanning Calorimetry," Appl. Phys. Lett., 70, 43-46(1997). @footnote 3@S. L. Lai, J. Carlsson and L. H. Allen, "Melting Point Depression of Al Clusters Generated During the Early Stages of Film Growth: Nanocalorimetry Measurements," Appl. Phys. Lett., Appl. Phys. Lett. 72, 1098 (1998)

9:40am NS+AS-FrM5 Recent Advances in Scanning Capacitance Microscopy, C.C. Williams, J.S. McMurray, V.V. Zavyalov, J. Kim, University of Utah INVITED

Scanning Capacitance Microscopy is a rapidly developing technique for the characterization of the electronic properties of semiconductor materials on a 10 nanometer scale. Oxide, semiconductor interface and near surface "bulk" properties can be studied by this technique. Recently, new capabilities have been developed to extract quantitative dopant/carrier profiles near electrical junctions in cross-sectioned devices. Built-in internal electric fields have been measured and compared with electrical models. Random distributions of nanometer scale "defects" have been observed at the surface of uniformly doped and oxidized silicon samples. Two-dimensional diffusion of dopant impurities has been measured by SCM and directly compared with predictions of TSUPREM4. These new developments and capabilities of the Scanning Capacitance Microscope will be described and presented.

# 10:20am NS+AS-FrM7 Nanometer-scale Electrical Characterization of Semiconductor with a Scanning Capacitance Microscope, *H. Tomiye*, *Y. Takafumi*, Tohoku University, Japan

Recently, nanometer-scale material characterization has become a necessity in Si technology. The scanning probe microscope is one of the most powerful characterization techniques at this scale. A variety of information can be obtained by this technique which is of importance in the assessment of material and device aspects of silicon. Scanning capacitance microscope (SCaM) can clearly show local variation of capacitance, which reflects the electrical properties of a Si substrate, SiO@sub 2@/Si interface and SiO@sub 2@ layer. We have developed a SCaM/AFM consisting of a W wire cantilever and a capacitance sensor. The unique features of our microscope are as follows: (1) Our SCaM can directly detect a sample capacitance with / without using lock-in amplifier. It means we can measure capacitance (C) of the sample in addition to the capacitance derivative (dC/dV). (2) We can simultaneously obtain a SCaM image and C-V characteristics. It means we can achieve quantitative measurements of the capacitance at a nanometer-scale. (3) The spatial resolution for the capacitance measurements is estimated to be less than 20 nm. It is demonstrated that the local impurity concentration profiling of lateral p-n junction is achieved by the C-V measurements. We have injected electrical charge into a SiO2 layer and investigated the nature of charge storage at the SiO@sub 2@/Si system by the SCaM and C-V characterization. A shift of the flat-band voltage due to the trapped charges is observed, which enables one to estimate the density of trapped charge. This paper will report on the development of a SCaM and its applications to the characterization of SiO@sub 2@/Si and fabrication of a charge storage device.

#### 10:40am NS+AS-FrM8 A Study on the Post-stress Charges in SiO@sub2@ Films on Si by Scanning Capacitance Microscope, K. Mang, Samsung Electronics, Korea; C.J. Kang, G.H. Buh, C.K. Kim, S. Lee, C. Im, Y. Kuk, Seoul National University, Korea

Using scanning capacitance microscope, The induced traps on SiO@sub2@ were imaged with @<=@20nm spatial resolution. The static and dynamic behaviors of the electronic charges were evaluated. After a voltage stress, an anomalous post-stress charge generation and relaxation effect were found. Depending on the polarity of stress voltage, post-stress tip voltage and stressed time, different relaxation trend exists at the stressed area. The induced charge density in the stressed SiO@sub2@ film was higher with the stressed time The trap dynamics is also a function of initial stress field. With the high field applied to the SiO@sub2@ film, the larger and faster generation(or relaxtion) was observed. After the trapped charges are formed, the amount and polarity of the charge vary dynamically. It is believed that thermal excitation or tunneling of one or two electrons, can cause the turn around effect, which has been poorly understood so far.

### 11:00am NS+AS-FrM9 Imaging Buried Interfacial Lattices with Quantized Electrons, *I.B. Altfeder*, *D.M. Chen*, The Rowland Institute for Science

We demonstrate that interfacial lattices buried under as much as 100 Å of a metal can be directly imaged by low temperature scanning tunneling microscopy with an unexpectedly high lateral resolution.@footnote 1@ To achive such a remarkable resolving power we expolite the presence of the quantum-size sigularities in the electron energy spectrum in the metal as well as its high sensitivity to the defects at the boundaries. Our theoretical model shows that this unique phenomena can be atrributed to the nondifractive scattering of the quantized electron waves at the interface as a result of their highly anisotropic motions in a two dimensional nanostructure. @FootnoteText@ @footnote 1@I. B. Altfeder, D. M. Chen, K. A.

### Friday Morning, November 6, 1998

Matveev, Phys. Rev. Lett. (in press). @footnote 2@I. B. Altfeder, K. A. Matveev, D. M. Chen, Phys. Rev. Lett. 78, 2815 (1997).

11:20am NS+AS-FrM10 Nanoparticle Near-Field Spectroscopy by a Microscopically Narrow (Subnanometer) Electron-Beam, H. Cohen, Weizmann Institute of Science, Israel; T. Maniv, Technion, Israel; Y. Rosenfeld Hacohen, R. Tenne, Weizmann Institute of Science, Israel; O. Stephan, C. Colliex, University of Paris-Sud, France

Single nanoparticle near-field spectroscopy is performed in a scanning transmission electron microscope at non-intersecting beam-particle configuration. Separating the surface collective modes from the entire excitations spectrum, the energy loss signal is quantitatively accounted for, using a relatively simple theoretical model. Advantaged by the sub nanometer size of the e-probe, the highly controlled beam-surface distance introduces an effective window in momentum space, exposed to a long wavelength relativistic dispersion, which provides an enhanced sensitivity to beam and particle size effects. The spatial dispersion of the particle dielectric function, associated with the electronic band structure, is practically filtered out. "Particle spectroscopy", namely the selective excitation of modes which characterize the particle geometry and size, is available at selected beam-particle distances.

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