

## Nanotubes - Science and Applications

### Room 309 - Session NM+NS-MoM

#### Carbon Nanotubes: Functionalization and Applications

Moderator: P.M. Ajayan, Rensselaer Polytechnic Institute

#### 8:20am NM+NS-MoM1 Surface Interactions Used to Probe Metallic Carbon Nanotubes, *M. Dresselhaus*, MIT **INVITED**

A brief overview will be given of the remarkable structure and properties of carbon nanotubes and how surface interactions are used for some of these studies. Carbon nanotubes are tiny structures of molecular dimensions in the form of hollow cylinders with about 20 carbon atoms around the circumference of the cylinders and microns in length. The unique electronic properties of carbon nanotubes are that they can be either semiconducting or metallic depending only on their geometry. From this, stem other remarkable and unique properties of their vibrational spectra, allowing us a means to distinguish metallic from semiconducting nanotubes in samples containing both. Interactions of the nanotubes with nanostructured metallic substrates have provided a powerful tool to gain a fundamental understanding about why the Raman spectra of metallic nanotubes are different from those for semiconducting nanotubes. Though less than a decade since their discovery, carbon nanotubes are already finding practical applications based on their unique properties.

#### 9:00am NM+NS-MoM3 Purification and Functionalization of Single-wall Carbon Nanotubes, *I. Chiang, R. Saini, J. Margrave, R. Hauge, R.E. Smalley, R. Billups*, Rice University

A purification method has been developed which leads to 99.9% pure single wall nanotubes. It combines the well-known nitric acid treatment with water reflux and a two-stage gas-phase oxidation. Air oxidation of SWNTs is correlated to the amount of metals in the samples. For sidewall fluorinated SWNTs, two distinct types of C-F bonds have been observed, ionic and covalent bonds. These 'fluorotubes' have served as precursors for further substitution on the nanotubes, such as sidewall alkylation etc. The degree of substitution is found to correlate with the extent of covalent sidewall C-F bonding.

#### 9:20am NM+NS-MoM4 Chemical Functionalization of Single-Walled and Multi-Walled Carbon Nanotubes through Ion Bombardment: Predictions from Molecular Dynamics Simulations Nanotubes, *B. Ni, S.B. Sinnott*, The University of Kentucky

Molecular dynamics simulations have been performed to study the bombardment of single-walled and multi-walled (double and triple) nanotube bundles by  $\text{CH}_3^+$  ions at impacting energies 10, 45, and 80 eV. The reactive empirical bond order potential for hydrocarbons was used in the classical simulations. The simulations predict that there is a high probability of radical or fragment adsorption to the nanotube walls which could serve as precursors to the chemical functionalization of the nanotube walls. In addition, the simulations show that ion bombardment at 80 eV can lead to cross-linking among the single-walled nanotubes that could stabilize the bundle relative to shear. The multi-walled nanotubes are predicted to be stiffer to scattering than the single-walled nanotubes, leading to increased fragmentation of the incident ions at lower energies. The results are compared to preliminary experimental results for the ion bombardment of multi-walled nanotubes and found to be in good agreement. This work was supported by the NSF (CHE-9708049) and the NSF MRSEC at the University of Kentucky, DMR-9809686) and by the NASA Ames Research Center (NAG 2-1121). @FootnoteText@ @footnote 1@S.B. Sinnott, L. Qi, O.A. Shenderova, D.W. Brenner, in Chapter 1 of Volume IV of ADVANCES IN CLASSICAL TRAJECTORY METHODS, Molecular Dynamics of Clusters, Surfaces, Liquids, and Interfaces, Ed. W. Hase (JAI Press, Inc., Stamford, CT, 1999), pp. 1-26. @footnote 2@B. Ni and S.B. Sinnott, Physical Review B 61, 2000 (in press).

#### 9:40am NM+NS-MoM5 A Novel Mechanism of Hydrogen Storage in Carbon Nanotubes, *Y.H. Lee, S.M. Lee*, Jeonbuk National University, Korea

We have carried out systematic calculations for hydrogen adsorption and storage mechanism in the nanotubes. Hydrogen atoms first adsorb on the tube wall in an arch type and zigzag type up to a coverage of  $\theta=1.0$ , and are stored in the capillary as a form of  $\text{H}_2$  molecule at higher coverages. Hydrogen atoms can be stored dominantly through the tube wall by breaking the C-C midbond, with relatively low activation barrier of 1.51 eV, while preserving the wall stability of a nanotube after complete

hydrogen insertion, rather than by the capillarity effect through the ends of nanotubes. In the hydrogen extraction processes,  $\text{H}_2$  molecule in the capillary of nanotubes first dissociates and adsorbs onto the inner wall, and is further extracted to the outer wall by the flip-out mechanism. Our calculations describe suitably an electrochemical storage process of hydrogen, which is applicable for the secondary hydrogen-battery.

#### 10:00am NM+NS-MoM6 Local Solvation Shell Measurement in Water using a Carbon Nanotube Probe, *S.P. Jarvis*, JRCAT-NAIR, Japan; *T. Uchihashi*, JRCAT-ATP, Japan; *H. Tokumoto*, JRCAT-NAIR, Japan

Oscillatory forces between two approaching surfaces in solvent have long been the subject of study due to their possible influence on any surface-surface interactions mediated through a liquid or in the presence of a fluid film. Of particular interest is water, due to its omnipresence in all but the most stringently controlled environments and its role as the primary medium for biological interactions. Combining a carbon nanotube probe with an AFM has enabled us to measure oscillatory forces in water on approaching a surface that has been laterally characterized on a nanometer scale. One important aspect of the utilization of carbon nanotubes as the AFM probe is to remove the unwanted hydrodynamic damping effect caused by the bulk of the tip. We used a multi-walled carbon nanotube attached to a PtIr coated silicon lever in a specially designed FE-SEM. Another aspect is the usage of a magnetically activated AFM, which has been possible to resolve molecular layers of large molecules. With this method, magnetic material is deposited directly behind an AFM tip on the back-side of the cantilever so that the tip position can be controlled by the addition of a magnetic field. The lever can be vibrated in an oscillating magnetic field in order to make dynamic measurements. This success opens up the possibility of investigating water layers under a variety of experimental conditions and as a function of precise lateral position on any surface including biological membranes and macromolecules. Among the many and varied roles of water layers are effects on biomolecular adhesion, colloid dispersion and tribology, which can now be investigated with nanometer lateral resolution.

#### 10:20am NM+NS-MoM7 Single Wall Nanotube Probes for Structural and Functional Imaging in Fluid, *L. Chen, J. Hafner, C. Cheung, C.M. Lieber*, Harvard University

Scanning force microscopy is a powerful tool for probing nanometer scale objects in fluid, ambient and vacuum environments. The contrast of SFM is based on the interaction between surface and probe which is additive over a wide spectrum of forces including Van der Waals, electrostatic and magnetic forces. Therefore, the resolution of SFM imaging greatly depends on the geometrical and mechanical properties of the probes. Carbon nanotubes make potentially ideal tips for SFM. First, carbon nanotubes can give unprecedented high resolution in structural imaging because of the intrinsic small diameters, high aspect ratio and reversible buckling. Second, carbon nanotubes can be functionalized to give chemically well-defined SFM probes, which enables functional or chemically sensitive imaging. Here we report recent progress in addressing critical issues associated with nanotube probes including the preparation of nanotube tips, structural imaging in fluid, and the functionalization of nanotube ends. Nanotube probes have been prepared by chemical vapor deposition (CVD) on commercial cantilever-probe surfaces. CVD nanotube probes have been used to image individual molecules of supercoiled DNA plasmid pBR322 on mica-fluid interface with high resolution. The relaxation of the supercoiled molecules was observed in real time in aqueous buffer solution. The chemical functionality of the nanotube end group was identified as carboxylic groups, by carrying out force titration experiments. Nanotube probes have been functionalized with synthetic oligonucleotides, and the resulting probes were capable of recognizing complementary oligonucleotide strands on surfaces. The force needed to unbind the 14 base pair duplexes was shown to be 450pN, which is in agreement with previous chemical force microscopy measurements.

#### 10:40am NM+NS-MoM8 Nonlinear Optical Properties of Some Polymer/Multi-walled Carbon Nanotube Composites, *Z.X. Jin*, National University of Singapore, Singapore; *X. Sun, G.Q. Xu, S.H. Goh, W. Ji*, National University of Singapore

Several polymer-coated and polymer-grafted multi-walled carbon nanotubes (MWNTs) were synthesized and characterized using TEM and HRTEM. The polymer-coated or polymer-grafted MWNTs formed stable solutions in DMF. Their nonlinear optical properties were investigated using 532 nm nanosecond laser pulses. These polymer-MWNT composites still possess strong nonlinear optical properties.

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## Surface Science

### Room 209 - Session SS2+NS+TF-MoM

#### Nucleation and Growth

Moderator: G.L. Kellogg, Sandia National Laboratories

8:40am **SS2+NS+TF-MoM2 Homoepitaxial Growth of Ni on Ni(110) : Surface with Anisotropic Diffusivity and Energy Barriers, B.-Y. Choi**, Seoul National University, Korea; S.-J. Kahng, University of Illinois at Urbana-Champaign; J.-Y. Park, Y. Kuk, Seoul National University, Korea

The homoepitaxial growth of Ni was studied on Ni(110) surface with scanning tunneling microscopy. The Ni(110) surface does not reveal surface reconstruction, but it has corrugated channels along the [1 -1 0] direction and the resultant surface diffusion anisotropy. Homoepitaxial islands grow with high aspect ratio, as large as 1:1000, due to i) the diffusion anisotropy and ii) the energy barrier from an [1 -1 0] side wall to an [001] side wall around the adatom islands. The aspect ratio of the islands decreases with increasing temperature. The competition between these two mechanism will be discussed. At the high coverage ( $\theta \geq 10\text{ML}$ ), the aspect ratio decreases substantially and the growth structure transforms to 3D islands without asymmetry. At these coverage, the surface roughness saturates. Although the microscopic origin is unclear, the phenomenon can be explained with a KPZ type equation with some modifications.

9:00am **SS2+NS+TF-MoM3 Thermodynamics of Thin Film Alloys: Experimental and Theoretical Study of Ag and Co on Ru(0001), G.E. Thayer**, University of California, Davis; A.K. Schmid, V. Ozolins, N.C. Bartelt, Sandia National Laboratories; S. Chiang, University of California, Davis; R.Q. Hwang, Sandia National Laboratories

It has long been known that the structure of materials in thin film configurations can differ significantly from their bulk phase. This is particularly true of alloy films. It has recently been shown that strain induced by the lattice mismatch between substrate and film can lead to the formation of novel alloys that do not exist in the bulk. One prototypical example is the system of one monolayer Ag/Co films grown on Ru(0001). In the bulk, Ag and Co are immiscible. However, on the Ru surface alloy phases of distinct stoichiometries are formed. We have investigated the phase diagram of 1ML AgCo/Ru(0001) films using scanning tunneling microscopy (STM) and first principles calculations. For Ag rich films, segregation between a pure Ag phase containing dislocations and a pseudomorphically strained alloy of stoichiometry  $\text{Co}_{0.6}\text{Ag}_{0.4}$  is found. The driving force for the phase segregation is the competition between two strain relief mechanisms: dislocation formation and alloying. In the Ag saturated  $\text{Co}_{0.6}\text{Ag}_{0.4}$  alloy, atomic resolution STM images show that the alloy consists of a Co film containing disordered, elongated Ag droplets with an average size of 30 atoms separated by an average distance of 10 Co atoms. As the composition of Ag in the film is decreased, the Ag droplets become more uniform in size and decrease to an average minimum size of 10 atoms. Simultaneously, the distribution of the droplets becomes uniform and dilute. This behavior has been predicted using first principles, local density approximation (LDA) calculations. These calculations, together with atomically resolved STM images, have quantified configuration energies for various stoichiometries of this system. This investigation has led to a quantitative interpretation of the competition between the chemically repulsive interaction of Ag and Co (also seen in their bulk miscibility gap) and the strain fields in the thin film alloy that they form.

9:20am **SS2+NS+TF-MoM4 Theory of the Nucleation and Growth of Iron on GaAs, S.C. Erwin**, Naval Research Laboratory; M. Scheffler, Fritz Haber Institute, Germany

By growing ultrathin films of ferromagnetic metals on semiconductor substrates, it is possible to create a partially spin-polarized electrical current in the semiconductor. This phenomenon is the starting point for a wide variety of magnetoelectronic devices based on the "spin-valve" effect, whereby an externally applied magnetic field can switch the current on and off. Considerable experimental effort has focused on ultrathin films of Fe grown by MBE on GaAs, in part because their small lattice mismatch results in nearly epitaxial films. Although a number of experiments have contributed to a detailed description of the phenomenology of magnetism in these films, little is known about the microscopic physics of magnetism at an intimate metal-semiconductor interface. We use spin-polarized density-functional total-energy methods to investigate the nucleation and initial growth phases of Fe on GaAs(001), focusing on the roles played by diffusion, magnetism, and defect chemistry. Our findings include the following results: (1) magnetism develops in the very first monolayer, and

is in fact enhanced by the presence of the interface; (2) surface diffusion is strongly suppressed in the submonolayer regime by the tendency of Fe adatoms to form very stable Fe-As bonds. Finally, we discuss the influence of the initial GaAs reconstruction on the structure and magnetic properties of the Fe film.

9:40am **SS2+NS+TF-MoM5 Nucleation and Growth in Metal-on-Metal Epitaxy - Complications beyond Simple Systems, R.J. Behm**, University of Ulm, Germany

INVITED

The quantitative atomic scale understanding of a surface process, as a sequence of elementary steps, is one of the primary objectives in Surface Science. Molecular beam epitaxial growth (MBE) is one of the first areas where extensive and systematic studies, both experimental and theoretical, have led to such kind of understanding, at least for simple processes such as nucleation and two-dimensional growth on inert, bare and non-reconstructed substrates. In this talk I will focus on complications which arise when these conditions are not fulfilled, predominantly on the effect of coadsorbed species on nucleation and growth processes. These coadsorbates, which are generally present, e.g., in the case of chemical vapor deposition (CVD), are shown to affect not only the nucleation behavior, but may lead also to the formation of new, (metastable) film structures. A second topic to be discussed involves nucleation and growth on 'unstable' surfaces, where growth competes with exchange processes and surface alloy formation. On such systems and under certain conditions metal deposition was found to cause a severe restructuring of the substrate, leading to pit formation. Mechanistic ideas will be presented and discussed. @FootnoteText@ @footnote 1@H. Brune, Surf. Sci. Rept. 31, 121 (1998).

10:20am **SS2+NS+TF-MoM7 Preferential Nucleation of Metal Nanoclusters on S(4x4)/W(111), Q. Wu, W. Chen, T.E. Madey**, Rutgers, The State University of New Jersey

Interactions of several metals (Cu, Pt and Pd) with the highly ordered S(4x4)/W(111) surface have been studied by means of Auger electron spectroscopy (AES), low energy electron diffraction (LEED), and scanning tunneling microscopy (STM). The substrate is a sulfur-induced nanoscale reconstruction of W(111) with (4x4) periodicity, characterized by broad, planar terraces ( $\sim 30$  nm in width). We find that fractional monolayers of vapor-deposited Cu grow as 3D clusters on the S(4x4) surface over a wide coverage range. At low Cu coverages ( $\theta \leq 0.1$  ML), Cu nanoclusters are observed to nucleate preferentially at characteristic 3-fold hollow sites on the S(4x4) surface; there is a clear energetic preference for one type of site over others. The formed Cu nanoclusters are uniform in size ( $\sim 0.7$  nm) up to 0.25 ML, indicating self-limiting growth. As coverage increases, additional sites are populated and Cu clusters grow in size. On the other hand, Pt and Pd exhibit a different behavior, disordering the (4x4) reconstruction and adsorbing beneath the outer S-layer. STM data are supported by LEED and AES measurements. The data are interpreted in terms of relative surface free energy, relative reactivity, and the metal-W, metal-S, and S-W binding energies, as supported by our previous TPD data.

10:40am **SS2+NS+TF-MoM8 Ultra-thin Al Films Grown Epitaxially on CaF@sub 2@/Si(111), Y.V. Shusterman, N.L. Yakovlev, L.J. Schowalter**, Rensselaer Polytechnic Institute

Electron scattering at metal boundaries becomes increasingly important as interconnect structures shrink further. To improve our understanding of this phenomenon, studies of electron transport through ultra-thin metal films are needed. In such experiments, it is desirable to have smooth, single crystal metal layers grown on an insulating substrate. For this reason, we are studying epitaxial growth of Al on CaF@sub 2@/Si(111), with metal thickness as low as several nanometers. The structures discussed in this work were grown by molecular beam epitaxy and investigated by reflection high-energy electron diffraction (RHEED), scanning tunneling microscopy (STM) and atomic force microscopy (AFM). Hydrogen-terminated Si(111) was used as a substrate. The CaF@sub 2@/Si interface was formed at 700°C, followed by fluoride overgrowth at temperatures below 200°C, which resulted in improved continuity of the subsequent Al layer. The roughness of the final CaF@sub 2@ surface was below 1nm, as measured by AFM. The Al films grew epitaxially, as revealed by RHEED patterns that only contained streaks. The spacing between the streaks from Al was about 4/3 times larger than that from CaF@sub 2@, in agreement with bulk lattice mismatch. The growth proceeded through formation of 3-dimensional islands that then coalesced as the thickness increased. Using an Al deposition rate above 1nm/s at room temperature, we were able to obtain continuous films as thin as 10nm with roughness less than 2nm. The STM images of these films showed atomic steps, some of them originating

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at screw dislocations. The density of these dislocations was around  $10^{11}$  cm<sup>-2</sup>. The dependence of Al conductivity on film thickness will also be presented.

## Nanotubes - Science and Applications

Room 309 - Session NM+NS-MoA

### Carbon Nanotubes: Nanoelectronics and Field Emission

Moderator: S. Sinnott, University of Kentucky

#### 2:20pm NM+NS-MoA2 Analysis of Long-Channel Nanotube Field-Effect Transistors (NT FETs), *T. Yamada*, NASA Ames Research Center

Recent experiment on carbon NT p-channel FETs with a long channel (3  $\mu\text{m}$ )<sup>1</sup> is analyzed theoretically. They observed saturation of drain current ( $I_{\text{d}}$ ) as a function of drain voltage ( $V_{\text{d}}$ ), which plays a crucial role in digital applications. Two possible mechanisms can make the carrier acceleration by  $V_{\text{d}}$  ineffective and bring about the  $I_{\text{d}}$  saturation: (1) Coulomb repulsion from other carriers forming a self-consistent spatial distribution, leading to channel pinch-off; (2) phonon scattering due to a large field created by  $V_{\text{d}}$ , leading to carrier velocity saturation. Since the former causes the saturated current  $I_{\text{d}}$  to depend quadratically on gate voltage ( $V_{\text{g}}$ ) and the latter linearly, two mechanisms are distinguishable. Noticing the quadratic dependence in their measurement, we argue that the Coulomb-induced pinch-off formation was the mechanism in these long-channel NT FETs. The maximum field was about 10 kV/cm and the velocity saturation for holes still did not occur. This field is comparable to that of electrons in silicon.  $I_{\text{d}}-V_{\text{g}}$  characteristics did not show a sharp rise at the onset of strong inversion, during the transition from accumulation to inversion, as  $V_{\text{g}}$  was increased. This is significantly different from the familiar behavior of metal-oxide-semiconductor FETs. In NT FETs,  $I_{\text{d}}$  was practically zero for a wide range  $V_{\text{g}} = 3$  to 40 V beyond accumulation. We argue that a high Schottky barrier for electrons existed at the source/drain metal-semiconductor contact and the electron flow was blocked in the inverted case although the hole flow was not in the accumulated case. Making Ohmic contact to both p- and n-NTs is mandatory for complementary circuitry, and will have to be explored for future electronics. <sup>1</sup> Zhou, J. Kong, and H. Dai, Appl. Phys. Lett. 76, 1597 (2000).

#### 2:40pm NM+NS-MoA3 Electrical Transport in Carbon Nanotubes, *Ph. Avouris*, P.G. Collins, R. Martel, H.R. Shea, IBM T.J. Watson Research Center; *H. Stahl*, J. Appenzeller, Physikalisches Institut, Germany INVITED

We will discuss studies of the electronic structure and electrical transport properties of individual and ropes of single-wall (SW) and multi-wall (MW) carbon nanotubes (CNTs). Both metallic and semiconducting NTs have been investigated. In the case of metallic NTs we will present results on the observation and interpretation of negative magneto-resistance, weak localization, strong localization, anti-localization, and Coulomb blockade phenomena. In the case of semiconducting NTs, we have observed band-bending, field switching, carrier depletion and inversion phenomena. Some evidence for non-local transport at elevated temperatures in MWNTs will be discussed. In the case of SWNTs ropes we have been able to obtain results on the efficiency of inter-tube electrical transport. Finally, the operation and characteristics of model NT-based devices such as field-effect transistors, single-electron transistors and low-pass filters will be discussed.

#### 3:20pm NM+NS-MoA5 First Principles Study of Electronic Properties of Molecule Functionalized Carbon Nanotube, *J. Zhao*, A. Buldum, J.P. Lu, University of North Carolina at Chapel Hill; *J. Han*, NASA Ames Research Center

We studied the functionalized carbon nanotubes by using first principles methods based on density functional theory (DFT). The adsorption energy and structures are studied for various molecules including:  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{NO}_2$ ,  $\text{CO}_2$ ,  $\text{NH}_3$ . The electronic structures calculations show that SWNT can be either charge donor or acceptor depending on the molecule. Thus the conductive properties of SWNTs can be dramatically changed by exposing to gases.

#### 3:40pm NM+NS-MoA6 Effect of Commensurate Contact on the Resistance Across the CNT/HOPG Interface, *S. Paulson*, M.R. Falvo, A. Seeger, R.M. Taylor II, S. Washburn, R. Superfine, The University of North Carolina

We report measurements of the effect of atomic interlocking on the mechanical behavior and electrical conduction between bodies. We have manipulated carbon nanotubes on an HOPG substrate with a conducting AFM tip as an electrical probe. Along with our lateral force evidence of commensurate contact, we present the first data measuring the change in

contact resistance between two atomically smooth surfaces as they go between the commensurate and incommensurate states. The conducting AFM tip contacts the CNT from the top and current is collected in the HOPG substrate, therefore our measurement of resistance is across the CNT diameter as opposed to its length. We find very low resistance for this circumferential current as compared to longitudinal currents that have been reported. Our results will be interpreted in light of models of coupling between the tip and the various electrical modes in the CNT. This work was supported by the National Science Foundation (HPCC, ECS), the Office of Naval Research (MURI), and National Institutes of Health (NCRR).

#### 4:00pm NM+NS-MoA7 Ultra-Low Bias Operation of Field Emitter using Single Wall Carbon Nanotube Directly Grown onto Silicon Tip by Thermal CVD, *K. Matsumoto*, Electrotechnical Laboratory, Japan; *S. Kinoshita*, Meiji University, Japan; *Y. Gotoh*, Tsukuba University, Japan; *T. Uchiyama*, Advanced Technology Institute, Japan; *S. Manalis*, Massachusetts Institute of Technology; *C. Quate*, Stanford University

The new carbon nanotube field emitter with single wall carbon nanotubes of a diameter of  $1\sim 2\text{nm}$  which were grown directly by thermal CVD onto the Si tips and protruded from them<sup>1</sup> was developed. Owing to the 10 to 20 times smaller diameter of nanotube than the conventional Si tip, the new carbon nanotube field emitter showed the ultra-low threshold bias of 10V for the field emission of electron which is more than  $10\sim 50$  times smaller value than the conventional Si emitter. The n-type silicon was etched by  $\text{SF}_6$  gas to form the 10900 silicon tips. After the chemical catalyst was spin coated, the sample was set in the furnace with hydro-carbonate gas flow at high temperature. The single wall carbon nanotube then started to grow and followed up the wall of the silicon tip to the top and protruded from the silicon tip. Three kinds of spacers of  $6\mu\text{m}$ ,  $10\mu\text{m}$ ,  $21\mu\text{m}$  were prepared to change the distance between the anode and the carbon nanotube emitter. When the spacer is  $21\mu\text{m}$ , the electrons began to emit at the applied bias of  $\sim 25\text{V}$ . The narrower the spacer, the threshold bias becomes smaller. When the spacer is  $6\mu\text{m}$ , the electron starts to emit at the applied bias of as small as 10V. In the Fowler-Nordheim plot, the current follows the linear lines in  $4\sim 5$  orders of magnitudes even at the different spacer thickness, which means the electrons are really field emitted from the carbon nanotube through the Fowler-Nordheim tunneling. This single wall carbon nanotube field emitter could be applicable to any kind of low power consumption flat panel displays in future. <sup>1</sup> J. Kong, C. Zhou, A. Morpurgo, H. T. Soh, C. F. Quate, C. Marcus, H. Dai, Applied Physics A69, p. 305, (1999).

#### 4:20pm NM+NS-MoA8 Field Emission Properties of Vertically Aligned Carbon Nanotubes Dependent Upon Gas Exposures and Growth Conditions, *S.C Lim*, D.J. Bae, K.H. An, Y.C. Choi, H.J. Jeong, Y.H. Lee, Jeonbuk National University, Korea

Vertically aligned carbon nanotubes have been grown with different growth conditions by microwave plasma chemical vapor deposition and thermal chemical vapor deposition. The field emission properties of such grown carbon nanotubes are studied. Carbon nanotubes under high bias voltage are exposed to hydrogen, nitrogen, and oxygen. After each exposure, changes on turn-on voltage and slope of Fowler-Nordheim plots are observed. The saturation region of emission current at high bias voltage has been shifted. Degradation of field emission current from hydrogen and oxygen exposures has been observed. The ratio of change of emission current shows that oxygen exposure degrades the emission current more severely.

#### 4:40pm NM+NS-MoA9 Study on Field Emission Mechanism of Carbon Nanotube using High-resolution Electron Microscopy, *T. Kuzumaki*, H. Ichinose, Y. Horiike, The University of Tokyo, Japan

In investigation of the relation between the tip structures and the field electron emission characteristics of the nanotube, we have found that the nanotube tip is plastically deformed during cold emission. Our studies with a high-resolution transmission electron microscope reveal that the deformation occurred at the local domain containing an isolated pentagonal carbon ring in the polyhedral cap and a convexity is formed along the electric field direction. Semi-empirical molecular orbital calculations show that the pentagon and heptagon pair is introduced into the hexagonal network with a pentagonal carbon ring by heterogeneous nucleation mechanism, and the resulting convexity structure is formed at the tip. The electron emission characteristics of the closed nanotube show that the threshold voltage was high for the first run and the current increased quickly. After the second run the emission started at rather lower voltage and increased gradually. Fowler - Nordheim (F-N) plots show that

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the first run does not show a straight line. There is no marked change to gradient the second run. The decreasing of the threshold voltage after the second run is possibly due to the structural change of the nanotube tip. The opened nanotubes also show the notable structural change at the tip. The deformed structure can be explained by introducing sp<sup>3</sup>-like line defects in the hexagonal carbon network. In the opened nanotube, the gradient of the F-N plots is decreasing corresponding to change of structure. The formation of the emission site contributes greatly to the stable and highly efficient electron emission from the nanotubes.

**5:00pm NM+NS-MoA10 A Carbon Nano-Tube Based Electron Gun for Electron Microscopy**, *O. Zik*, El-Mul Technologies Ltd., Israel; *J.G. Leopold*, Dept. of Appl Phys., Rafael Labs, Israel; *D. Rosenblatt*, Rosenblatt Associates  
A novel electron gun geometry is proposed with a carbon nano-tube (CNT) grown in a conducting microfabricated crater separated from a gate by an insulating layer. Electron microscopy preferably utilizes point sources. Field enhancement is responsible for the increased emitted current. The field decreases after a very short range to the free space field of the device and the beam diverges. Because the point source and the electron optics are in practice far from ideal, large angular emission density is required with apertures cropping the diverging beam. Thus, a very small portion of the emitted current is useful. Best performance for point sources is gained when the emitter is a rounded, long and very sharp whisker - a 'point on plane' geometry. Such geometry can be realized with CNT's which have exceptional electron emission properties with very low energy spread. However, due to beam divergence, bare CNT's 'on a plane' are inadequate for Scanning Electron Microscopy (SEM) and to miniature SEM's, termed 'microcolumns', in particular. The crater geometry decreases field enhancement on the tip by an order of magnitude so that increased voltage is needed. Due to the excellent field emission of CNT's this voltage is acceptably low so as to obtain about 500nA while not exceeding the breakdown limit of the insulating layer. Our computer simulations support these results. For such geometry the CNT is immersed in a gun which in itself comprises a lens aligning the beam. Almost all the emitted current can be used in a micro-column. In addition to the low energy spread inherent to CNT's, this electron gun has very high brightness which is an advantage for SEM and lithography applications.

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6-MoA

### Manipulation of Atoms and Molecules

**Moderator:** J.W. Lyding, University of Illinois, Urbana

**2:00pm NS+NANO6-MoA1 Atomic/Molecular Manipulation with the Low Temperature STM**, *G. Meyer*, Paul Drude Institut für Festkörperelektronik, Germany

**INVITED**

The scanning tunneling microscope (STM) opened up new fascinating possibilities for the development of atomic scale technologies. The force between the STM-tip and a single adsorbate can be used to laterally position atoms/molecules with atomic precision (lateral manipulation), which allows the buildup of small nanostructures in an atom by atom way. By exploiting electric field and inelastic tunneling effects individual molecules can be desorbed, transferred to the STM-tip (vertical manipulation), dissociated or even synthesized. For future applications the basic physical mechanisms controlling these techniques have to be investigated first. As model systems we have studied the lateral/vertical manipulation of single metal adatoms (Cu), small molecules (CO, C<sub>60</sub>H<sub>4</sub>I<sub>2</sub>), and large molecules (CuTBPP) adsorbed on metal surfaces (Cu(111), Cu(100), Cu(211)) at low temperatures. Adsorbates can be moved laterally by applying attractive or repulsive forces. This results in a simple pulling or pushing behavior, which can be directly observed in the STM tip height or current signal. In the case of large molecules, which have additional internal degrees of freedom, a more complicated movement behavior is observed. Apart from single adsorbates, also complete chains of molecules can be laterally manipulated and information on the correlated motion of the molecules can be extracted. Finally several examples for the application of the manipulation techniques in the area of surface science will be given. These include: determination of adsorbate binding sites, buildup of small domains of ordered adsorbate phases, atomic scale structuring of the underlying substrate, measurement of weak substrate mediated long range adsorbate interactions, dissociation/synthesis of molecules, manipulation on thin insulating films.

**2:40pm NS+NANO6-MoA3 Fundamental Approaches to Silicon-based Molecular Nanotechnology using the Scanning Tunneling Microscope**, *M.C. Hersam*, *N.P. Guisinger*, *L. Liu*, *J.W. Lyding*, University of Illinois

The importance of molecular nanotechnology has recently been underscored by increased media, public, and government awareness of the subject. This paper outlines an approach for fabricating and characterizing single molecule units on the technologically significant Si(100) surface with the ultra-high vacuum scanning tunneling microscope (UHV-STM). Using feedback controlled lithography (FCL), individual H atoms can be removed from the Si(100)-2x1:H surface. The remaining dangling bond patterns serve as atomically precise templates upon which other materials can spontaneously self-assemble. By utilizing this selective chemistry in situ, several organic molecules (e.g., norbornadiene (NBE), copper phthalocyanine (CuPc), C<sub>60</sub>, etc.) have been isolated. The mechanical, chemical, and electronic properties of these individual adsorbed species can then be immediately detected with the STM. For CuPc, the spatial extent of charge transfer from the substrate to the adsorbate is measured as a function of binding orientation. When the CuPc is reduced with ammonia, single molecule rotation is observed. STM spectroscopic measurements on C<sub>60</sub> reveal intra-molecular variations in the electronic density of states. A fundamental understanding of such phenomena will enable the design of nanoscale devices. For electronic applications, the application of lateral electrical fields to individual molecules is crucial. A fully compatible electrical contacting scheme based on p-n junctions will be presented. Efficient STM potentiometric location of these p-n junctions suggests their additional use as alignment markers. Finally, the robustness of this technique will be outlined. Even following exposure to ambient conditions, the in situ H-passivated Si(100) surface remains atomically pristine. The ability to combine nanolithography with wet chemical processing opens up new opportunities for nanoscale chemical and biological applications.

**3:00pm NS+NANO6-MoA4 STM Atom Manipulation**, *D.M. Eigler*, IBM, Almaden

**INVITED**

PLEASE SEND US AN ABSTRACT. Thank you.

**3:40pm NS+NANO6-MoA6 Scanning Tunneling Spectroscopy via Adsorbate Resonances: Kondo vs. Non-magnetic Systems**, *M. Plihal*, *J.W. Gadzuk*, National Institute of Standards and Technology

Resonance tunneling STM studies of transition metal atoms adsorbed on noble metal surfaces, some combinations forming Kondo systems defined by the presence of a localized magnetic moment on the adsorbate and a spatially extended spin compensation cloud within the substrate, have recently been reported. The intriguing additional influence of artificially-synthesized boundaries such as other atoms, quantum wires, and quantum corrals on the surface has also received much attention. Typically the spectroscopic results are given as spatially-dependent differential conductance versus voltage spectra, usually observed to be close in form to an asymmetric Fano lineshape. This form is characteristic of the interfering "decay" of an initially excited localized state (here an electron or hole state within the STM tip) into a continuum which has been perturbed by a discrete localized state (here the substrate conduction band states coupled to the adsorbate valence electron states). We sketch a detailed theory of the spectral properties of the tunneling for such systems valid for arbitrary intra-adsorbate electron-electron interactions, taking into account nonequilibrium effects when tunneling into the resonance is strong in comparison with direct tip-to-surface tunneling. We present the temperature and tip-position dependence for tunneling through both a Kondo resonance and that of a non-interacting, hence non-magnetic adsorbate. Most of the predicted tunneling characteristics are common to both types of resonances. Issues associated with the spatial dependences, including impurity scattering and mirages within corrals are treated in a natural way using our model.

**4:00pm NS+NANO6-MoA7 Manipulation and Inelastic Tunneling Spectroscopy Measurement of CO on Pd(110) using Low-temperature STM**, *T. Komeda*, *Y.S. Kim*, *M. Kawai*, Riken, Japan

The ability of the scanning tunneling microscopy (STM) to manipulate a single molecule adsorbed on metal surfaces and induce local chemical reaction makes it an idealistic tool to investigate fundamentals of chemical reactions and the catalytic phenomena. In addition, the recent success of inelastic tunneling spectroscopy (IETS) to obtain chemical information by measuring its vibration state makes it more attractive. In this paper, we show the results of the manipulation and the IETS measurement by STM on carbon monoxide (CO) adsorbed on Pd(110) surface at the temperature of 4.8 K. Intriguing 1D chains of CO molecules along Pd(110) row [-110]

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direction) was observed at very low-coverage region which grew to islands with local ordered structure of  $c(2 \times 2)$ . No movement of the molecule was observed during the STM measurement (tunneling current of 1 nA with bias of 100 mV) for the temperature range up to 40 K. The manipulation on isolated CO molecules were done with the following method. The tip was positioned to give 0.1 nA tunneling current with 10 mA substrate bias, then feedback loop was turned off. The bias between the tip and the sample was varied between -350 mV and +350 mV with the frequency of 3 Hz which gives the maximum current less than 5 nA which is quite moderate for metal observation. However, this method caused the CO molecule hopped single site along  $[-110]$  direction. The driving force for the CO movement can be understood by the interaction between the electric field formed by the tip-and-sample and the dipole-moment of CO, which is considered to be very controllable method without touching between the tip and the sample. Also inelastic tunneling spectroscopy (IETS) has been carried out on an isolated CO molecule and on the CO chains. The I-V curve showed characteristic features around 250 mV.

4:20pm **NS+NANO6-MoA8 Benzene and Its Derivatives on Transition Metal (111) Surfaces**, *Y.-J. Song, J.-Y. Park, U.D. Ham, H.J. Kim, Y. Kuk*, Seoul National University, Korea

Benzene and its derivatives such as bromobenzene, iodobenzene, and xylene, adsorbed on transition metal surfaces (Cu and Pd) have been investigated with an ultrahigh vacuum low temperature scanning tunneling microscope (STM). At room temperature, these molecules are highly mobile so that it is difficult to study low coverage behaviors. On the other hand, they form close-packed ordered structure with three different domains at saturation coverage, as observed with STM. At low temperature, the mobility of molecules is reduced that single molecules can be studied. With high-resolution imaging capability of our STM, internal structures of molecules are revealed. Therefore, different isomers can be distinguished. Furthermore, manipulation of these molecules is performed with the STM tip. Possible mechanisms of different manipulation modes will also be discussed. The possibility of STM-induced molecular reaction will also be presented.

4:40pm **NS+NANO6-MoA9 Artificial Surface Structure on H/Si(100)4x3-In Surface Using STM Tip**, *O. Kubo, T. Harada, T. Kobayashi, N. Yamaoka, M. Katayama, K. Oura*, Osaka University, Japan

Scanning tunneling microscopy (STM) recently has considerable attention as an apparatus not only for observation of surface structures but also for manipulation of surface atoms. Since Lyding et al. @footnote 1@ reported about hydrogen (H) extraction from Si(100)2x1-H (monohydride) surface using STM tip, H extraction has been investigated both experimentally and theoretically. When superstructures on Si surface induced by metal (Ag, In, etc.) adsorption are exposed to atomic H, peculiar H terminated surface phases are induced. For instance, Si(100)4x3-In surface phase is produced by the deposition of 0.5 ML In onto Si(100)2x1 surface at 400°C. When this surface is exposed to atomic H at 300°C, In atoms form clusters, and the reconstructed Si layer which have been formed under In layer is frozen by H termination of Si dangling bonds. @footnote 2@ This H terminated Si layer has monohydride dimer rows whose interval is  $4a$  ( $a = 3.84 \text{ \AA}$ ), and among the dimer rows, it has dihydride Si atoms (what we call Si(100)4x1-H). In this study, we have performed H extraction from the Si(100)4x1-H surface using STM tip. The H extraction could be performed by applying about 5 V of sample voltage and about 0.2 nA of tunneling current, which were almost the same condition for the H extraction from Si(100)2x1-H. After H extraction, the  $4a$  periodicity of dimer rows was preserved and the H extracted area was observed higher than a surrounding H terminated surface as well as the extracted area on Si(100)2x1-H surface. This result means that peculiar Si surface structure can be formed artificially. The dimer rows consisted of buckled dimers in spite of room temperature observation contrary to the case on Si(100)2x1-H surface. It is most likely that these dimers were induced by surface stress as same as seen on step edges. @FootnoteText@ @footnote 1@ J.W. Lyding et al., J. Vac. Sci. Technol. B 12 (1994) 3735 @footnote 2@ J.T. Ryu et al., J. Vac. Sci. Technol. B 17 (1999) 983.

5:00pm **NS+NANO6-MoA10 Adsorption and Manipulation of Endohedral Fullerenes on Silicon Surfaces**, *M.J. Butcher, P.H. Beton*, University of Nottingham, UK; *P. Kuran, L. Dunsch*, IFW Dresden, Germany

The adsorption and manipulation of an endohedral fullerene, LaC82 has been investigated using a scanning tunneling microscope (STM) operating at room temperature in ultra-high vacuum. These molecules are adsorbed on top of both rows and troughs on the Si(100)-2x1 surface, and like C60 may be manipulated in a repulsive mode using the STM tip. However for

LaC82 a much stronger interaction between adsorbate and tip is required to promote manipulation resulting in a lower voltage threshold for this process. Adsorption of this species on a less reactive substrate (for example, Ag/Si(111)) results in the formation of close packed hexagonal islands oriented, primarily, parallel to the principal axes of the Si surface. This result confirms an expected commensurability of the intermolecular spacing,  $d$ , and the surface lattice constant, namely  $d \sim 3a$ , indicating that it should be possible to form close packed rows of LaC82 along the troughs of Si(100)-2x1. The formation of pairs and other simple close packed assemblies of molecules has been demonstrated. A comparison of the response to manipulation of a range of fullerenes and heterofullerenes will also be presented and discussed in terms of the effects of size and chemistry of the adsorbed molecules.

## Electronics

### Room 312 - Session EL+NS-TuM

#### Molecular Electronics

**Moderator:** Y. Liang, Pacific Northwest National Laboratory

8:20am **EL+NS-TuM1 Molecules, Hierarchical Assembly and Architecture of Moletronic Computers, W.L. Warren**, Defense Advanced Research Projects Agency **INVITED**

While the rapid miniaturization of electronics has driven much of the development of computation and information systems, it is anticipated that future computational demands for performing calculations orders of magnitude faster with less power. To create discovery beyond silicon, the DARPA Moletronic program is proposing that both computational functionality and input/output architecture can be designed in molecular systems. These systems will have high device density (scalable to 10<sup>12</sup> device/cm<sup>2</sup>) and be amenable to greatly simplified manufacturing fabrication using hierarchical-assembly. It is anticipated that architecture, a top-down approach will play an immense role in steering discovery. Moletronic is focused on developing technologies based on the self-assembly of molecular components to act as devices, switches, and interconnects. The program does not clone contemporary electronic devices with molecular scale components, but rather create functionality that exploits what molecules are good at (self-assembly, thermodynamic equilibrium, and charge storage media.) The trick for molecular computers will be to cope with defects and to realize hierarchical processes to manufacture the hardware. Hierarchical assembly is envisaged as a set of processes that will first assemble individual devices, then create functional blocks (circuits) from those devices and finally assemble systems from the blocks. It is a bottom-up manufacturing process whereas current microelectronics fabrication is based on top-down manufacturing using lithography. The talk will discuss three over-riding themes of the Moletronic program: (1) molecular devices, (2) hierarchical assembly, and (3) architecture including defect tolerance and algorithm development to find the defects. @FootnoteText@ @footnote 1@ The contributions of the contractors of the DARPA Moletronic program and fellow program managers, Bruce Gnade, Christie Marrian, and John Pazik are gratefully acknowledged.

9:00am **EL+NS-TuM3 Molecular Wires, Switches and Memories, J. Chen**, Yale University **INVITED**

Molecular electronics has recently attracted much attention because of its potential application in future computational systems. Molecular wires and related strategically engineered structures (e. g., molecular switches and memories) offer an increased versatility in device design with reduced elements. Nanoscale molecular devices utilizing active self-assembled monolayer (SAM) as the active component will be reported. We will present results on: (1). measurements of through-bond conductance of symmetric metal/conjugated SAM/metal junctions where non-Ohmic thermionic emission was observed to be the dominant process, with isocyanide-Pd contacts showing the lowest thermionic barrier of 0.22 eV; (2). first realization of robust and large reversible switching behavior in an electronic device that utilizes molecules containing a nitroamine redox center (2'-amino-4,4'-di(ethynylphenyl)-5'-nitro-1-benzenethiolate) exhibiting negative differential resistance (NDR) and an on-off peak-to-valley ratio (PVR) exceeding 1000:1; while molecules containing a nitro-only redox center (4,4'-di(ethynylphenyl)-2'-nitro-1-benzenethiolate) exhibiting room temperature NDR; (3) demonstration of a two-terminal electronically programmable and erasable molecular memory cell with bit retention time greater than 10 minutes.

9:40am **EL+NS-TuM5 Dissimilar Metal Electrodes with Nanometer Interelectrode Distance: Fabrication and Application to Characterizing Self-Assembled Molecular Electronic Devices, M.A. Guillorn, I. Lee**, University of Tennessee; *D.W. Carr, R. Tiberio*, Cornell Nanofabrication Facility; *E. Greenbaum, J. Lee*, Oak Ridge National Laboratory; *M.L. Simpson*, Oak Ridge National Laboratory and The University of Tennessee

In order to advance the state of molecular-scale electronics research, electrode structures capable of realizing metal/molecular monolayer/metal heterojunctions have been fabricated using a variety of novel techniques that allow direct electrical contact with single molecules and small groups of molecules. By using Au as the electrode material, thiol-based self-assembly techniques have been successfully applied to deposit organic-synthetic molecules between these electrodes, thereby permitting their

electrical characterization. This is possible due to the symmetry of these molecules. Unfortunately these techniques do not lend themselves to measuring the electrical properties of asymmetric molecules. An example of this type of molecule is the Photosystem I (PSI) reaction center which is of demonstrated interest to molecular-scale electronics research. Self-assembly techniques have been developed to preferentially orient the PSI with respect to a Au substrate, however, the polar nature of this molecule inhibits the use of this technique for the formation of Au/PSI/Au heterostructures. In this paper we will discuss a flexible and reproducible process for fabricating dissimilar metal electrodes with nanometer interelectrode distance (DiMEND) using high-resolution electron beam lithography and liftoff pattern transfer. This process is capable of realizing electrode pairs with a minimum interelectrode distance of less than 6 nm. This technique provides a reproducible method for creating lateral structures well suited for the electrical characterization of asymmetric molecules for molecular-scale electronics applications. Applications of this technology to characterizing self-assembled molecular electronic devices will be presented.

10:00am **EL+NS-TuM6 Characterization of Gold/Pentacene and Pentacene/Gold Interfaces, Q.T. Le, S. Zorba, E.F. Forsythe, L. Yan, N.J. Watkins, Y. Gao**, University of Rochester; *S.F. Nelson*, Colby College; *C.S. Kuo, T.N. Jackson*, The Pennsylvania State University

Among the conjugated oligomers used as active materials in the fabrication of organic thin-film transistors (OTFT), pentacene is one of the most promising materials due to its high field-effect mobility. The performance of pentacene based OTFTs is now comparable to that obtained from hydrogenated amorphous silicon TFTs. The interface formed between the organic semiconductor and the conducting layer strongly influences the current-voltage characteristics of OTFTs. We report the interface formation between gold (Au) and pentacene using x-ray and ultraviolet photoelectron spectroscopy (XPS and UPS). The morphology of the pentacene film deposited on the substrate was measured using atomic force microscopy, showing a pentacene layer with small grain size. XPS results indicate that Au does not chemically react with pentacene if Au is deposited onto pentacene or if pentacene is deposited onto Au. For Au deposited on pentacene the UPS results show that there is an initial binding energy shift of both the highest occupied molecular orbital of pentacene and the vacuum level attributed to the formation of a common fermi level for the Au and pentacene as the Au is deposited. The initial increase is then followed by a decrease in binding energy shifts that is attributed to a reduction of the polarization energy caused by the formation of a Au overlayer on the surface of the pentacene. For pentacene deposited onto Au the UPS results show that there is only a shift of the vacuum level to lower binding energy shift of the vacuum level to lower binding energy.

10:20am **EL+NS-TuM7 Current-Induced Organic Molecule-Silicon Bond Breaking: Consequences for Molecular Devices, S.N. Patitsas, G.P. Lopinski, O. Hul'ko, D.J. Moffatt**, National Research Council of Canada; *R.A. Wolkow*, National Research Council of Canada, Canada

The current carrying capacity of individual organic molecules covalently bound to silicon has been studied. Adsorbates comprised exclusively of saturated C-C bonds were found to be entirely stable whereas adsorbates containing pi-bonds could be controllably dislodged under modest conditions. The pi-bonds act as a chromophore, taking energy from a scattered electron, energy that can be selectively channeled into Si-C bond breaking. The class of adsorbates that are dislodged is closely related to unsaturated molecules widely investigated for enhanced molecular wire character. It is predicted that molecular devices containing such molecules will fail when operated. Measures to avoid current-induced bond breaking are described. New processes based upon the controlled bond breaking phenomenon are suggested.

10:40am **EL+NS-TuM8 Molecular Arrays and Individual Dopants Studied by Ultrahigh Vacuum Scanning Tunneling Microscopy, L. Liu, J. Yu, N. Viernes, M.C. Hersam, N.P. Guisinger, J.W. Lyding**, University of Illinois

One aspect of molecular electronics is the fabrication of atomically precise molecular arrays on silicon substrates. Feedback controlled lithography (FCL) is used in conjunction with UHV STM to create atomically precise templates for molecular adsorption. Cobalt phthalocyanine (CoPc) molecules have been sublimed onto these templates and subsequently imaged in the STM. The difference between filled state and empty state images is explained. Current imaging tunneling spectroscopy (CITS) of CoPc is also performed and discussed. CoPc Molecular symmetry as well as the feature associated with the central Co atom are studied in all STM images. CITS has also been used to identify and study individual dopants on or near

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Si(100)2X1:H surfaces. For high quality UHV H-passivated Si(100) the Fermi level is unpinned at the surface, thus enabling the observation of subtle features including subsurface dopants. For example, boron atoms appear as slight hillocks in filled state images and as centro-symmetric depressions in empty states. Full CITS data sets and their analysis and interpretation will be presented. The same techniques will be used to present results for isolated Si dangling bonds created by FCL.

11:00am **EL+NS-TuM9 Direct Measurements of Electrical Transport through DNA Molecules, D. Porath**, Tel Aviv University, Israel & Delft Univ. of Tech., The Netherlands, Israel; *A. Bezryadin*, Harvard University; *S. de Vries*, *C. Dekker*, Delft University of Technology, The Netherlands **INVITED** Is DNA a conductor for electrical charges? This question has been heavily debated over the past few years, spurred by optical experiments that measured fluorescence quenching for DNA molecules and recent electrical measurements. Here we present measurements of electrical transport through short and well-defined DNA molecules that show large-gap semiconducting behavior. A 10.4 nm long, double-stranded poly(G)-poly(C) DNA molecule is connected to two metal nanoelectrodes that are separated by 8 nm. Nonlinear current-voltage curves with a voltage gap at low bias are observed at ambient conditions as well as in vacuum and at cryogenic temperatures. The voltage dependence of the differential conductance exhibits a peak structure, suggesting that transport of charge carriers is mediated by the molecular energy bands of DNA. Recent STM measurement on similar DNA molecules will be shown as well.

11:40am **EL+NS-TuM11 Whole-Cell Bio-computing in a Microelectronic Format, M.L. Simpson**, Oak Ridge National Laboratory and The University of Tennessee; *G.S. Saylor*, *J. Fleming*, *B. Applegate*, *S. Ripp*, *D. Nivens*, University of Tennessee Center for Environmental Biotechnology  
Even simple cells perform tremendously complex information processing operations involving memory (genes), sensing and feedback (promoters, regulatory proteins), and in some cases, interconnectivity (quorum sensing). For example, *Escherichia coli*, with a 4.6 million base-pair chromosome, has the equivalent of a 9.2-megabit memory to code for as many as 4,300 different polypeptides under the inducible control of perhaps several hundred different promoters. Yet, all of this functionality is contained in an area of approximately 1 square micron. Conversely, current predictions of the future of silicon integrated circuits indicate that 1 square micron of silicon will contain only a 245 bit memory or 1.66 simple logic gates by 2014. Obviously, silicon technology will not approach bacterial-scale integration within the foreseeable future. Furthermore, microorganisms have some qualities that are quite desirable for information processing devices and systems. Bacterial cells are relatively rugged "devices" that subsist in even extreme environments. Also, they are fairly easy to manipulate genetically, and have a diverse set of gene regulation systems. Furthermore, cells easily can be incorporated into a 3-dimensional structure instead of the planar structure of integrated circuits. And finally, cells self-assemble and self-replicate, and therefore are easy to manufacture. We will present our work to incorporate the information processing capabilities of living cells into a microelectronic format. This will include our work on the bioluminescent bioreporter integrated circuit (BBIC) for sensing applications, as well as our recent work to engineer more complex information processing and communication functions into whole cells deployed on integrated circuits.

## Magnetic Interfaces and Nanostructures

### Room 206 - Session MI+NS+NANO 6-TuM

#### Magnetic Imaging I

Moderator: M. Miller, Naval Research Laboratory

8:20am **MI+NS+NANO 6-TuM1 Scanning Electron Microscopy with Polarization Analysis (SEMPA) Imaging of Surface and Thin Film Magnetic Microstructure, J. Unguris**, National Institute of Standards and Technology **INVITED**

Scanning Electron Microscopy with Polarization Analysis (SEMPA) provides a direct image of a sample's magnetization by measuring the spin polarization of secondary electrons emitted in a scanning electron microscope (SEM). SEMPA therefore generates a high resolution picture of the direction and relative magnitude of the magnetization, in the same way that an SEM images topography by measuring the secondary electron intensity. With submonolayer magnetic sensitivity and probe sizes as small as 10 nm, SEMPA is sensitive to extremely small amounts of magnetic material. In particular, SEMPA's surface sensitivity makes it especially well

suitable for the direct, quantitative mapping of the magnetization direction in thin films and at the surface of magnetic materials. Comparisons between magnetic and physical structure in these systems are further facilitated by the natural ability of SEMPA to separate the magnetic and topographic contrast. When combined with other compatible surface analytical techniques such as Auger, RHEED and STM, SEMPA can also provide information about the relationship between the magnetic structure, the chemical structure, and the atomic scale order. SEMPA can also be used for real time, in situ imaging of magnetic structure during thin film growth and processing. This talk will describe the SEMPA technique by presenting examples of measurement applications from thin film and multilayer magnetism, surface magnetism of ferromagnets and antiferromagnets, and depth profiling of magnetic structures in multilayers. These measurements have provided a better understanding of thin film domain structures, spin reorientation transitions, interlayer exchange coupling, magnetic ordering in antiferromagnetic films, and the relationship between magnetic domain structure and magnetoresistance in multilayers.

9:00am **MI+NS+NANO 6-TuM3 'Magnetic-Laboratory' on an AFM Tip, B.K. Chong<sup>1</sup>, H.P. Zhou**, University of Glasgow, UK, United Kingdom; *G. Mills*, *L. Donaldson*, *J.M.R. Weaver*, University of Glasgow, UK

We present novel magnetic nanosensors based on the functionalisation of an AFM probe for use in measuring, imaging and manipulating magnetic specimens. The probes are fabricated using bulk silicon micromachining and electron-beam nanolithography (EBL). The use of conventional lithographic techniques and a microfabricated substrate allows the batch production of a large number of similarly functionalised probes without the need for individual processing of single probes and hence gives very good reproducibility. @footnote 1@ We have demonstrated two classes of magnetic probe. The eMFM probe is one in which the permanent magnetic coating used in conventional MFM tips is replaced by a small electro-magnetic coil to form a magnetic-sensitive AFM tip. Initial results indicate controllability of coil size (spatial resolution) to 1/4  $\mu$ m diameter, demonstrated capability of magnetic imaging and possible application in local magnetic modification. The Hall bar magnetometer for SHPM, is also integrated with a tip and cantilever. This involved the development of a new fabrication technique in which the resist was supported by a lattice of sacrificial structures which spanned the spaces between probes. This allowed the use of low melting point or chemically reactive materials as the sensor. The combination of eMFM and Hall bar magnetometer forms a novel type of magnetic sensor-actuator probe, a 'Magnetic-Lab' on a tip. Such a probe will allow the magnetic imaging of a specimen without significant distortion due to stray fields from the probe using Hall probe magnetometry as well as the deliberate modification of its magnetic state using the coil. @FootnoteText@ @footnote 1@ H.Zhou, G.M. Mills, B.K.Chong, L. Donaldson & J.M.R. Weaver, 'Recent Progress in the Functionalisation of AFM Probes using Electron-Beam Nanolithography', J. Vac. Sci. Technol. A17(1) 2233-9(1999).

9:20am **MI+NS+NANO 6-TuM4 High Anisotropy, High Gradient Magnetic Tips For Magnetic Resonance Force Microscopy, H.J. Mamin, B.C. Stipe, C.S. Yannoni, D. Rugar**, IBM Almaden Research Center; *T.D. Stowe*, *T.W. Kenny*, Stanford University; *D. Streblichenko*, *M.R. Scheinfein*, Arizona State University

In magnetic resonance force microscopy (MRFM), imaging is performed by detecting forces on a magnetic tip, but arising from only those spins within a thin resonant slice. To achieve the ultimate goal of single-spin detection, the tip must produce sufficient field gradients, at least 10 G/nm. At the same time, thermal fluctuations in the tip moment must be sufficiently small that the tip does not perturb the spin under study. These requirements have driven us to the use of tips based on high anisotropy, rare-earth bulk magnets. Small particles of NdFeB, PrFeB, and SmCo have been attached to cantilevers and oriented in an external magnetic field. The particles were then sculpted to the desired sub-micron size and shape with a focused ion beam. The magnetic properties have been characterized through cantilever-based magnetometry as well as electron holography. By measuring dissipation and applying the fluctuation-dissipation theorem, we have set upper limits on the low frequency fluctuations in the tip moment. Using a tip optimized for a 1 nm slice thickness, we have detected MRFM signals from the order of 100 net spins. This work is supported, in part, by the Office of Naval Research.

<sup>1</sup> Falicov Student Award Finalist

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9:40am **MI+NS+NANO 6-TuM5 Quantitative Magnetic Force Microscopy and Exchange Force Microscopy: New Tools for Magnetic Imaging**, *H.J. Hug, P.J.A. van Schendel, S. Martin, R. Hoffmann, P. Kappenberger, M.A. Lantz, H.-J. Guentherodt*, University of Basel, Switzerland **INVITED**

Magnetic Force Microscopy has become a well established technique for studying the topography and the micro-magnetic structure of various samples with a high lateral resolution. Among these are ferromagnetic and superconducting materials, and magnetic recording read/write-heads. Recently there has been growing interest in the quantitative analysis of measurement data obtained using a magnetic force microscope (MFM). Recent tip calibration procedures allow quantitative stray field measurements, the determination of the stray field distribution of the tip, and its stray field sensitivity. The best lateral resolution currently is around 30nm. However, the combination of ultra-sharp SFM-tips coated with ultra-thin magnetic layers and improved instrumental sensitivity may allow a lateral resolution around 10nm. A higher lateral resolution may be reached by the measurement of exchange forces. The principles of this new technique and first experiments will be discussed. @FootnoteText@ @footnote 1@ P.J.A. van Schendel et al., J. Appl. Phys. 88, 435-445 (2000)

10:20am **MI+NS+NANO 6-TuM7 Magnetic Force Microscopy of Coupled and Decoupled Micrometer Scale Permalloy Structures**, *U. Memmert, A.N. Müller, U. Hartmann*, University of Saarbrücken, Germany; *J. Jorzick, C. Krämer, S.O. Demokritov, B. Hillebrands*, University of Kaiserslautern, Germany; *E. Sondergard, M. Bailleul, C. Fermon*, CEA Saclay, France

The magnetic structure and the magnetization reversal process of arrays of micron size rectangular magnetic permalloy islands were investigated by magnetic force microscopy in external magnetic fields. The samples were prepared by e-beam lithography and ion etching of UHV deposited 35 nm thick permalloy films. Islands of 1  $\mu\text{m}$  x 1.75  $\mu\text{m}$  dimensions were investigated with inter-island spacings between 0.1  $\mu\text{m}$  and 1  $\mu\text{m}$ . The data show a transition from not interacting islands for large inter-island spacing to interacting islands for the smallest spacing. For not interacting islands flux-closure structures were found to be present without external fields. Either a simple Landau structure with one single cross-tie within in the 180° wall or diamond structures consisting of 90° walls were found. For interacting islands the individual structures were often found in a magnetized state even in zero field. Edge domains were present on both short edges of each structure. A demagnetization of the interacting islands in an ac-magnetic field along the longer axis of the islands leaves the individual structures in a magnetized state with edge domains. Within the rows along the long island axis all structures are magnetized in the same direction. The rows show a row by row alternating magnetization resulting in a net zero magnetization for the entire sample. A demagnetization in an ac-field perpendicular to the long axis leaves each individual structure in a individually demagnetized state. Magnetization reversal for external fields along the long island axis takes place by simultaneous switching of structures being located together in the same row within the pattern.

10:40am **MI+NS+NANO 6-TuM8 Correlation of Structural and Magnetic Properties of Fe/Cr(001) Studied by Combined SP-STM and MFM**, *M. Kleiber, R. Ravlic, M. Bode, R. Wiesendanger*, University of Hamburg, Germany

The magnetic structure of ultrathin Fe films on the (001)-surface of a chromium single crystal is the result of the competition between the antiferromagnetic coupling to the alternately magnetized Cr(001)-terraces and the ferromagnetic coupling in the iron layer. It is expected that for thin Fe-films the antiferromagnetic coupling between the Cr-substrate and the Fe-overlayer dominates the surface domain structure while Fe-exhibits a single-domain state for thicker films. By combining STM, spin-polarized STS and UHV-MFM we have correlated the structural and magnetic properties of the Fe/Cr(001) system. These microscopic techniques reveal that the domain structure depends on the local step density. On areas with high step density no domains are found which is expected as the terraces are too small to induce domains in the iron film. In contrast a low step density leads to a domain structure of the Fe-film which is directly linked to the step structure of the underlying Cr-substrate.

11:00am **MI+NS+NANO 6-TuM9 Flux Lattice Imaging of a Patterned Nb Film with a Cryogenic Magnetic Force Microscope**, *M. Roseman, P. Grutter*, McGill University, Canada; *V. Metlushko*, Argonne National Laboratory

Using our cryogenic magnetic force microscope, we have investigated a superconducting Nb thin film, 100 nm in thickness with  $T_c \sim 6.6$  K. The film is patterned with a square array (1  $\mu\text{m}$  by 1  $\mu\text{m}$ ) of antidots, which

serve as artificial pinning centers for magnetic flux. We have observed the flux lattice as a function of temperature (5.5 K - 6.6 K) and applied magnetic field, for field strengths up to 62.1 G, the third matching field (a matching field is one where the flux lattice spacing is commensurate with the antidot array). Evidence of flux dragging by the tip reveals information about both tip-vortex and vortex-vortex interactions, and provides an indication of localized sample pinning potentials. Force distance curves acquired at temperatures near  $T_c$  clearly demonstrate an observable Meissner force between tip and sample, and allow for an estimation of the value of the temperature dependent London penetration depth,  $\lambda_L(T)$ .

11:20am **MI+NS+NANO 6-TuM10 Magnetic Field Measurements of Current-Carrying Devices by Force Sensitive Magnetic Force Microscopy with Potential Correction**, *R.A. Alvarez, S.V. Kalinin, D.A. Bonnell*, University of Pennsylvania

Magnetic force microscopy (MFM) is a well-known technique based on the detection of the dynamic response of a mechanically driven cantilever to a magnetic field. MFM image contrast of non-conductive or biased surfaces includes contributions of electrostatic forces that can in some circumstances dominate the total force gradient. Since current-carrying devices, e.g. lines or circles are recognized as convenient calibration standards to determine first and second order magnetic moments of the MFM probes, this ambiguity is not inconsequential. An approach to imaging is proposed that combines surface potential nulling measurements with magnetic force microscopy to eliminate the electrostatic forces. Unlike conventional MFM, this technique measures force rather than force gradient. The distance, line bias and modulation frequency dependence of cantilever response was found to be in excellent agreement with magnetostatic calculations. Based on these observations, a new type of MFM on current carrying devices is proposed. In this technique, the device is ac biased at the off-resonant frequency and the current induced magnetic field results in cantilever deflection. At the same time, ac voltage bias at the resonant frequency is applied to the tip and conventional SSPM feedback is used to match tip and surface potentials. This technique allows simultaneous collection of surface potential and magnetic force images. To the best of our knowledge, this is the first example of an SPM technique that utilizes simultaneous active and passive modulation of the tip and allows simultaneous measurement of magnetic and electrostatic forces.

11:40am **MI+NS+NANO 6-TuM11 Evaluation of MFM for Probing Electromigration Processes**, *R. Yongsunthon, J. McCoy, E.D. Williams*, University of Maryland

The study of electromigration in metals requires correlation of current densities with the evolution of defects in current-carrying lines. In principle, magnetic force microscopy (MFM) is an appropriate tool for this purpose. Most use of MFM has concentrated upon determining magnetic polarity across magnetic domain boundaries, rather than quantification of magnetic field variation. Such quantification is non-trivial, because the extended nature of the tip-sample interaction involves complicated factors such as coupling of the system geometry. To explore the MFM capability to yield reliable analysis, we are evaluating the MFM instrument response for known structures, such as lines containing defects of simple geometry. The instrumental response function is defined by tip parameters, such as tip magnetization and shape, which make predicting the response function impractical. However, it is possible to make meaningful relative quantification and calibration, by comparison with response from structures where the behavior is well understood. To analyze the data from such known calibration samples, the fields around the lines are numerically calculated and compared with deconvolution of the measured signal. Preliminary results suggest that meaningful relative quantification of the signal can be achieved to within 20% and that current variations can be detected to at least 10%. Continuing work to relate this to the limiting levels of current crowding that will be detectable is underway. (Work supported by NSF-MRSEC, grant# DMR 96-3252.)

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## Nanotubes - Science and Applications

### Room 309 - Session NM+NS-TuM

#### Carbon Nanotubes: Synthesis

**Moderator:** D. Herr, Semiconductor Research Corporation

8:20am **NM+NS-TuM1 Orientated Nanotube Growth with Cobalt Catalyst**, **S. Shah**, University of Illinois at Urbana-Champaign; **L. Rotkina, H. Choi**, Beckman Institute for Advanced Science and Technology; **J.W. Lyding**, University of Illinois at Urbana-Champaign

Growth of carbon nanotubes (CNTs) is an important area of research in the area of molecular nanotechnology. The full potential of these devices as a building block for nanometer scale structures has not been fully realized and therefore new processing techniques and observations could have impact on the field. This paper will focus on the main aspects of our cobalt catalyst based growth process as well as our observations of the resulting carbon nanotubes. We will then suggest some applications of this technique for the development in the area of nanometer scale research. With the purpose of observing the growth behavior on a Si(100) surface, we have grown carbon nanotubes by means of chemical vapor deposition (CVD). Two distinct types of growth orientations were observed on the surface using a cobalt based catalyst and methane gas as a source of carbon. The first is a series of catalyst islands that have nanotubes growing randomly from them. The patterning of these tubes seems to "bridge" islands of catalyst together. The second growth mechanism is cobalt silicide based, where orientation of both the silicide and carbon nanotubes, appears highly oriented. Growth of oriented CNTs on silicon is particularly important to integrate CNTs with silicon devices. From the observations stated above, we hope to demonstrate a processing technique in which we can grow and characterize nanotubes grown in a highly oriented manner. Finally, our main objective will be to integrate our fabrication techniques to form the basis for the growth of nanotubes of nanometer scale devices.

8:40am **NM+NS-TuM2 Carbon Nanotube Growth on Nanoparticle Catalyst Patterns by Chemical Vapor Deposition**, **J.W. Ward, P.M. Ajayan, G. Ramanath**, Rensselaer Polytechnic Institute; **L. Kish, R. Vajtar**, Uppsala University, Sweden

Growing nanotubes on catalyst template patterns on flat substrates by chemical vapor deposition (CVD) is a promising approach for creating nano- and meso-scale architectures for a variety of applications such as micro- and bio- electronics devices, and skeletal reinforcements for layered composites. Here, we report the unique morphology and junction-formation potential of CVD-grown carbon nanotubes on catalyst patterns fabricated by a nanoparticle writer. Patterns of Ni, Co, and Ni-Co alloys with different average particle sizes and spatial distributions were prepared on Si substrates and exposed to methane at 1000 °C. Our results show that multiwalled carbon nanotubes grow on nanoparticles. There is a close correlation between the nanotube diameter and the catalyst particle size. The nanotubes exhibit a large number of bends and turns. In several cases, the nanotubes grow from one particle and terminate at another, thereby connecting two nanoparticles lying on the substrate. Based upon our results, we propose a phenomenological explanation for nanotube-bridging. Controlling the formation of such nanobridges could provide a basis for simultaneous selection of both nucleation and termination sites, which is an important requirement for realizing nanotube-based network architectures.

9:00am **NM+NS-TuM3 Carbon Nanotube Catalyst Optimization Using Combinatorial Methods**, **A.M. Cassell, M. Meyyappan, S. Verma, J. Han**, NASA Ames Research Center

Libraries of liquid-phase catalyst precursor solutions were printed onto various substrates and evaluated for their effectiveness in catalyzing the growth of carbon nanotubes by chemical vapor deposition (CVD) of ethylene. The catalyst precursors were composed of inorganic salt solutions of Al, Si, Fe, Co, Ni, and a removable tri-block copolymer structure-directing agent. Scanning electron microscopy (SEM) was used to rapidly screen the catalyst libraries for activity. The optimized catalysts were then employed in the growth of aligned multi-walled carbon nanotube arrays. Successful implementation of combinatorial optimization methods in the development of high yielding carbon nanotube catalysts is demonstrated, as well as useful techniques for obtaining nanotube films of various configurations.

9:20am **NM+NS-TuM4 Effects Gas Adsorption and Collisions on the Physical Properties of Single-Walled Carbon Nanotubes**@footnote 1@, **P. Eklund**, Pennsylvania State University **INVITED**

A single-walled carbon nanotube (SWNT) can be thought of as a graphene sheet rolled into a seamless cylinder. They are usually found in bundles containing several hundred tubes, and these bundles present an ideal microporous medium accessible to small gas molecules and ions. For this reason SWNTs are expected to be a sensitive chemical sensor, as has been reported recently. Bundles of SWNTs undergo charge transfer reactions similar to graphite, and this charge transfer when large enough can be monitored by Raman scattering. Even when very weak charge transfer, or just gas collisions with tube walls occurs, electrical transport (resistivity and thermopower) is found to be a very sensitive probe of the perturbation on the nanotube. We first review the effects on the physical properties with reagents that produce large amounts of charge transfer (e.g., alkali metals and iodine), then move to results on gases suspected of weak charge transfer reactions with SWNT (i.e., NH<sub>3</sub>) and then finally to weaker perturbations caused by physisorbed gases, such as CO. Finally, the surprisingly strong effects on the resistivity and thermoelectric power from collisions of inert gas molecules (e.g., He) with the SWNT walls will be presented. @FootnoteText@@footnote 1@Work supported by the ONR and NSF.

10:00am **NM+NS-TuM6 Time-Resolved Diagnostic Investigations of Carbon Nanotube Synthesis**, **D.B. Geohegan, A.A. Puzos, X. Fan, M.A. Guillorn, D.C. Joy, M.L. Simpson, V.I. Merkulov, S.J. Pennycook**, Oak Ridge National Laboratory

Time-resolved imaging and spectroscopy measurements are applied in conjunction with ex situ TEM and FESEM investigations to understand the growth rate and mechanisms of carbon nanotube growth during laser vaporization synthesis inside a hot oven. Condensation times of atomic and molecular species in the plume are estimated using population densities of ground state species as measured by laser-induced fluorescence. Rayleigh scattering, induced blackbody emission, and real-time video techniques are used to measure the dynamics and annealing time of these condensed aggregates of clusters, nanoparticles, and nanotubes as they propagate inside the oven. By varying the growth time with these diagnostics, we have explored the rate and mechanism of single-wall carbon nanotube growth by laser vaporization through the use of high-resolution transmission electron microscopy and field emission scanning electron microscopy of deposits collected for various growth times. Z-contrast STEM combined with EELS is used to investigate the effects of size and composition of metal catalyst nanoparticles through the ability to compositionally profile individual catalyst nanoparticles. FESEM imaging in bright and backscatter modes is also used to provide a three-dimensional perspective of nanotube growth. We conclude that nanotube growth during the laser vaporization process occurs over seconds of time by the condensed phase conversion of nanoparticle feedstock by the metal catalyst nanoparticles. Ex situ annealing experiments of incompletely-converted, short nanotube 'seeds' are described which show that nanotube growth can occur outside the hot oven, supporting the condensed phase conversion growth mechanism. The possibility of varying the growth conditions to enable economically viable scale-up of nanotubes by this technique will be discussed. Research sponsored by the Laboratory Directed Research and Development program at Oak Ridge National Laboratory.

10:20am **NM+NS-TuM7 Growth of Well-Aligned Carbon Nanotubes on Nickel by Hot-Filament-Assisted DC Plasma Chemical Vapor Deposition in a CH@sub 4@/H@sub 2@ Plasma**, **Y. Hayashi, T. Negishi, S. Nishino**, Kyoto Institute of Technology, Japan

Carbon nanotubes are expected for the electron emitters of a field emitter display (FED). In order to realize the FED, a growth method of carbon nanotubes perpendicularly well-aligned on a large-area substrate has to be developed. Recently it was reported that such aligned carbon nanotubes were grown on nickel by plasma-enhanced hot filament chemical vapor deposition (CVD) and microwave plasma CVD. We have succeeded to grow well-aligned carbon nanotubes in the area of 4 cm in diameter by hot-filament-assisted DC plasma (HF-DCP) CVD in the gas of CH@sub 4@/H@sub 2@. The growth method and conditions were as follows. DC voltage of -250V was applied to substrates relative to hot filaments. A luminous region was observed just above the substrates. By the optical emission spectroscopy, it was confirmed that the luminescence was derived from excited hydrogen and hydrocarbon radicals. Therefore the process is called HF-DCP CVD. Nickel substrates were heated by the filaments around 600 °C. The substrates were pretreated in pure hydrogen

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plasma for 15 min before the growth of carbon nanotubes in 20% CH<sub>4</sub>/H<sub>2</sub> for 15 min. Well-aligned carbon nanotubes about 100 nm in diameter and about 5 microns in length were observed by scanning electron microscopy in the density of about 10<sup>9</sup> cm<sup>-2</sup> on the surface of the treated specimens. Positive ions of hydrogen, carbon and hydrocarbon are accelerated in the plasma sheath toward substrates. The sheath region of DC plasma plays important roles in the growth, while hot filaments assist the maintenance of DC plasma under such low voltage. By this method, the large-area growth of well-aligned carbon nanotubes is expected.

10:40am **NM+NS-TuM8 Modeling of HiPco Process for Carbon Nanotube Production**, *T. Gokcen, C.E. Dateo*, Eloret Corporation, NASA Ames Research Center; *M. Meyyappan*, NASA Ames Research Center; *D.T. Colbert, K.A. Smith, R.E. Smalley*, CNST, Rice University

High-pressure carbon monoxide (HiPco) reactor, developed at Rice University, is used to produce single-walled carbon nanotubes (SWNT) from gas-phase reactions of iron carbonyl and nickel carbonyl in carbon monoxide at high pressures (10 - 100 atm). Computational modeling is used to better understand the HiPco process. In the present model, decomposition of the precursor, metal cluster formation and growth, and carbon nanotube growth are addressed. Decomposition of precursor molecules is necessary to initiate metal cluster formation. The metal clusters serve as catalysts for carbon nanotube growth. Diameter of metal clusters and number of atoms in these clusters are some of the essential information for predicting carbon nanotube formation and growth, which is then modeled by Boudouard reaction (2CO → C(s) + CO<sub>2</sub>) with metal catalysts. The growth kinetic model is integrated with a two-dimensional axisymmetric reactor flow model to predict reactor performance.

11:00am **NM+NS-TuM9 A Gas-Phase Method for Large-scale Production of Carbon Single-walled Nanotubes**, *M.J. Bronikowski, R.K. Bradley, P.A. Willis, D.T. Colbert, K.A. Smith, R.E. Smalley*, Rice University

We have demonstrated large-scale production of high-purity carbon single-walled nanotubes (SWNT) using a gas-phase CVD process we call the HiPCO process. SWNT grow in high-pressure (10 - 100 atm), high-temperature (700 - 1200 °C) flowing CO, on clusters of catalytic metals such as iron and nickel. The metal clusters are formed in situ: metal is added to the gas flow in the form of organometallic compounds such as Fe(CO)<sub>5</sub> and Ni(CO)<sub>4</sub>. Upon heating, the organometallics decompose and the metal atoms condense into clusters of 10 - 100 atoms. These clusters serve as catalytic particles upon which SWNT nucleate and grow (in gas phase) via CO disproportionation: CO + CO → CO<sub>2</sub> + C(SWNT). SWNT material of up to 99 mole-% purity has been produced at rates of up to 350 mg/hr. The dependence of the quantity and quality of SWNT material produced on parameters such as temperature, pressure, catalyst concentration, catalyst composition and the presence of various catalyst enhancers (e.g., hydrogen and sulfur) will be discussed. The HiPCO process is currently being optimized and scaled for bulk production of 10 - 100 grams/day of high-purity SWNT material.

11:20am **NM+NS-TuM10 Diameter Selective Laser Ablation Synthesis of SWNTs: from 0.8 to 1.8 nm**, *M. Kappes*, Universitaet Karlsruhe and Forschungszentrum Karlsruhe, Germany; *S. Lebedkin*, Forschungszentrum Karlsruhe, Germany

Two-pulse Laser ablation of carbon/catalyst composite rods comprising a variety of different metal combinations has been used to generate single-walled carbon nanotubes of widely varied diameter distribution. Resulting SWNTs have been characterized by (n)IR absorption and Raman spectroscopy as well as by dynamic light scattering, SEM and NMR.

11:40am **NM+NS-TuM11 Initial Growth Study of Well-aligned Carbon Nanotubes on Fe-coated Silicon Substrate by MWCVD Process**, *C.-Y. Wen*, National Taiwan University; *L.C. Chen*, National Taiwan University, Taiwan; *C.S. Shen, Y.F. Chen*, National Taiwan University; *K.H. Chen*, Institute of Atomic and Molecular Sciences, Taiwan

Synthesis of well-aligned carbon nanotubes (CNTs) on a large area unveils the possibility to explore their properties and applications. Many synthesis methods have been reported for the growth of CNTs. Generally, it is relatively easy to generate aligned CNTs by the CVD processes. In our study, well-aligned multi-walled CNTs have been synthesized on 70 Å iron-coated silicon substrate by microwave plasma enhanced chemical vapor deposition process, wherein methane was used as carbon source. To further investigate the growth mechanism, we adopted ex-situ microscopic observation of the CNTs grown in very short growth time of several seconds. The high-resolution SEM image shows that clusters formed at the

very initial stage. As the growth time extended to 40 seconds, the back-scattering image shows that metal particles were present at the tips of CNTs while metal cones appeared in the root of CNTs. After careful Ar ion beam thinning, CNTs specimens without contamination were analyzed by high-resolution TEM. The HRTEM image indicates that the clusters were precipitated iron particles of about 10 nm in diameter and graphene layers surrounded each of them. The iron precipitation continued to form cones and part of the iron was separated and encapsulated at the tip of CNTs. The growth mechanism of CNTs could be proposed from these microscopic observations. We attempt to postulate the growth kinetics of CNTs and diffusion paths of carbon species and the catalyst iron.

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6+MM-TuM

### Nanomechanics

Moderator: W.N. Unertl, University of Maine

8:20am **NS+NANO6+MM-TuM1 Nanomechanical Properties of Molecular Organic Thin Films**, *J. Caro*, Institut de Ciencia de Materials de Barcelona (CSIC), Spain; *P. Gorostiza, F. Sanz*, Universitat de Barcelona, Spain; *J. Fraxedas*, Institut de Ciencia de Materials de Barcelona (CSIC), Spain

Using Atomic Force Microscopy we have studied the nanomechanical response to nanoindentations of surfaces of highly-oriented molecular organic (MO) thin films (thickness < 1000 nm). Fundamental parameters as the Young's modulus E, unknown for most MO materials, can be estimated from the elastic deformation using Hertzian mechanics. In the case of the quasi-one-dimensional MO conductor tetrathiafulvalene tetracyanoquinodimethane (TTF-TCNQ) we obtain E ~ 22 GPa, in excellent agreement with previous reported values obtained on single crystals using neutron scattering (E ~ 20 GPa). Above ~ 200 nN the surface deforms plastically as evidenced by discrete discontinuities in the indentation curves (~ 1 nm) associated to molecular layers being expelled by the penetrating tip. The estimated critical shear stress @tau is ~ 2 GPa. Nanoindentation permits the determination of nanomechanical parameters of MO metastable polymorphs. This is illustrated with the MO radical p-nitrophenyl nitronyl nitroxide (p-NPNN). The @alpha-phase of p-NPNN, stabilized as thin film, @footnote 2@ exhibits values of E and @tau@ two times smaller than the corresponding values of the thermodynamically most stable @beta@-phase. Measurements were performed with the same tip under the same experimental conditions, thus eliminating the uncertainty associated to the cantilever constant and tip radius. @FootnoteText@ @footnote 1@ J. P. Pouget et al. Phys. Rev. B 19 (1979) 1792. @footnote 2@ J. Fraxedas et al. Europhys. Lett. 48 (1999) 461.

8:40am **NS+NANO6+MM-TuM2 Quantitative Imaging of Dynamic Mechanical Properties by Hybrid Nanoindentation**, *S.A.S. Asif*, University of Florida; *K.J. Wahl, R.J. Colton*, Naval Research Laboratory

In this paper, we present a novel quantitative stiffness imaging technique and demonstrate its use to directly map the mechanical properties of materials with nm-scale lateral resolution. This is a powerful new approach that can eliminate tedious point-by-point analyses of indentation arrays to obtain quantitative mechanical properties of surfaces. For the experiments, we use a 'hybrid' nanoindenter, coupling depth-sensing nanoindentation with AFM scanning capabilities. AC force modulation electronics have been added, enhancing instrument sensitivity and enabling measurements of time dependent materials properties (e.g. loss modulus and damping coefficient) not readily obtained with DC techniques. Tip-sample interaction stiffness images are acquired by superimposing a small AC force (10's of nN) onto the DC imaging force (1-2 µN), and recording the AC displacement amplitude and phase as the surface is scanned. Combining a dynamic model of the indenter (having known mass, damping coefficient, spring stiffness, resonance frequency and modulation frequency) with the AC response of the tip-surface interaction allows evaluation of complex stiffness maps. We will demonstrate the use of this approach to obtain quantitative loss and storage stiffness images for elastic and viscoelastic surfaces, as well as discuss a method to directly determine loss and storage moduli from the images.

9:00am **NS+NANO6+MM-TuM3 Force-Modulated Nanoindentation of Fluorinated Polymer Thin Films Grown by PECVD**, *S.A.S. Asif*, University of Florida; *E.J. Winder, K.K. Gleason*, Massachusetts Institute of Technology; *K.J. Wahl*, Naval Research Laboratory

Thin polymer films have been of considerable interest recently in applications for electronics packaging, solid lubrication, MEMS devices,

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antifouling and adhesives. However, evaluating the mechanical properties of polymer thin films is difficult due to the low elastic moduli and viscoelastic behavior typically observed with polymers. In this paper, we present an approach for measuring the mechanical and dissipative properties of thin, compliant polymer films using AC force-modulated nanoindentation. The dynamic response of the indenter is monitored during tip-sample approach, enabling sensitive detection of the surface. Adhesive interactions, contact stiffness and damping are monitored during force-displacement measurements, and hardness and modulus evaluated. In this study, we apply the above approach to investigate the correlation between polymer thin film deposition conditions and the resulting mechanical properties. The thin polymer films were deposited on Si wafers using pulsed plasma-enhanced chemical vapor deposition (PECVD). Two different source gases were used, HFPO (hexafluoropropylene oxide) and HFC-134 (1,1,2,2, tetrafluoroethane); growth conditions were varied by altering the plasma duty cycle during deposition (plasma on-time/plasma off-time). Film thickness was measured by ellipsometry and profilometry, and chemistry examined by XPS and FTIR. Film thickness varied between 100 and 400 nm. Hardness of the films varied between 0.04 to 0.2 GPa, and complex modulus between 2 and 20 GPa, with considerable damping losses observed. Comparisons between the film deposition conditions and resulting chemistry and mechanical properties will be presented and discussed. *FootnoteText@ @footnote 1@ S.A.S. Asif, K.J. Wahl, and R.J. Colton, Rev. Sci. Instrum. 70 (1999) 2408.*

## 9:20am NS+NANO6+MM-TuM4 Dynamic Contacts to Adhesive Viscoelastic Materials, *M. Giri, W.N. Unertl*, University of Maine

Dynamic mechanical contacts with nanometer to micrometer dimensions are important in scanned probe microscopy, ultra-low load indentation, microelectromechanical systems, compact discs, etc. These contacts are poorly understood if they involve adhesive viscoelastic materials such as polymers. We have studied contacts to styrene-butadiene latex films with  $T_g$  in the range 253-301 K. Contact times were in the range 0.01-1000 s and loads were up to 1 mN. Nanoindentation was used, rather than scanned force microscopy, because of its well-defined geometry and capability to control the applied load while simultaneously measuring the displacement. Diamond probes with Berkovich and spherical end shapes were used. Load vs. displacement data showed substantial adhesion hysteresis between the loading and unloading portions. The hysteresis is at least partially due to creep as indicated by the continued increase in penetration after the start of unloading. Works of adhesion were estimated by extrapolating the measured pull-off forces to long times as suggested by Johnson. Theoretical models that include adhesion but neglect long-range creep effects could not fit the data over an entire loading-unloading cycle. Creep tests were carried out under constant load. The model of Hui, Baney, and Kramer (HBK), which predicts the response of an adhesive viscoelastic contact under increasing load, was used to extract a Mode I stress intensity functional. When this functional is normalized by the indentation strain rate, it has a simple universal time-dependence. This result supports the suggestion of HBK that the stress intensity functional may be a simpler alternative to surface energy for characterization of adhesion of viscoelastic materials. *FootnoteText@ @footnote 1@ K.L. Johnson in Microstructure and Tribology of Polymers, Eds. V.V. Tsukruk and K.J. Wahl (ACS Books, 2000). @footnote 2@ C.Y. Hui, J.M. Baney, E.J. Kramer, Langmuir 14 (1998) 6570.*

## 9:40am NS+NANO6+MM-TuM5 Precision Nanoscale Machining with STM-QCM, *J. Krim, B. Borovsky*, North Carolina State University

We have constructed an apparatus which allows us to investigate the nanoscale machining of metal surfaces resulting from the contact of a sharp tip with a high speed vibrating surface (maximum speeds over 1 m/s). The tip (tungsten or platinum alloy) is that of a Scanning Tunneling Microscope (STM), and the surface is that of a metal film deposited onto a Quartz Crystal Microbalance (QCM). The STM-QCM combination enables machining-and-imaging experiments in which the topography of the substrate is compared before, during, and after tip-surface rubbing contact at well-defined locations. The rubbing contact is either direct tip-surface contact or tunneling contact through an oxide layer. While the STM tip alone is able to machine softer materials (such as copper), the high speed vibrations of the QCM greatly enhance machining of more durable materials and oxide films. Specially prepared surfaces permit the creation of sharper, more detailed structures with 10 to 100 nm dimensions, as is demonstrated using copper and silver surfaces exposed to oxygen gas. Our talk focuses on the robustness of resulting structures compared to the ease with which they were machined. Research supported

by the NSF and the AFOSR. *FootnoteText@ @footnote 1@ B. Borovsky, B. Mason, and J. Krim, submitted to J. Appl. Phys.*

## 10:00am NS+NANO6+MM-TuM6 Size-Dependent Mechanical Properties of MoS<sub>2</sub> Nanoplates, *J. Wang, K.C. Rose, J.W. Hutchinson, C.M. Lieber*, Harvard University

The mechanical properties of materials on the nanometer scale are of great interest both for furthering our fundamental understanding as well as for use in a wide range of micro- and nano-mechanical systems. Previous experimental studies have focused on one-dimensional systems, including carbide nanorods and carbon nanotubes. For example, atomic force microscopy (AFM) has been used to show that silicon carbide nanorods have similar Young's moduli to defect free macroscopic crystals and that carbon nanotubes are much stiffer than carbon whiskers and fibers. Here, we used AFM to determine the bending stiffness of individual, structurally-isolated molybdenum oxide (MoS<sub>2</sub>) nanocrystal nanoplates (5-16 nm thick). These nanoplates were pinned to molybdenum disulfide (MoS<sub>2</sub>) surfaces on one side and were suspended freely over MoS<sub>2</sub> steps on the other side. Bending forces were measured versus displacement on the unpinned side of these MoS<sub>2</sub> nanoplates. Finite element analysis revealed that the effective Young's moduli of these MoS<sub>2</sub> nanocrystals are significantly smaller than that of bulk MoS<sub>2</sub> single crystals and that the moduli decrease with decreasing nanocrystal thickness. This novel behavior was further substantiated in subsequent experiments where it was shown that MoS<sub>2</sub> nanocrystals (1.4 - 5 nm thick) had enormous flexibility when slid over multilayer MoS<sub>2</sub> steps. These results have important implications for the sliding of nanoscale structures on rough surfaces and even for the fabrication and manipulation of smaller mechanical systems evolving in nanotechnology.

## 10:20am NS+NANO6+MM-TuM7 High Frequency Nanomechanical Systems, *D.W. Carr*, Lucent; *L. Sekaric, A. Olkhovets, S. Evoy, J.M. Parpia, H.G. Craighead*, Cornell University

INVITED

Nanofabricated mechanical systems are highly useful tools for research in physics, optics, and dynamics. We have developed fabrication processes that allow us to make suspended nanostructures in silicon and silicon nitride. We can actuate motion in these structures using electrostatic forces, and this motion is detected optically using interferometric effects. This measurement technique is sensitive to sub-nanometer motion. We have measured doubly-clamped silicon beams with fundamental resonant frequencies as high as 380 MHz. Such structures are being considered for use as chemical and biological sensors, force gauges and frequency filters. One of the obstacles for practical applications are intrinsic losses which lower the mechanical quality factor (Q-factor) of these devices. We see a strong dependence in the Q-factor on the width of these beams. As the width decreases below 100 nm, the Q factor drops sharply, indicating that the dominant energy loss mechanisms are surface related. We are also focusing on surface treatment and the effects of device geometry on dissipation. We have conducted a study of the effects of amorphous metal layers that are used in driving and detection schemes for NEMS and found that the metal layers have a detrimental impact on the devices' mechanical quality factor. We are also studying the effects of various levels of doping in single-crystal silicon on dissipation and driving schemes, a study significant for industrial use in integration with electronic devices. We have also studied the effect of parametric amplification in very small torsional resonators. An applied bias voltage effectively changes the spring constant of the system. Oscillating this bias at a specific frequency results in an amplification of the resonant motion. Swept-frequency measurements show interesting properties of the resonant spectrum, and these results agree well with the theory. Such systems may have interesting application in resonant sensors and surface probes.

## 11:00am NS+NANO6+MM-TuM9 Quantum Well Micromechanical Photon Detectors, *P.G. Datskos, S. Rajic, L.R. Senesac*, Oak Ridge National Laboratory; *I. Datskou*, Environmental Engineering Group, Inc.

We have developed a method of fabricating quantum well microstructure arrays for a variety of sensing applications. Microstructures with quantum wells allow real-time manipulation of energy states using external stress. For example this can result in an effective and rapid change in electron energy levels in photon detection devices. Such changes make possible tuning the levels to respond to desired wavelengths. We applied such GaAs/GaAlAs micromechanical quantum well arrays to detection of photons and especially uncooled infrared detection. We will present and discuss our results.

# Tuesday Morning, October 3, 2000

11:20am **NS+NANO6+MM-TuM10 Nanomechanical Systems, M.L. Roukes<sup>1</sup>**, California Institute of Technology **INVITED**

Microelectronics technology is now pushing deep into the submicron regime, yet, for the most part, work on micromachines still remains back at the few micron scale, or larger. The time is ripe to embark upon a concerted exploration of mechanical systems at the nanoscale. In this talk will highlight the promise and intrigue of this domain. Nanoelectromechanical systems, or NEMS, are MEMS scaled to submicron dimensions. In this size regime, it is possible to attain extremely high fundamental frequencies while simultaneously preserving very high mechanical responsivity (small force constants). This powerful combination of attributes translates directly into high force sensitivity, operability at ultralow power, and the ability to induce usable nonlinearity with very modest control forces. In this overview, I shall provide an introduction to NEMS and will outline several of their exciting initial applications. Our recent efforts at Caltech have culminated in nanomechanical devices with potential for new applications in electronics and metrology. These include development of the first VHF (very high frequency) mechanical resonators;<sup>1</sup> the development of mechanical electrometers yielding sensitivity below a single electron charge;<sup>2</sup> explorations of thermal transport and energy equilibration in nanoscale devices,<sup>3</sup> which have recently culminated in the measurement of the quantum of thermal conductance;<sup>4</sup> and development of mechanically-detected magnetic resonance imaging.<sup>5</sup> However, a rather stiff entry fee exists at the threshold to this new domain, new engineering is crucial to realizing the full potential of NEMS. Our work also serves to indicate some of the most crucial issues that must be addressed before the full potential of nanomechanical systems can be realized. An important example is that certain mainstays in the methodology of MEMS will, simply, not scale usefully into the regime of NEMS. Most problematic among these issues are the size of the devices compared to their embedding circuitry, their extreme surface-to-volume ratios, and their unconventional "characteristic range of operation". These give rise to some of the principal challenges in developing NEMS. Prominent among these are the need for: ultrasensitive, very high bandwidth displacement transducers; an unprecedented control of surface quality and adsorbates; novel modes of efficient actuation at the nanoscale; and precise, yet robust and reproducible approaches to surface and bulk nanomachining. Ultimately nanomechanical devices will permit access to a regime where mechanics is determined by atomistic properties; where quantum, rather than thermal, fluctuations predominate; where force and displacement detection can meet, or even exceed, the standard quantum limit; and where thermalization involves "granular" heat flow via individual phonons.<sup>6</sup> I will conclude by making some projections about this domain that initially seems exotic, but is, in reality, imminent. <sup>1</sup>A.N. Cleland and M.L. Roukes, "Fabrication of High Frequency Nanometer Scale Mechanical Resonators from Bulk Si Substrates", *Appl. Phys. Lett.*, 69, 2653 (1996). <sup>2</sup>A.N. Cleland and M.L. Roukes, "A Nanometre-Scale Mechanical Electrometer", *Nature* 392, 160 (1998). <sup>3</sup>T.S. Tighe, J.M. Worlock, and M.L. Roukes, "Direct Thermal Conductance Measurements on Suspended Monocrystalline Nanosturctures", *Appl. Phys. Lett.* 71, 2678 (1997). <sup>4</sup>K. Schwab, E.A. Henriksen, J.M. Worlock, and M.L. Roukes, "Measurement of the Quantum of Thermal Conductance", *Nature* 404, 974 (2000). <sup>5</sup>P.C. Hammel, Z. Zhang, G.J. Moore, and M.L. Roukes, "Subsurface Imaging with the Magnetic Resonance Force Microscope", *J. Low Temp. Phys.* 101, 59 (1995). / P.C. Hammel, Z. Zhang, M. Midzor, M.L. Roukes, P.E. Wigen and J.R. Childress, "The Magnetic Resonance Force Microscope", in "Frontiers in Magnetism of Reduced Dimensional Systems", B.G. Bar'yakhtar et al., eds., (Kluwer Academic, 1998). <sup>6</sup>M.L. Roukes, "Yoctocalorimetry: Phonon Counting in Nanostructures", *Physica B: Condensed Matter* 263-264, 1 (1999).

**Surface Science**

**Room 209 - Session SS2+NS+BI+EL-TuM**

**Self-Assembled Monolayers**

**Moderator:** D.H. Fairbrother, Johns Hopkins University

8:20am **SS2+NS+BI+EL-TuM1 Creating Highly Selective Organic Surfaces using Self-assembly: A New Family of Organothiols, R. Arnold**, Ruhr-Universität Bochum, Germany; **A. Terfort**, Universität Hamburg, Germany; **C. Wöll**, Ruhr-Universität Bochum, Germany

The creation of organic surfaces with specific properties via the adsorption of correspondingly functionalized organothiols has recently attracted considerable interest, e.g. in the context of bio-sensors and biomimetics. In case of alkanethiols some functional groups, however, interact so strongly with each other that the ordering within the SAMs is affected, e.g. in the case of -COOH functional groups.<sup>1</sup> The situation can be improved by using more rigid backbones, e.g. oligophenyl units.<sup>2</sup> With regard to biochemical applications in many cases the distance between adjacent organothiol units (4.97 Å) is too small to immobilize larger molecules, e.g. small proteins. In the past these problems could be overcome in some cases by diluting the functionalized organothiol in a shorter, nonfunctionalized thiol. Here, we present a different approach where a more bulky thiol is used, which increases the nearest neighbor distance. We will present the results of a study using several homologues of triptycethiols. SAMs formed from these compounds were characterized by using XPS, IRRAS, NEXAFS, LEED and TDS. The results reveal the formation of well ordered monolayers, which are anchored to the gold surface in a more distant lateral structure than alkane- or p-terphenylthiols. <sup>1</sup>Himmel, H.-J.; Jäger, B.; Buck, M.; Wöll, C. *Thin Solid Films* 1997, 307, 9885-9893 <sup>2</sup>Himmel, H.-J.; Terfort, A.; Wöll, C. *J. Am. Chem. Soc.* 1998, 120, 12069-12074.

8:40am **SS2+NS+BI+EL-TuM2 Characterization of the Alkanthiol/Metal Interface by High Resolution Core Level Spectroscopy, K. Heister, H. Rong, M. Buck**, University Heidelberg, Germany; **L. Johansson**, University Karlstad, Sweden; **M. Zharnikov, M. Grunze**, University Heidelberg, Germany

During the last decade X-ray Photoelectron Spectroscopy with a laboratory X-ray source became a conventional technique to characterize thiol derived SAMs. However, due to the mostly poor energy resolution, a strong attenuation of the photoelectron signal, and a low photoionization cross-section of the relevant core levels at high photon energies a precise binding energy analysis of an important building block of a SAM, the SAM/metal interface was hardly possible, even though high resolution photoelectron spectroscopy could give important information about the chemical state of the atoms in this region. Taking advantage of the high performance and tuneability of the third generation synchrotron sources we have firstly applied the synchrotron-based High Resolution Core Level Spectroscopy to study the SAM/metal interface. The variable photon energy of the synchrotron light and a high energy resolution of the spectrometer (0.1-0.3 eV) enabled us to resolve the bulk and surface components of the substrate emission peak (Au 4f / Ag 3d) and monitor the evolution of these components upon the alkanethiol and biphenylthiol adsorption. Simultaneously, the interaction of the thiol-derived molecules with the substrate was followed by monitoring the S2p doublet attributed to the sulfur head group of these molecules. Only one sulfur species was found in the densely packed SAMs, which implies an equivalent bonding geometry for all adsorbed molecules. In SAMs comprising of specially designed, mixed aliphatic-aromatic molecules a periodical, 'odd-even' shift of the S2p binding energy with the varying length of the aliphatic part was observed. This shift can be attributed to the distortion of the substrate-S bonding angle resulting from the unfavorable package conditions occurring at definite lengths of the aliphatic part. This work has been supported by the German Bundesministerium für Bildung, Wissenschaft und Technologie through grant No. 05 SL8VHA 2 and by DAAD.

9:00am **SS2+NS+BI+EL-TuM3 The Influence of the Endgroup and the Chain Length on the Growth of CH@sub 3@- and CF@sub 3@-terminated Alkanetiols on Au(111), J. Pflaum**, Princeton University; **G. Bracco**, University of Genova, Italy; **G. Scoles**, Princeton University; **R. Lee**, University of Houston; **A. Kahn**, Princeton University

The influence of the functional endgroup and the CH@sub 2@ chain length on the growth of alkanethiols on Au(111) was studied by scanning tunneling microscopy (STM) and x-ray surface diffraction in grazing-incidence geometry (GIXD). Thiols are model systems for self-assembled monolayers (SAMs) and exhibit a complex phase diagram as function of

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coverage. The structure and the electronic properties of the SAMs are determined by the sulfur headgroup, the CH@sub 2@ backbone and the functional endgroup. Leaving the sulfur headgroup unchanged, we studied how the film structure depends on the endgroup by comparing CH@sub 3@- and CF@sub 3@-terminated thiols. All films were prepared from solution on an atomically ordered Au(111) surface. The lateral order of the as-grown CH@sub 3@-terminated films corresponds to the c(4x2) phase, i.e. the highest density standing-up phase. From STM studies we conclude that the arrangement of CH@sub 3@ endgroups corresponds to a pinwheel-like structure rather than to a zig-zag-like structure. In contrast, CF@sub 3@(CH@sub 2@)@sub 9@SH showed no lateral ordering as seen by STM and GIXD. However, the difference between electron densities at the CF@sub 3@/vacuum and the SH/Au interfaces induces an oscillation of the GIXD reflectivity. Fitting the periodicity of the modulation using the Parratt formalism leads to an estimate of the film thickness and its roughnesses at both interfaces. In spite of the lack of lateral order the film appears to be made by standing-up molecules. Differences between the thickness measured by ellipsometry and x-rays will be discussed. We will also explore the lying-down phase of alkanethiols and fluorinated thiols as organic templates for organic heterostructures. Initial results on the growth of PTCDA on such templates will be presented. This work was supported by the MRSEC program of the National Science Foundation (DMR-9809483). J.P. thanks the Deutsche Forschungsgemeinschaft for support (Grant No. PF 385/1-1).

9:20am **SS2+NS+BI+EL-TuM4 Self-Assembled Monolayers of Terphenyl Derivatized Thiols; Adsorption, Insertion Process and Electrical Conduction**, *T. Ishida*, JRCAT-NAIR and PRESTO-JST, Japan; *W. Mizutani*, JRCAT-NAIR, Japan; *N. Choi*, JRCAT-ATP, Japan; *H. Tokumoto*, JRCAT-NAIR, Japan

The investigation of SAMs made from conjugated molecules is highly activated from a viewpoint of molecular electronics as well as stable SAM formation. In the present study, we have investigated an insertion process of conjugated molecules, terphenylthiol (TPO), terphenyl methanethiol (TP1), terphenyl propanethiol (TP3), into nonanethiol self-assembled monolayers (SAMs) on Au(111) by STM. STM observation revealed that the insertion process is dependent on the molecular length of conjugated molecules. At the initial stage of insertion, replaced area of TP1 is larger than those of TPO and TP3. However, when the immersing time is more than 12h, the replaced area of TP3 is larger than those of TPO and TP1. The insertion process is likely to be determined by the solubility of the conjugated molecules and thermal dynamics. The single molecular resistance were increased with the number of the methylene groups, and obtained about 20G-ohm (TPO), 40 G-ohm (TP1) and 66 G-ohm (TP3). The vertical conduction of the conjugated molecular domains of TP1 and TP3 depended on their lateral sizes, while strong dependence was not observed in the case of TPO, suggesting that methylene group is necessary between the sulfur and aromatic rings to increase the vertical conduction of molecular domain.

9:40am **SS2+NS+BI+EL-TuM5 Structure and Chemistry of Alkanethiol Self-Assembled Monolayers**, *G.E. Poirier*, *T.M. Herne*, *C.C. Miller*, *M.J. Tarlov*, National Institute of Standards and Technology

INVITED

Derivatized alkanethiols form dense, physically blocking films on Au surfaces thereby providing an effective and parsimonious method to control the chemical, physical, and electron-transfer properties of electrode surfaces. To predict the function of these monolayers in device applications, scientist require an understanding the molecular-scale structure and chemistry. Our structure studies were conducted using gas-phase transport of decanethiol onto clean Au(111). Characterization was accomplished using ultrahigh vacuum scanning tunneling microscopy. At low surface coverage, decanethiol exists as a 2-dimensional gas. With increasing coverage the molecules sequentially condense into islands of three discrete commensurate crystalline lattices, each characterized by alignment of the molecular axes with the surface plane (striped phases). Above saturation coverage of the densest striped phase, the monolayer undergoes an edge-mediated melting transition forming a supercooled 2-dimensional liquid. Domains of the c(3x2\*3) phase, characterized by alignment of the molecular axes close to the surface normal, nucleate and grow from this surface liquid. The reaction of these monolayers with ozone was characterized using scanning tunneling microscopy and x-ray photoelectron microscopy; our results show that exposure to ozone results in oxidation of the thiol terminus. The reaction initiates at the c(3x2\*3) domain boundary network and propagates into the domains. Above a threshold surface oxygen content, the monolayer converts to a two-dimensional fluid that can subsequently recrystallize to a commensurate

monolayer of partially oxidized thiol. Further exposure to ozone results in conversion of the monolayer to a fluid phase and a 10% to 30% expansion of the Au lattice at the Au-thiol interface with concomitant formation of Au islands. Our results demonstrate that crystallographic defects in monolayer films can play an important role in their chemical reactions.

10:20am **SS2+NS+BI+EL-TuM7 Characterization of SAMs with Contrast Variation SPR Technique**, *K. Tamada*, NIMC and RIKEN Frontier Program, Japan; *H. Akiyama*, *J. Nagasawa*, NIMC, Japan

We report characteristics of azobenzene-containing self-assembled monolayers (SAMs) which is designed and synthesized for surface photo-isomerization reaction. The surface reaction was monitored by Surface Plasmon Resonance Spectroscopy (SPR), in which the contrast variation technique with various organic solvents was used to improve the accuracy for determination of the optical thickness change by surface reaction. The SAM formation was monitored by kinetic mode experiment with SPR in 0.1mM hexane solution, and after rinsing, refractivity change by UV-VIS photo irradiation was studied in various solvents. In this study, hexyl azobenzene thiol (12-(4-((hexylphenyl)azo)phenoxy)dodecane-1-thiol) SAM was used as an unreactive surface and unsymmetrical azobenzene-disulfides SAMs with the same azobenzene functions were used as reactive ones. Following the previous reports, monomeric dispersion of dye function with disordered chains seems to be necessary to realize highly reactive surface. For our unsymmetrical azobenzene-disulfides SAMs, the free volume for photo-isomerization reaction are guaranteed by 50% dilution of dye functions on surface at monomolecular level. As a result, it was confirmed that unsymmetrical azobenzene-disulfides SAMs were highly reactive, especially, in good solvents (alkanes: C5, C6, C7, C8) and the length of alkyl side chains was quite efficient for surface reactivity.

10:40am **SS2+NS+BI+EL-TuM8 Characterization of a Polymerized Self-Assembled Monolayer Using NEXAFS**, *A.L. Marsh*, *D.J. Burnett*, University of Michigan; *D.A. Fischer*, National Institute of Standards and Technology; *C.E. Evans*, *J.L. Gland*, University of Michigan

Near-edge X-ray Absorption Fine Structure, or NEXAFS, at the C-K-edge was used to characterize the orientation of the polymeric backbone in a self-assembled monolayer of 15,9-polydiacetylene. Monolayers were fabricated from the assembly of molecules of dinonacos-10, 12-diyne-disulfide from a chloroform solution onto a 2000 Angstrom gold film on a mica substrate. Polymerization occurs across one of the C-C triple bonds in the chain, which results in a polymeric network located within the monolayer. Since resonance intensities in NEXAFS spectra are dependent on electric dipole selection rules, it is possible to determine the orientation of the polymeric backbone by comparing spectra at normal incidence (E vector parallel to the surface plane) with spectra at glancing incidence (E vector perpendicular to the surface plane). From the two spectra it was determined that the polymeric backbone is oriented parallel to the surface, while the alkyl chains are oriented perpendicular to the surface. Since NEXAFS probes unfilled molecular orbitals, it is capable of distinguishing between various bonds, i.e. a C-C double bond versus a C-C triple bond, making it possible to determine structural changes as a function of temperature. Upon increasing the temperature, the C-C double bond pi\* resonance increases, while the C-C triple bond pi\* resonance decreases. These changes would be consistent with a degradation of the polymer backbone. Above a threshold temperature, the changes are irreversible, leading to eventual thermal degradation of the monolayer.

11:00am **SS2+NS+BI+EL-TuM9 An Estimation of Effective Mean Free Path of Photo- and Auger Electrons in Partial Yield Measurements using Self-assembled Monolayers**, *M. Zharnikov*, *S. Frey*, *K. Heister*, *M. Grunze*, Universität Heidelberg, Germany

In the partial electron yield (PEY) acquisition mode commonly used in X-ray absorption spectroscopy both elastically and inelastically scattered electrons contribute to the signal with the latter contribution presumably dominating. In this case a majority of inelastic scattering events will not result in the signal attenuation as it happens in the X-ray photoelectron spectroscopy (XPS). The scattered electrons will still have a kinetic energy in the acquisition range of the spectrometer. The related values of mean free path (MFP) should be, therefore, noticeably larger than the well-known inelastic mean free paths for electrons of definite kinetic energy. We have performed XPS and near edge X-ray absorption fine structure (NEXAFS) spectroscopy measurements for series of self-assembled monolayers of alkanethiols on gold substrate. The length of the alkyl chain and, subsequently, the film thickness was varied. In agreement with the expectations, the obtained effective MFPs for the Au 4f photoelectrons and C@sub KLL@ Auger electrons in the PEY acquisition mode exceed the

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respective values for the elastically scattered electrons of the same kinetic energies (the Au 4f and C<sub>1s</sub> electrons made up the elastic component of the acquired PEY signals). Furthermore, the PEY-MFP for the C<sub>1s</sub> Auger electrons increased with decreasing retarding voltage of the PEY detector, which correlates with the increasing contribution of the inelastically scattered electrons in the acquired signal. The obtained results are of importance for the analysis of NEXAFS spectra in both self-assembled monolayers and polymers. This work has been supported by the German Bundesministerium fuer Bildung, Wissenschaft und Technologie through grant No. 05 SF8VHA 1 and by the Fonds der Chemischen Industrie.

11:20am **SS2+NS+BI+EL-TuM10 Growth Process and Thermal Stability of Semifluorinated Alkanethiol Self-Assembled Monolayers on Au(111)**, *M. Hara*, Frontier Research System, RIKEN, Japan; *A. Suzuki*, Tokyo Institute of Technology, Japan; *K. Tamada*, National Institute of Materials and Chemistry, Japan; *H. Fukushima*, Seiko Epson Co., Japan; *T.R. Lee*, University of Houston

Growth process and thermal stability of semifluorinated alkanethiol (CF<sub>3</sub>(CF<sub>2</sub>)<sub>m</sub>(CH<sub>2</sub>)<sub>n</sub>SH) self-assembled monolayers (SAMs) on Au(111) have been investigated by thermal desorption spectroscopy (TDS) and scanning tunneling microscopy (STM). The growth kinetics showed nearly the Langmuir adsorption isotherm and the etch pits were formed in the upright phase, while the striped phases were not observed in the initial growth stage for shorter (CH<sub>2</sub>)<sub>n</sub> semifluorinated SAMs. In TDS, no significant peaks can be obtained for dimer molecules and decomposed species, suggesting no associative desorption nor dimerization and thermal stability of the semifluorinated molecules during heating up to 650 K. Since longer (CH<sub>2</sub>)<sub>n</sub> semifluorinated SAMs remained the same chemisorbed state in the monolayer after annealing at around 480 K, it has been confirmed that also the alkyl chain part plays an important role for the thermal stability and the ordering in the semifluorinated alkanethiol SAMs. Following those results, we propose more detailed surface phase transition model of semifluorinated alkanethiol SAMs in the growth and annealing processes.

11:40am **SS2+NS+BI+EL-TuM11 Multi-technique Study of Self-Assembled AuCN Monolayers on Au(111) Formed by Electrochemical Deposition**, *T. Yamada*, Waseda University, Japan; *R. Sekine*, Shizuoka University, Japan; *T. Sawaguchi*, AIST/MITI, Japan

Two kinds of monolayers of AuCN electrodeposited on Au(111), indexed (1.15xsr@3R-30°) and (1.41x2@sr@3R-30°), have been investigated by XPS, UPS and HREELS as well as LEED, AES and STM to determine the geometrical, electronic and vibrational properties. Electrodeposition was performed in an aqueous 1 mM KAu(CN)<sub>2</sub> solution by applying an electrode potential about 0 - +0.1 V vs SCE on the Au(111) crystal. Sharp LEED patterns were obtained for these two kinds of adlayers. AES indicated that both of these adlayers were composed Au, C and N without impurity. Well ordered adlattices composing domain structures (domain size ca. 10 nm) were observed by STM. XPS yielded Au 4f signals from AuCN indicating small fractional positive charges on the Au atom incorporated in AuCN. The UPS of AuCN/Au(111) was composed of the Au orbitals and weak signals from CN orbitals, assigned by relativistic DV-Xa molecular orbital calculation. The binding energies of CN orbitals are in the order of 4@sigma@ > 5@sigma@ > 1@pi@, which indicates that the C-Au bond is essentially covalent. HREELS yielded vibrational spectra similar to that obtained for AuCN crystalline powder. The C-N stretching frequencies were found to be 2140-2160 cm@super -1@, which are consistent with the covalent nature of the C-Au bond. In the frequency region below 300 cm@super -1@, loss peaks related to the Au-N bonds were seen. The (1.15xsr@3R-30°) adlayer is concluded to be composed of -AuCN- linear chains (polymer chains) that are identical to those embedded in the AuCN crystal. For the (1.41x2@sr@3R-30°) adlayer, HREELS indicated distortion or breaking of Au-N bonds. Some structural models are proposed for this. These results reveal a special inorganic polymeric feature of the self-assembled AuCN adlayers lying parallel along the surface. @FootnoteText@ @footnote 1@G. A. Bowmaker, B. J. Kennedy and J. C. Reid, Inorg. Chem. 37, 3968 (1998).

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## Electronics

### Room 312 - Session EL+NS-TuA

#### Nanoelectronics

**Moderator:** R.S. Goldman, University of Michigan

#### 2:00pm EL+NS-TuA1 Nano-switches Using Vacuum Nano-electronics and Superconducting Weak Links, *D.G. Hasko*, Cambridge University, UK

INVITED

New physical principles influence device operation when size is reduced to the nanometre range. Recent research has led to two types of switching devices; vacuum nanoelectronic (VNE) and superconducting switches have been described and are reviewed in this paper. Conventional vacuum microelectronics exploits cold field emission of electrons in devices made by microcircuit fabrication techniques but requires UHV vacuum operating conditions. By reducing the field emission tip radius and the tip-extractor electrode spacing a new class of VNE devices may be operated at lower voltages and with improved stability. Tips of nanometre size also show much smaller angular spreads and reduced energy spread. Diode and triode devices, with electron transport path length of  $\sim 100$ nm (shorter than the mean free path in air) were fabricated and their electrical characteristics reported. Hot phonon injection from an electrically isolated heater has demonstrated effective switching behaviour in Nb weak link junctions and is of great interest for high speed and quantum effect circuit functions. This method isolates the control circuit from the weak link and has demonstrated significant device gain in contrast to previous device structures.

#### 2:40pm EL+NS-TuA3 Persistent-current Qubits for Quantum Computation, *E.L. Mooij*, DIMES Institute, The Netherlands

INVITED

In a collaboration between MIT and Delft University we have designed a quantum bit (qubit) for quantum computation that consists of a superconducting loop with three small Josephson junctions in series. When the flux through the loop is close to half a superconducting flux quantum, the qubit has two stable macroscopic quantum states with persistent currents in opposite directions. Quantum transitions between the two states are possible if the capacitance of the junctions is small. Samples are fabricated from aluminum and measurements are performed at very low temperatures to reduce decoherence effects. First measurement results have been obtained that demonstrate the quantum superposition of the states. Next experiments will focus on time-dependent response. With these qubits in principle a scalable quantum computer can be constructed if the decoherence time is long enough. A quantum computer of sufficient size can perform calculations beyond the power of a conventional computer. However, many challenges will have to be faced before a quantum computer is realized. @FootnoteText@ @footnote 1@J.E. Mooij, T.P. Orlando, L.Levitov, Lin Tian, Caspar H. van der Wal and Seth Lloyd, Science 285, 1036 (1999)

#### 3:20pm EL+NS-TuA5 Coulomb Blockade Devices Fabricated by AFM-manipulation of Nanoparticles, *S. Carlsson, T. Junno, H. Xu, L. Samuelson*, Lund University, Sweden

We report successful fabrication of Coulomb blockade devices obtained by manipulation of pre-fabricated nanoparticles, using an atomic force microscope (AFM) as a nano-engineering tool. This approach, together with in-situ electrical measurements during manipulation, allows the formation of tunnel gaps with accuracy on the Ångstrom scale. Three-terminal single-electron transistors (SETs) with ideal electrical characteristics are obtained, demonstrating Coulomb blockade as well as Coulomb staircase in the I-V characteristics, and with hundreds of current oscillations as function of gate voltage. Furthermore, we have built double-island structures with three gaps trimmed to tunneling dimensions and with two addressing gates, allowing control of the charge distribution, or polarization, of these two-atom artificial molecule objects.

#### 3:40pm EL+NS-TuA6 A Novel Scheme for the Fabrication of Ultra-short Metal-oxide-semiconductor Field-effect Transistors, *R. Martel*, IBM T.J. Watson Research Center; *J. Appenzeller, J. Knoch*, Physikalisches Institut, RWTH Aachen, Germany; *K. Chan*, IBM T.J. Watson Research Center; *M. Tanner, S. Thomas, K.L. Wang*, University of California, Los Angeles; *Ph. Avouris*, IBM T.J. Watson Research Center; *J.A. del Alamo*, Massachusetts Institute of Technology; *P. Solomon*, IBM T.J. Watson Research Center

We present a novel scheme for the fabrication of ultra-short channel length metal-oxide-semiconductor field-effect transistors (MOSFETs) involving nanolithography (proximal probe or e-beam) and molecular beam epitaxy (MBE). The active channel is undoped and is defined by a combination of nanometer-scale patterning and anisotropic etching of a n<sup>+</sup>-layer grown on a silicon on insulator (SOI) wafer. The method is self-limiting and can produce MOSFET devices with channel lengths of less than 10nm. Measurements on the first batches of n-MOSFET devices fabricated with this new approach will be presented. They show very good output characteristics and good control of the short channel effects. The combination of highly doped contact areas (n<sup>+</sup> with a nominally undoped channel region (p<sup>+</sup>) is now being explored further to keep the parasitic resistances low and possibly achieve ballistic transport at room temperature.

#### 4:00pm EL+NS-TuA7 Quantitative Analysis of Charge Injection and Discharging of Si Nanocrystals and Arrays by Electrostatic Force Microscopy, *L.D. Bell*, Jet Propulsion Laboratory, Caltech; *E. Boer, M. Ostraat*, Caltech; *M.L. Brongersma*, Caltech, US; *R.C. Flagan, H.A. Atwater*, Caltech

Charge injection and storage in dense arrays of silicon nanocrystals in SiO<sub>2</sub> is a critical aspect of the performance of potential nanocrystal flash memory structures. We have performed charging experiments on Si nanocrystals both embedded within and deposited on SiO<sub>2</sub> using conducting-tip atomic force microscopy (AFM). In the case of both isolated aerosol-deposited nanocrystals and those formed by ion implantation and annealing of SiO<sub>2</sub> films, charging has been accomplished by moving a conducting AFM tip close enough to the nanocrystal to transfer charge. This charging and subsequent discharging were characterized by monitoring the apparent change in nanocrystal height detected by AFM. The trapped charge produces an electrostatic force component that changes the response of the AFM tip, causing a change in the apparent height of the nanocrystal. This mode of electrostatic force microscopy (EFM) together with electrostatic modeling enables quantitative measurement of the trapped charge and discharging dynamics. Simulation enables the EFM sensitivity to be estimated systematically as functions of tip radius and height. Forces due to interaction with this charge and the induced charge on the tip can be determined, and AFM response to these forces can be calculated. Constant-force-gradient contours have been calculated that agree well with measured profiles, and we can determine the amount and location of the injected charge as well as some details of the discharge mechanism. Trapped charge as small as 7e is detected in isolated small nanocrystals, and charge in the range 100e - 1000e is observed in larger isolated nanocrystals or embedded nanocrystal ensembles. The combination of EFM imaging and simulations can be used to estimate the homogeneity of the charge density and to probe for high conductance paths within a nanocrystal floating gate. Modeling indicates a discharge mechanism consistent with tunneling through a field-lowered barrier.

#### 4:20pm EL+NS-TuA8 Quantized Conductance in AuPd Alloy Nanocontacts, *A. Sakai, A. Enomoto, J. Sasaki, S. Kurokawa*, Kyoto University, Japan

Quantization of conductance can be observed most beautifully in Au nanocontacts but much less clearly in transition metals such as Pd. Then, an interesting problem is how the quantized conductance changes by alloying Au with Pd. Do all quantized peaks in the conductance histogram of Au disappear by a small amount of Pd, or do they survive even for Pd-rich nanocontacts? To answer this problem, we have carried out conductance measurements on AuPd nanocontacts in air at room temperature. We prepared Au<sub>x</sub>Pd<sub>1-x</sub> alloy wires with x = 20, 40, 50, 80, and 95 wt%, and measured the transient conductance at the break of two contacting wires. We find that the transition from the conductance behavior of Au to that of Pd takes place gradually with increasing the Pd concentration: sharp peaks in the histogram of Au are suppressed and replaced by a broad and featureless distribution observed in the histogram of Pd. At x = 40 and 50 wt%, both quantized peaks of Au and broad background of Pd coexist in a conductance histogram. This result

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implies that the conductance of AuPd nanocontacts becomes that of Au or Pd depending on which one of two constituent atoms occupies the narrowest constriction of the contact.

**4:40pm EL+NS-TuA9 Bistability in Conductance of Point Contact formed between a Metal Tip and Ga-terminated Si(111), S.L. Pryadkin, D. Chen, Rowland Institute for Science**

Recently, it has been found that voltage-current dependence of a tunnel junction formed by an STM tip and a Ga terminated Si(111) surface exhibits large hysteresis at 77K, similar to that of a double barrier structure. This new finding raises the possibility of creating nanoscale storage and switching devices, compatible with silicon technology. To further explore this potential, we have studied the effects of temperature and doping. It is found that the hysteresis exists in a wide range of temperature. Moreover, when the tip is brought to a point contact with the surface, it still gives rise to the bistable transport. This allows us to simplify the junction structure even further and to determine the timing characteristics of this new nanoscale switching/storage device. @FootnoteText@ @footnote 1@ I.B.Altfeder, D.M.Chen Phys.Rev.Lett. 84, p.1284(2000).

**5:00pm EL+NS-TuA10 Analysis of Non-linear Behaviour in Gold Nanowires, A. Wlasenko, P. Gruetter, McGill University, Canada**

In the presented experiment, a gold nanowire is formed with a mechanical break junction. A voltage bias is applied in the form of a triangle wave (typically 0.1 Hz, 2V@sub pp@) plus a small sine wave (typically 10kHz, 10mV@sub rms@) across the nanowire in series with a load resistor. A current pre-amp measures I(V), while a lock-in amplifier measures its partial derivative with respect to voltage. While others have made I(V) measurements of nanowires (Costa-Kramer et al., PRB 55, 5416 (1997) and Costa-Kramer et al., Nanoscale Science and Technology pp. 1-10 (1998) Kluwer Academic), the simultaneous measurement of the derivative allows powerful analysis without choosing a physical model or using mathematical fits. In general, the current is not just a function of voltage [I(X,V)=g(X)f(X,V)]. For instance, the geometry of the nanowire or presence of scatterers should have an effect on the current. The analysis indicates how changes in these non-voltage factors [X] are changing the current [dI/dV @DELTA@I(@DELTA@X,V)] without having to know explicitly what these are factors are or how they are changing. It is also possible to determine how the form of current f(X,V) is changing with respect to the voltage without knowing g(X). Several individual sets of data shall be investigated that illustrate particular features of both typical and atypical nanowire behaviour. A discussion is presented of the possible physical arguments concerning these features and general trends.

## Magnetic Interfaces and Nanostructures

### Room 206 - Session MI+NS+NANO 6-TuA

#### Magnetic Imaging II

Moderator: F.J. Himpsel, University of Wisconsin, Madison

**2:00pm MI+NS+NANO 6-TuA1 Correlation of Ferromagnetic and Antiferromagnetic Spin Orientation Observed by Photoemission Electron Microscopy, S. Anders, A. Scholl, F. Nolting, H.A. Padmore, Lawrence Berkeley National Laboratory; J. Stohr, J. Luening, Stanford Synchrotron Radiation Laboratory; J.W. Seo, University of Neuchatel, Switzerland; J. Fompeyrine, J.-P. Locquet, IBM Research Division, Switzerland; M. Scheinfein, FEI Company**

INVITED

Photoelectron emission microscopy (PEEM) using polarized x rays is a unique tool for the study of ferromagnetic (FM) and antiferromagnetic (AFM) materials. FM materials are studied using x-ray magnetic circular dichroism (XMCD) and AFM materials using x-ray magnetic linear dichroism (XMLD). The elemental specificity of PEEM allows to study individual layers in multilayer structures, and to investigate the coupling between them. Increasingly complex layered structures containing magnetic and antiferromagnetic materials are used in modern magnetic devices, and knowledge of the magnetic properties of the layers and interfaces is essential for the understanding of the properties of these devices. Of particular interest is the effect of exchange biasing at the interface of an AFM and an FM. AFM materials have been difficult to study so far because of a lack of methods with sufficient spatial resolution and surface sensitivity. We have investigated the magnetic and topographic surface structure of several AFM materials, in particular thin singlecrystalline and polycrystalline NiO and LaFeO@sub 3@ films. We were able to resolve the antiferromagnetic surface structure of those materials, showing

antiferromagnetic domains, and antiferromagnetic patterns, correlated to the surface topography. Local NEXAFS spectra yielded information about the antiferromagnetic orientation at the sample surface. The study of an FM Co thin film on top of an AFM LaFeO@sub 3@ film showed for the first time a direct correlation between AFM and FM domains.

**2:40pm MI+NS+NANO 6-TuA3 Magnetic Imaging of NiO/Ag(001) Thin Film using PhotoEmission Electron Microscope, W. Zhu, University of Connecticut, US; L. Seve, B. Sinkovic, University of Connecticut; A. Scholl, S. Anders, Lawrence Berkeley National Laboratory**

PhotoEmission Electron Microscope (PEEM) combined with linearly polarized synchrotron X-rays provides a powerful way of imaging magnetic domains in antiferromagnetic thin films. We have performed magnetic imaging on antiferromagnetic thin film of NiO with PEEM. The 90-Å thick NiO film is of (001) orientation, epitaxially grown on a Ag (001) single crystal substrate. The magnetic contrast is found to be correlated with the topological contrast, which is caused by the local thickness variation in the film. Micro-X-ray absorption spectra (XAS) in areas of different contrast revealed that the directions of magnetic moments within these areas are differently oriented with respect to the X-ray polarization direction. The difference in Micro-XAS from these areas disappeared at temperature of ~350 °C (above the Neel temperature), where the antiferromagnetic order disappears. Experiments with the X-ray polarization direction parallel to [100] and [110] direction of the film give similar results, which indicates that the magnetic contrast is due to the in-plane vs. out-of-plane magnetic moments orientation rather than the differently oriented in-plane moments. These results are consistent with our recent spectroscopic studies of NiO/Ag(001) films of various thickness.

**3:00pm MI+NS+NANO 6-TuA4 Magnetic Imaging by Local Tunneling Magnetoresistance - A High Resolution Technique, W. Wulfhekel, H.F. Ding, J. Kirschner, MPI Halle, Germany**

INVITED

We give an overview over our recent efforts of magnetic imaging using scanning tunneling microscopy with a ferromagnetic tip. Magnetic sensitivity is obtained on the basis of local tunneling magnetoresistance between a soft magnetic tip and the sample. The imaging capacities of the technique are illustrated with exemplary studies of the surface domain structure of different itinerant ferromagnets. On Co(0001) we find surprisingly narrow sections of the walls of only 1.1nm width, over an order of magnitude less than previously observed in Co. Recording quantitative profiles of the perpendicular component of the magnetization across the wall and comparing the experimental data with micromagnetic calculation, the narrow sections are identified as 20° domain walls. Besides magnetic imaging, we focus on the influence of the stray field of the tip on the magnetic structures under investigation. In the limit of soft magnetic materials or strong stray fields, the wall mobility and magnetic susceptibility can be studied on the local scale. Finally, measurements of magnetoresistance versus tunneling voltage and tip sample distance give deeper insight into the mechanisms of spin polarized tunneling.

**3:40pm MI+NS+NANO 6-TuA6 Self-assembled Magnetic Nanowires Studied with Spin-polarized Scanning Tunneling Microscopy, T.-H. Kim, W.-G. Park, Y. Obukhov, Y. Kuk, Seoul National University, Korea**

In thin film of immiscible Co and Ag alloys, nanowires have been observed. The alternating stripes, magnetic Co stripes and non-magnetic Ag stripes, are formed on W(110) substrate by the driving force of the phase separation. The film can be grown sequentially, or deposited simultaneously. The periods of the stripes are found to be 20 to 30 Å, perpendicular to the long axis of the stripes. The relations between the morphology and the magnetic contrast of the self-assembled magnetic nanowires have been studied with spin-polarized scanning tunneling microscopy. Using an electromagnet for tip magnetization, the magnetic field can be applied to the soft magnetic tip both parallel and perpendicular to the axis of the tip. With this setup, we are able to image the direction of the magnetization of the sample.

**4:00pm MI+NS+NANO 6-TuA7 Direct Visualization of Magnetic Nanowires by Spin-Polarized Scanning Tunneling Spectroscopy, O. Pietzsch, A. Kubetzka, M. Bode, R. Wiesendanger, University of Hamburg, Germany**

While scanning tunneling microscopy (STM) and spectroscopy (STS) are the established methods of choice for the study of structural and electronic surface properties at ultimate real space resolution, no equivalent technique for magnetic imaging was available so far. The most widely applied surface sensitive methods as, e. g., magneto-optical Kerr effect (MOKE), average over comparably large sample fractions. Here we present a recent spin-polarized STS study, carried out with an STM especially

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designed for magnetic imaging. @footnote 1@ We will show high resolution images of a self-organized array of Fe nanowires grown on a stepped W(110) single crystal. @footnote 2@ The magnetic wires have a periodicity of 8 nm, an average width of 4 nm, and a thickness of two atomic layers. Making use of ferromagnetically coated STM tips with the appropriate anisotropy we were able to image the magnetic domain structure in detail. The magnetism of the stripe system is governed by perpendicular anisotropy. @footnote 3@ Adjacent stripes exhibit antiferromagnetic coupling mediated by the stray field. Our images allow the investigation of the influence of local structural defects as, e. g., non-uniform stripe width or dislocation lines, on the magnetic properties on a sub-nanometer scale. The width and orientation of domain walls within single stripes is determined. We will show how the domain structure is affected by applied external fields of up to 0.5 Tesla. The contrast mechanism will be explained. The imaging method is of general applicability for the study of the surfaces of magnetic nanostructures. @FootnoteText@ @footnote 1@ O. Pietzsch et al., Rev. Sci. Instrum. 71, 424 (2000) @footnote 2@ O. Pietzsch, A. Kubetzka, M. Bode, and R. Wiesendanger, Phys. Rev. Lett., in press. @footnote 3@ J. Hauschild, U. Gradmann, and H. J. Elmers, Appl. Phys. Lett. 72, 3211 (1998).

4:20pm **MI+NS+NANO 6-TuA8 Real-Space Imaging of Two-Dimensional Antiferromagnetism on the Atomic Scale**, *M. Bode*, University of Hamburg, Germany; *S. Heinze*, Forschungszentrum Jülich, Germany; *A. Kubetzka*, O. Pietzsch, University of Hamburg, Germany; *X. Nie*, *S. Blügel*, Forschungszentrum Jülich, Germany; *R. Wiesendanger*, University of Hamburg, Germany

The ultimate limit of two-dimensional antiferromagnetism (2D-AFM) is a magnetic monolayer of chemically equivalent atoms, where adjacent atoms at nearest-neighbor sites have magnetic moments with opposite directions, deposited on a non-magnetic substrate. @footnote 1@ Since the total magnetization of this film is zero spatially averaging techniques like spin-polarized photoelectron spectroscopy cannot be used for an experimental verification of (2D-AFM). We have resolved the two-dimensional antiferromagnetic structure within a pseudomorphic monolayer film of chemically identical manganese atoms on tungsten (110) by spin-polarized scanning tunneling microscopy (SP-STM) at 16 Kelvin. @footnote 2@ While images of the chemical surface unit-cell without any magnetic contribution were obtained using a non-magnetic W-tip, spin-polarized electrons from magnetically coated tips probe the change in translational symmetry due to the magnetic  $c(2 \times 2)$ -superstructure of Mn/W(110). Based on fundamental theoretical arguments it will be shown that SP-STM is a powerful technique for the investigation of complicated surface magnetic configurations. @FootnoteText@ @footnote 1@ S. Blügel, M. Weinert, and P.H. Dederichs, Phys. Rev. Lett. 60, 1077 (1988). @footnote 2@ O. Pietzsch et al., Rev. Sci. Instr. 71, 424 (2000).

4:40pm **MI+NS+NANO 6-TuA9 Ballistic Electron Magnetic Microscopy Studies of Ferromagnetic Films and Tunnel Junctions**, *W.H. Rippard*, *A.C. Perrella*, *R.A. Buhrman*, Cornell University **INVITED**

A new magnetic imaging technique, ballistic electron magnetic microscopy (BEMM), has been developed to study the magnetic structure in ferromagnetic multilayer films and nanostructures as a function of magnetic field  $H$ . In BEMM we exploit the hot electron transport properties of the ferromagnetic films in order to probe their magnetic structure. This technique allows not only the magnetic imaging in applied fields with nm-scale spatial resolution, but also allows the direct investigation of spin dependent transport through the ferromagnetic multilayers. As we are not using a magnetic probe to image these films, we are able to investigate very thin and soft magnetic structures which are the most relevant to technological applications. The magnetization reversal process of both continuous and patterned ferromagnetic films have been investigated. Using a UHV compatible stencil-mask technique, sub-micron structures have been fabricated and imaged with BEMM. In particular, I will discuss the switching behavior of permalloy 'diamonds' and 'rectangles' ( $1.5 \times 0.3$  microns<sup>2</sup>), as well as other shapes of smaller dimension. Using this technique the nanometer scale imaging of tunnel junctions can also be performed. Ballistic current transport through magnetic tunnel junctions will be presented, both in terms of the imaging of 'pin holes' in the junctions and spin-dependent transport through the barrier. The energy dependence of the transport in the ferromagnetic multilayer structures as well as in the tunnel junction systems will also be presented.

**Processing at the Nanoscale/NANO 6**

**Room 302 - Session NS+NANO6+SS+MC-TuA**

**Self-assembly and Self-organization**

**Moderator:** R. Wiesendanger, University of Hamburg, Germany

2:00pm **NS+NANO6+SS+MC-TuA1 Dip-Pen Nanolithography: A New Tool for Generating and Studying Soft Nanostructures**, *C.A. Mirkin*, *S.A. Brennan*, *L.M. Demers*, *S. Hong*, *P.V. Schwartz*, *D.A. Weinberger*, Northwestern University **INVITED**

A new type of ultrahigh resolution soft-lithography, Dip-Pen Nanolithography (DPN) that is interfaceable with biomolecules and biofunctionalized building blocks will be presented. This soft lithography allows one to routinely pattern structures, in serial or parallel fashion, with sub 50 nm spatial and line-width resolution with near-perfect alignment. Implications in materials synthesis, electronics, and biodiagnostics will be discussed.

2:40pm **NS+NANO6+SS+MC-TuA3 A Step Toward Making and Wiring-up Molecular-Scale Devices with a Self-Directed Growth Process**, *G.P. Lopinski*, *D.D.M. Wayner*, *D.J. Moffatt*, National Research Council of Canada; *R.A. Wolkow*, National Research Council of Canada, Canada

Our understanding of and control over molecular adsorption on silicon has advanced very significantly in the last several years. It is now possible to provide a microscopic picture of structure and bonding in covalently attached molecule-silicon surface systems. This detailed understanding of adsorbate-surface structures was entirely lacking when the first wave of enthusiasm for molecular devices crested roughly 20 years ago. While many ideas for molecule-scale devices have been put forward in the past, the tools - both synthetic and analytical - to pursue those ideas did not exist. Now, the control necessary to begin exploring ways to incorporate organic function into existing technologies or, eventually, to make new molecule-scale devices is within reach. @footnote 1@ Experimental and modeling methods have emerged that effectively extend the resolution of STM to see the details of adsorbed molecule structure and bonding. In the next several years it is now realistic to expect structures and concepts dreamed about for decades to begin to be realized. This talk will focus on a self-directed growth process for creating molecular nanostructures on silicon. @footnote 2@ @FootnoteText@ @footnote 1@ Controlled Molecular Adsorption on Si: Laying a Foundation for Molecular Devices, R.A. Wolkow, Annual Review of Physical Chemistry, volume 50, 413-41, 1999. @footnote 2@ Self-Directed Growth of Molecular Nano-Structures on Silicon, G.P. Lopinski, D.D.M. Wayner and R.A. Wolkow, Nature in press.

3:00pm **NS+NANO6+SS+MC-TuA4 Control of Spatial Distribution of Self-Assembled Diacetylene Compounds by Co-deposition with Fatty Acid Molecules**, *Y. Kuwahara*, *G.-M. Zhang*, *J.-W. Wu*, *M. Akai-Kasaya*, *A. Saito*, *M. Aono*, Osaka University, Japan

Control of self-assembled surface structure of functional organic molecules has been attracting intensive interest from a viewpoint of future applications such as novel material structures for nanometer-scale molecular devices. We have investigated self-assembled surface structures of two different chain organic molecules co-adsorbed on HOPG by use of scanning tunneling microscopy. The subject molecule was 10,12-tricosadiynoic acid, which is one of the diacetylene compounds possessing the possibility of being polymerized into macromolecular wire and/or sheet, and several kinds of fatty acids were used as buffer molecules. We used Langmuir Blodgett method for the fabrication of the molecular monolayers. In order to achieve the parallel molecular arrangement, the surface pressure for the deposition was deliberately controlled much lower than the saturate pressure and the substrate was horizontally oriented. A variety of molecular patterns inside the two-component monolayers were revealed, which could be briefly grouped into 'phase separation pattern', where microscopically pure 10,12-tricosadiynoic acid and fatty acid were observable respectively, and 'alternative pattern', in which the lamellae of the two sorts of molecules emerged alternatively. In order to evaluate the mechanism for the two dimensional surface ordering, we have also done the ab-initio molecular orbital calculation and the proposed structural model of the surface self-assembly is in good agreement with the theoretical simulations. Consequently, the possibility of controlling the spatial distribution of the diacetylene compounds on the solid surface has been demonstrated.

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3:20pm **NS+NANO6+SS+MC-TuA5 The Interaction of Metal Atoms with Self-assembled Organic Monolayers**, *A.V. Walker, B.C. Haynie, N. Winograd*, The Pennsylvania State University

Organic monolayers show great promise as materials for a wide range of technological applications. An understanding of the nature of the metal atom - organic monolayer interaction is vital in the development of molecular electronic devices. Recently it was demonstrated that deposited Al atoms can penetrate through an n-alkyl monolayer to the monolayer / Au (111) interface. This phenomenon is believed to occur via thermally activated transient defects in the monolayer. In this paper, we explore the thermodynamics of this system using time-of-flight secondary-ion-mass-spectrometry (TOF SIMS) and demonstrate that at low temperatures the rate Al atom penetration into the monolayer is reduced. We have also studied the interaction between other promising molecular wire candidates and metal atoms.

3:40pm **NS+NANO6+SS+MC-TuA6 Chiral Surface Reconstruction by Large Molecules**, *M. Schunack, L. Petersen, A. Kühnle, E. Laegsgaard, I. Steensgaard, F. Besenbacher*, University of Aarhus, Denmark

Temperature-controlled scanning tunneling microscopy studies provide insight into the bonding, ordering and mobility of large organic molecules at metal surfaces. This is illustrated by investigations of disc-like molecules on a Cu(110) surface with a variable temperature STM, which can be operated down to 25 K. Hexa-tert-butyl decacyclene (HtBDC) self-assembles upon deposition onto Cu(110) above 250 K and forms a double-row structure in two directions surrounded by fast-diffusing single molecules. Nano-manipulation experiments with the STM at low temperatures revealed an underlying chiral reconstruction of the Cu surface. This consists of holes of approximately 14 Cu atoms pulled out of the surface. Surprisingly, the observed reconstruction is chiral. By gently annealing of the molecule structure at higher coverages, large enantiomeric pure domains with two different orientations build up. By means of simple effective medium theory calculations, we estimate the lower bound of the adsorption energy to be  $E_{ad} = 0.45$  eV, and can give a plausible explanation for the observed structure. L. Petersen, M. Schunack et al., submitted to Review of Scientific Instruments.

4:00pm **NS+NANO6+SS+MC-TuA7 From Functionalisation of Single Molecules to Self-organisation of Nano-structured Thin Films**, *Q. Guo*, University of Birmingham, U.K. **INVITED**

The combination of functionalisation of individual molecules with self-organisation of the molecules into functional structures is a promising route for the fabrication of nanoscale electronic and optoelectronic devices. In this talk I will present experimental findings of nanostructured thin films prepared using this method. The dependence of the structure of molecular monolayers on the functionality of individual molecules will be demonstrated using chemisorbed acetate and benzoate species on TiO<sub>2</sub> surfaces as an example. Both acetate and benzoate attach to the substrate through strong bonding between the carboxyl end of the molecules and the metal cations at the surface of TiO<sub>2</sub>. In the case of benzoate, the phenyl ring offers an extra functionality for intermolecular linkage, leading to the formation of dimerised rows of benzoate. Self assembled monolayers (SAMs) of functionalised alkanethiols adsorbed on Au(111) surfaces will also be discussed. Functionalisation of the tail group of thiol molecules gives rise to SAMs with different surface energies, allowing the fine tuning of the reactivity of the surfaces towards binding of deposited atoms and molecules. The formation of nano-particles of gold on carboxyl terminated SAMs has been investigated using scanning tunnelling microscopy (STM) and electron energy loss spectroscopy (EELS) and results will be presented to demonstrate the important role of functionality of individual molecules on nano-structure formation.

4:40pm **NS+NANO6+SS+MC-TuA9 Ion Beam Assisted Self-Organization of Periodic Nanowire-Arrays on CaF<sub>2</sub> Substrates**, *M. Batzill, F. Bardou, K.J. Snowden*, University of Newcastle, UK

The fabrication of well ordered nanowire arrays over large areas is a challenge with many potential applications. Here we report a novel glancing incidence ion beam assisted self-organisation approach to form periodic wire-arrays over large areas on a CaF<sub>2</sub>(111) substrate. Preferential erosion of fluorine by the ion beam creates a surface enriched in calcium. The calcium self-organises in elongated island structures of preferential width and separation. If the sample is irradiated along a fixed azimuth we observe formation of nanowires with ~10 nm periodicity and wire lengths

of at least several micrometers oriented along the azimuthal direction of ion beam incidence. Electrical conductivity measurements reveal a three order of magnitude lower conductivity normal to the wires than along the wires.

5:00pm **NS+NANO6+SS+MC-TuA10 Quantum Engineering of a Pb Nanostructure: Controlling the Thickness with Monolayer Precision**, *C.-S. Jiang, H.-B. Yu, X.-D. Wang, C.-K. Shih*, University of Texas at Austin

We report a novel quantum engineering of Pb mesas on Si(111), designing the quantum number (N) of the electron resonator of Pb by modifying its thickness with monolayer precision. Pb deposition on Si(111) forms mesas on the surface, and the mesas serve as electron resonators because of the strong quantization along the surface normal direction. To modify the mesas into desired thickness, mass-transfer was first triggered by an STM-tip under controlled conditions. The triggering led to the formation of single layer with annular shape at the edge of the mesa by transferring the Pb mass from the wetting layer. Once triggered, the mass transfer from the wetting layer to the top of the mesa continues until the new layer involving millions of atoms is completed. Once this layer is completed, no more mass transfer is observed unless a new triggering is performed. Each triggering leads to addition of one complete monolayer on top of the plateau. Using this process, the Pb mesa thickness can be engineered in a quantized fashion. Detailed mechanisms involved in the engineering will be discussed.

<sup>1</sup> NSTD Student Award Finalist

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6+MC-WeM

### Nanomechanical and Interface Measurements

**Moderator:** R.J. Hamers, University of Wisconsin, Madison

#### 8:20am NS+NANO6+MC-WeM1 Surface Acoustic Wave Investigation by UHV Scanning Tunneling Microscopy, P.U. Voigt, S. Krauß, E. Chilla, R. Koch, Paul-Drude-Institut für Festkörperelektronik, Germany

Recently we have shown that the scanning tunneling microscope can also be used to investigate surface acoustic waves (SAW) of 35 MHz by adding a high frequency sinusoidal signal to the dc tip voltage. @footnote 1@ Due to mixing with the SAW oscillation at the tunneling gap the tunneling current contains both amplitude and phase information of the SAW. Here we report on the first UHV compatible version of this technique including provisions for sample transfer and in situ surface preparation. Since both signal-to-noise ratio and spacial resolution are significantly enhanced, the acoustic oscillation of single atoms and monoatomic steps can be investigated. At present SAW frequencies up to 500 MHz have been successfully fed into the UHV system. @FootnoteText@ @footnote 1@ E. Chilla, W. Rohrbeck, H.-J. Fröhlich, R. Koch, K. H. Rieder, Appl. Phys. Lett. 61, 3107 (1992).

#### 8:40am NS+NANO6+MC-WeM2 Q-Control: Characterizing Highly Sensitive Surface Structures with the AFM, B. Anczykowski, NanoAnalytics WWU Münster, Germany; L.F. Chi, H. Fuchs, Physikalisches Institut WWU Münster, Germany

When operating an atomic force microscope (AFM) in a dynamic mode the oscillation of the cantilever is influenced by non-linear interaction forces between the probing tip and the surface. In principle the instantaneous forces exerted on the sample while scanning the surface can be either repulsive or attractive. Experimental findings and corresponding computer simulations of the tapping mode show that by choosing appropriate system parameters the AFM can continuously be operated in the regime of net-attractive interaction forces. Thereby the risk of modifying the sample surface by the probing tip is minimized. However, in most cases the range in which the system parameters have to be adjusted is rather narrow and therefore a stable operation of the AFM in this interaction regime is difficult to achieve. With the help of the Q-Control module it is possible to reduce the damping of the dynamic system, i.e. to increase the effective quality factor of the oscillating cantilever and thereby to enlarge the regime of net-attractive interaction forces. @footnote 1@ This method allows to minimize the forces exerted by the probing tip on the sample surface. Therefore by applying Q-Control delicate and highly sensitive surfaces, such as ultrathin organic layers or DNA structures, can be characterized with high resolution. @FootnoteText@ @footnote 1@ B. Anczykowski, J. P. Cleveland, D. Krüger, V. B. Elings, and H. Fuchs, Appl. Phys. A 66, S885 (1998).

#### 9:00am NS+NANO6+MC-WeM3 Traceability for Nanoscale Properties, L.P. Howard, J. Pratt, National Institute of Standards and Technology INVITED

The accuracy of nanoscale materials properties measurements ultimately depends upon the accurate determination of many SI units. When measurements are pushed into the nano-scale, many difficult circumstances arise due to what may be effectively described as a poor signal to noise ratio. This talk will highlight work involving the integration of interferometers into scanned-probe microscopes and work in traceable, sub-micronewton force measurements. Interferometry provides us the means to realize the meter. Several applications of sub-nanometer laser interferometry will be presented in the context of scanned-probe microscopes. Atomic lattice spacings have been measured using interferometers, and the expanding role of the atomic lattice in scanned-probe microscope metrology will be explored. Nanonewton force measurements are the subject of a new NIST project with the goal of improving traceability below the micronewton level. The unit of force is derived from the SI base units. Extending force measurement accuracy to the nanonewton level can require a combination of difficult dimensional, mass and electrical measurements. Our development of an electromagnetic balance capable of interfacing to scanned-probe instruments will be highlighted. With this new instrument (and a related electrostatic balance), we will explore the practical limits of using an electrical representation of the newton while attempting to extend our traceability chain back to a purely mechanical realization of force using mass and the earth's gravitational acceleration.

#### 9:40am NS+NANO6+MC-WeM5 Low Temperature Scanning Force Microscopy of the Si(111) 7x7 Surface and Site-specific Measurements of Tip-Sample Interaction Forces, M.A. Lantz, H.J. Hug, S. Martin, A. Abdurixit, A. Baratoff, R. Hoffmann, P. Kappenberger, P.J.A. van Schendel, University of Basel, Switzerland; Ch. Gerber, IBM Research Division, Zuerich Research Laboratory; H.-J. Guentherodt, University of Basel, Switzerland

A low temperature scanning force microscope (SFM) operating in a dynamic mode in ultra high vacuum was used to study the Si(111)7x7 surface at 7.2K. Not only the twelve adatoms but also the six rest atoms of the unit cell are clearly resolved for the first time with SFM. In addition, the first measurements of the short-range chemical bonding forces above specific atomic sites are presented. The data is in good agreement with first-principles computations and indicates that the nearest atoms in the tip and sample relax significantly when the tip apex is within a few Å of the surface. @footnote 1@ New experiments with non-reactive tips reveal atomically resolved images with surprisingly different contrast from those obtained with a reactive tip. Careful analysis of frequency and damping versus distance curves clearly shows the obtained contrast does not result from the formation of a covalent bond, however atomic resolution is still obtained. This strongly suggests that true atomic resolution can be obtained with a new type of tip-sample interaction. The physical nature of this interaction mechanism will be discussed and compared to theoretical models. @FootnoteText@ @footnote 1@ Lantz et al., Phys. Rev. Lett 84, 2642 (2000).

#### 10:00am NS+NANO6+MC-WeM6 Simultaneous STM/nc-AFM Imaging and Force Spectroscopy of Si(100)-(2x1) Surface with Small Oscillation Amplitudes, H.O. Ozer, A. Oral, Bilkent University, Turkey; J.B. Pethica, University of Oxford, UK

We have used a new, fiber interferometer based, high force resolution nc-AFM to image the Si(100)(2x1) surface with atomic resolution, using very small tip oscillation amplitudes down to 0.5 ?pp. The lever is dithered with the small oscillation amplitude at a frequency below resonance and the changes in the oscillation amplitude recorded simultaneously with force gradient and STM topography. With this method we can measure the force gradients quantitatively. Simultaneous images of Force gradient and STM topography have been recorded as a function of tunnel current and bias voltage. The effect of tunnel current and bias voltage on the force gradient contrast will be presented. We have also present force-distance curves between tip and Si(100) surface, measured with sub-Angstrom oscillation amplitudes.

#### 10:20am NS+NANO6+MC-WeM7 A Liquid Helium Temperature Ultrahigh Vacuum Dual-tip Scanning Tunneling Microscope, H. Okamoto, D. Chen, Rowland Institute for Science

A dual-tip scanning tunneling microscope (D-STM) is a powerful instrument for investigating dissipative, diffusive, or ballistic transport phenomena of electrons in nanoscale structures. @footnote 1@ Here we present an ultrahigh vacuum compatible D-STM system working at liquid helium temperature for these new potential applications. Coarse positioning system consists of five rigid and compact inertial steppers, which has mechanical resonant frequency of ~900 Hz after integration. Each stepper has embedded capacitive position sensors with sub-micron resolution. An efficient new method, which we call tri-plane method, is used to navigate the two tips to proximity. The whole D-STM system is installed in a home-made vapor-cooled helium cryostat with a very low evaporation rate of 1.25 liter/day without liquid nitrogen radiation shield. @FootnoteText@ @footnote 1@ Q. Niu et al., Phys. Rev. B 51 5502 (1995).

# Wednesday Morning Poster Sessions, October 4, 2000

## Processing at the Nanoscale/NANO 6

### Room Exhibit Hall C & D - Session NS+NANO6-WeP

#### Poster Session

**NS+NANO6-WeP1 Nanometer Atomic Layer Growth and Removal from Surfaces of Inorganic Single Crystals: Scanning Probe Microscope Studies Under Controlled Solutions**, *J.T. Dickinson, R. Hariadi, S.C. Langford*, Washington State University

Using the tip of a Scanning Force Microscope in solutions with controlled ionic content, we combine the application of single asperity forces to aqueous surfaces of inorganic single crystals. With appropriate combinations of reactivity and stress, we are able to locally remove or deposit crystalline material in atomic layers and dimensions of nanometers. We show for the first time that layer by layer crystal growth can be controlled mechanically in such a fashion that fills in micron-sized features a few atomic layers deep. The recrystallization is done under supersaturated solution conditions. Step edges are stimulated with low contact force scanning using a scanning force microscope (which is also used to image the changes in surface topography). We present recent results on single crystal brushite (CaHPO<sub>4</sub>·2H<sub>2</sub>O) where we find strong dependencies on solution chemistry, mechanical parameters (e.g., normal force), and step crystallography. A model for both material removal and deposition will be presented. These studies allow production of nanoscale structures and atomically flat surfaces under low temperature conditions.

**NS+NANO6-WeP2 Attractive Mode Molecular Manipulation at Room Temperature**, *M.J. Humphry, P.H. Beton, P. Moriarty*, University of Nottingham, UK

We have investigated the manipulation of C60 on Si(100)-2x1 using the tip of a scanning tunneling microscope (STM) operating ultra-high vacuum at room temperature. During repulsive manipulation the tip follows a quasi-periodic trajectory as the molecule is displaced through integer multiples of the surface lattice constant and the tip is partially retracted during the manipulation process. A clear voltage threshold and characteristic tip response for this mode of manipulation is established. In addition molecules are observed to move through a displacement of up to three lattice constants (1.1nm) towards the STM tip due to an attractive interaction. This effect is observed over a wide range of tunneling parameters and is related to 'streaking' in STM images in which molecules may be dragged across the Si surface. The mechanism for this behaviour will be discussed together with the potential for the controlled assembly of molecular nanostructures.

**NS+NANO6-WeP3 Manipulation of Single Cu-TBPP Molecules by Low Temperature STM**, *F. Moresca, G. Meyer, K.H. Rieder*, FU Berlin, Germany; *H. Tang, A. Gourdon, C. Joachim*, CEMES CNRS Toulouse, France

The development of integrated nanoelectronic devices and the requirement of assembling perfectly identical nanostructures has recently focused the research attention on the vast range of possibilities offered by molecular structures. Controlled positioning of individual molecules can be performed by lateral manipulation with the STM tip. STM is in this respect a very promising technique because it contemporarily allows to determine the structure and conformation of the molecules and to build controlled molecular nanostructures without affecting the internal structure of the molecules. A detailed investigation of the processes involved in the manipulation of individual porphyrin-based molecules by scanning tunneling microscopy at low temperature is presented. The molecular conformation on different Cu surfaces is discussed. We show that the molecules occupy different configurations, depending on the interaction with the substrate and on the orientation of the four lateral substituent groups. By means of vertical and lateral manipulation it has been possible to switch between the different conformations and to exploit in detail the interaction between the STM tip and the molecule. Due to the complicated structure of the molecules we have explored different manipulation techniques, exploiting not only constant current but also constant height lateral manipulation.

**NS+NANO6-WeP4 Nanolithography on Silicon Surface using a Tuning-fork STM/AFM**, *S.Y. Lin, S.C. Yang, J.-D. Su*, National Taiwan University; *D.P. Tsai*, National Taiwan University, Taiwan; *F.S.-S. Chien, S. Gwo*, National Tsing-Hua University, Taiwan; *W.-F. Hsieh*, National Chiao-Tung University, Taiwan

A tapping-mode atomic force microscope (AFM)/scanning tunneling microscope (STM) system using a non-optical tuning fork force-sensing method has been developed for the scanning probe lithography. Comparing with the nano-lithography done by the AFM having the conductive cantilever tip, our method has the following advantages: (1) It has longer tapered length STM tip and smaller half cone angle to perform the nanometer scale patterning with high aspect ratio. (2) Its low cost tungsten or Pt/Ir STM tip can be easily fabricated and attached to our AFM force-sensing tuning fork. (3) It can be easily adapted to large-scale parallel processing because of the all-electric force-sensing method. Nanostructures with high aspect ratios and large depths have been successfully performed on the silicon surfaces by using our AFM/STM nano-patterning system followed by the differential etching process. Lines with different widths and matrix of dots with various diameters were demonstrated for potential applications.

**NS+NANO6-WeP5 Etching of GaN (0001) with Halogens: Pit Growth and Step Etching by Cl**, *K.S. Nakayama, S. Kuwano, Q.Z. Xue*, Tohoku University, Japan; *Q.K. Xue*, Chinese Academy of Science; *T. Sakurai*, Tohoku University, Japan

We have studied the etching of GaN (0001) by Cl using scanning tunneling microscopy (STM) to obtain morphological information that can be related to surface reaction and desorption pathway. We used N- and Ga- polar GaN surfaces prepared by N-plasma-assisted molecular beam epitaxy (MBE) followed by Ga deposition. A Ga rich GaN(0001) surface was exposed to molecular Cl at room temperature for the coverage of more than 1 monolayer. Heating to 600-700 °C induced etching by thermal desorption. STM images show that smooth and rough steps appeared and these triangle pits were initiated on the terrace after the thermally activated reaction. The height profiles of a pit and a step in the STM images reveal layer-by-layer removal of GaN. The etching directions of steps can be attributed to the number of dangling bond at the step edges. We also discuss possible desorption pathways with atomic scale including the formation of volatile GaCl<sub>x</sub> products and the spontaneous desorption of nitrogen molecule. When the Ga atom on the top layer removed by Cl, the nitrogen atom in the second layer appeared having two dangling bonds. This configuration would be energetically unfavorable and the distance of dangling bonds of neighboring nitrogen atoms is very close. Therefore, the nitrogen atoms would combine each other to form nitrogen molecules and the spontaneous desorption would occur on the surface.

**NS+NANO6-WeP6 Adsorption of Benzene and Pentacene on Metal Surface: A Scanning Tunneling Microscope Study**, *J.-Y. Park, Y.-J. Song, Y. Kuk*, Seoul National University, Korea

A single benzene molecule is known to have three different shapes in scanning tunneling microscope (STM) image depending on the adsorption sites. The ordered benzene layer and the (3X3) benzene layer coadsorbed with carbon monoxide on Pd(111) are studied with STM. It is revealed that the ordered benzene layer can transform to the disordered layer with temperature or defects on substrates, suggesting the small heat of adsorption. The (3X3) benzene layer coadsorbed with carbon monoxide on Pd(111) is more stable than that without carbon monoxide. Unusual phase boundary was found at every other benzene rows, suggesting elastic strain in the layer. On the other hand, pentacene is known as a candidate for organic device. The adsorption and electronic structure of pentacene, adsorbed on Cu(111) surface, is also investigated with STM. The electronic and geometric contribution to STM image will be discussed with temperature dependent scanning tunneling spectroscopy data.

**NS+NANO6-WeP7 Substrate-Directed Self-Assembly of Rigid Metallo-dendrimers**, *J.C. Poler*, University of North Carolina, Charlotte

The subject of our research is the study of rigid dendrimer molecules, assembled on surfaces. Dendrimers are a class of molecule synthesized from repeating units such that the molecular topology is self-similar. A new class of geometrically rigid metallo-dendrimers has been synthesized recently by MacDonnell et al., and is enantiopure. These multinuclear molecules with Ru<sup>2+</sup> centers are planar with D<sub>3</sub> symmetry. We shall present AFM images of the self-assembled dendrimers on functionalized and patterned surfaces. Because these dendrimers are charged we can direct their self-assembly with electrostatic interactions at the surface.

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Theoretical, molecular mechanics and experimental support for this process will be described.

**NS+NANO6-WeP8 Amino-Terminated Self-Assembled Monolayer on SiO<sub>2</sub> Surfaces Formed by Chemical Vapor Deposition, A. Hozumi,** National Industrial Research Institute of Nagoya, Japan; **H. Sugimura,** Graduate School of Nagoya University, Japan; **Y. Yokogawa,** National Industrial Research Institute of Nagoya, Japan; **K. Hayashi,** Graduate School of Nagoya University, Japan; **T. Kameyama,** National Industrial Research Institute of Nagoya, Japan; **O. Takai,** Graduate School of Nagoya University, Japan

In order to immobilize polymers, neurons, DNA and proteins etc., amino-terminated self-assembled monolayers (SAMs) have been applied. Although chemisorption of aminosilanes at the solid/liquid interface were studied extensively, there have been few reports on preparing aminosilane SAMs from vapor phase. Here, we report on the formation of an amino-terminated SAM on SiO<sub>2</sub>/Si sample substrates on the basis of chemical vapor deposition (CVD). Cleaned SiO<sub>2</sub>/Si substrates were exposed to vapor of aminosilane, that is, N-(6-aminohexyl)-aminopropyltrimethoxysilane (AHAPS) diluted with dehydrate toluene. Following CVD, these samples were washed by a successive immersion in dehydrate ethanol, dehydrate toluene, NaOH (1mM) and HNO<sub>3</sub> (1mM). This washing process was repeated several cycles. Finally, they were rinsed with MilliQ water and then blown dry with a N<sub>2</sub> gas stream. We have investigated in detail chemical and electrokinetic properties of this amino-terminated SAM. The SiO<sub>2</sub>/Si surface after CVD became hydrophobic showing a water-contact angle of ca. Thickness of the AHAPS-SAM was ca. 1.3 - (±)0.1 nm as estimated by ellipsometry. This value is comparable to the theoretical molecular length of AHAPS, i.e., 1.4 nm. As confirmed by AFM, the surface was very smooth and homogeneous with its roughness being almost identical to that of the SiO<sub>2</sub>/Si substrate. Zeta-potentials of the AHAPS-SAM covered SiO<sub>2</sub>/Si substrate were measured as a function of pH by means of an electrophoretic light scattering spectrophotometer. The surface was charged positively below its isoelectric point of pH 7-8, since the amine groups are easily protonated. Furthermore, we demonstrated micropatterning of the AHAPS-SAM based on the photolithography using an excimer lamp radiating vacuum ultra violet light of 172 nm in wavelength.

**NS+NANO6-WeP9 Formation of Ferroelectric Nano-domains using Scanning Force Microscopy for the Mass Storage System, H. Shin,** Samsung Advanced Institute of Technology & CRI, Korea; **J.G. Shin, S. Hong, J.U. Jeon,** Samsung Advanced Institute of Technology, Korea; **J. Woo, K. No,** Korea Advanced Institute of Science and Technology

Applying voltage between the conductive tip in atomic force microscope (AFM) and Pb(Zr,Ti)O<sub>3</sub> (PZT) films can cause the switching of ferroelectric domains in nanometer scale. Ferroelectric domains (less than 100 nm in diameter) were induced and analyzed. Imaging of ferroelectric domains was achieved by piezoresponse of the PZT films in contact mode of AFM with lock-in amplifier. De-convolution of the first harmonic signal from the lock-in amplifier reveals the details of the formation of nano domains in ferroelectric thin film materials. Formation and imaging of ferroelectric domains in nano size can be applicable to develop the future mass storage system with miniaturization and integration of the AFM through MEMS technology. In this paper, relevant issues, i.e. bit (induced ferroelectric domains) size dependence on poling voltage, pulse width, and film's thickness and microstructure as well as retention characteristics of the induced bits are discussed. In detail, the bit size showed a log-linear dependence on the pulse width and a linear dependence on the pulse voltage. Using the calculation of electric field distribution the size of the induced bits under certain pulse voltage and width was estimated and confirmed by the experiments. As a result it is clear that the thinner films are beneficial to induce smaller and more stable bits under the same poling voltage and pulse width. In addition retention loss phenomena of the induced ferroelectric domains were observed and carefully investigated. The retention loss can be described by an extended exponential decay which implies a narrow distribution of the relaxation times of the domains. Characteristic relaxation time was largely dependent upon poling time, domain size as well as film's microstructures. Finally, an effective way to improve retention failure of the induced ferroelectric domains was proposed and confirmed by experiments.

**NS+NANO6-WeP10 Nanostrands of Poly(carbamatepropylsiloxane) on SiO<sub>2</sub> Observed with Atomic Force Microscopy, H. Celio, H. Cabibil, J. Lozano, J.M. White,** University of Texas at Austin

Poly(organosiloxane) polymers have recently received considerable attention due to their potential use as dielectric materials in advanced microelectronic applications. Using atomic force microscopy (AFM) and various spectroscopic methods (XPS, IR, Raman, SIMS), we have investigated the formation of linear strands of poly(carbamatepropylsiloxane) polymer (CPS) of nanoscale dimensions supported on native silicon oxide of Si(100). The CPS nanostructures were fabricated by the hydrolysis of  $\gamma$ -aminopropylethoxysilane ( $\gamma$ -APS) in a basic aqueous solution containing 3  $\gamma$ -APS: 1 K<sup>+</sup> and excess CO<sub>2</sub>. The CPS is deposited on SiO<sub>2</sub> by a spincoating procedure. The CPS nanostrands have lengths greater than 1000 nm, heights between 2 to 3 nm and widths on the average of ~10 nm. These strands are stable under ambient conditions for weeks. The linear directionality of these structures is dependent on the following parameters: 1) K<sup>+</sup> cations, 2) strong hydrogen bonding interactions between propyl-NHCOO- and -NH groups. The former is responsible for the formation of linear Si-O-Si backbone and neutralization of the carbamate (NHCOO-) group, while the latter is responsible for the self-assembly of CPS strands.

**NS+NANO6-WeP11 Solution Deposition and Surface Characterization of Supported Gold Clusters, C.C. Chusuei, X. Lai, K.A. Davis, P.S. Bagus, D.W. Goodman, M.A. Omary, M.A. Rawashdeh-Omary,** Texas A&M University

Nanosized Au cluster agglomeration has been a long standing problem in the preparation of planar model catalyst systems. A novel Au/TiO<sub>2</sub> catalyst preparation method was investigated. Six-atom gold clusters (in the form of a Au<sub>6</sub>(PPh<sub>3</sub>)<sub>3</sub>) colloidal suspension) was deposited onto a TiO<sub>2</sub>(110) single crystal via CH<sub>3</sub>Cl solution. Scanning tunneling microscopy (STM) and spectroscopy (STS) revealed structures consistent with unagglomerated, single unit entities. Electron stimulated desorption (0.12 Coulombs/cm<sup>2</sup>) was then performed. X-ray photoelectron spectroscopy, high resolution electron energy loss spectroscopy (HREELS) and STM showed evidence for removal of the triphenyl phosphine ligands. Band gap measurements from HREELS, STS and theoretical calculations suggest that metal/metal oxide support interactions affect the Au electronic structure.

**NS+NANO6-WeP12 Optical and Structural Properties of Ball Milled Produced Small Si Particles Embedded into a Sol-Gel Matrix, F.J. Espinoza-Beltrán, L.L. Díaz-Flores, J.M. Yañez-Limón,** CINVESTAV-IPN, México; **J. Morales-Hernández,** Programa de Posgrado de Ingeniería, UAQ, México; **A. Mendoza-Galván,** CINVESTAV-IPN, México; **J. González-Hernández,** CINVESTAV-IPN, México, Mexico

Nano and micrometric silicon particles have been introduced into SiO<sub>2</sub> matrix produced by the sol-gel method, using tetraethyl-orthosilicate. The small Si particles were produced by grinding 99.999 % pure silicon granular polyfine in a low energy ball mill. The milling process, for various periods of time, was carried out in air conditions. For that reason, the small particles consist of a Si core surrounded by a SiO<sub>2</sub> cap layer. According to infrared and Raman measurements, the Si core contains molecular structures of the form Si<sub>n</sub>, where n is in the range of 6 to 7. From the position of TO IR active mode the value of x is estimated to be around 1.7. The silicon oxide cap also shows a strong LO mode, normally not observed at normal incidence in the IR absorption of planar SiO<sub>2</sub> thin layers. An IR line at about 630 cm<sup>-1</sup>, also indicates that during the milling process, some atomic hydrogen is attached to the silicon particles. Sol-gel layers, with various Si particle sizes and various particle densities were prepared. The structural and optical properties of the as-prepared and heat treated, in the range of 100 to 800°C, powder and film samples were studied. The photoluminescence emission and optical absorption of the Si particles are related with their size and concentration.

**NS+NANO6-WeP13 Fabrication of a Si Nanosize Pillar Array using Modified He/Cl<sub>2</sub> Plasma Etching Process, J.W. Kim, M.Y. Jung, D.W. Kim, S.S. Choi,** Sun Moon University, Korea

We have studied the effect of He flow rate on the dry etching process of Si and SiO<sub>2</sub> layers using chlorine-based plasma. Experiments were performed using reactive ion etching system in order to fabricate a nanosize Si structure. The ion damage on the etch mask during etching process would hinder deeper and controllable etch profile. Rather than Ar gas, a He feed gas with Chlorine etch gas was exploited in order to reduce the ion damage. We have examined various etching characteristics of the He/Cl<sub>2</sub>

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plasma and applied to fabrication of a Si pillar arrays. In this study, the fabrication of the Si pillar array with 5 micrometer height was successfully fabricated with 500 nm thickness electron beam resist only. We also fabricated the 10 micrometer deep Si trench with this modified He/Cl<sub>2</sub> reactive ion etching system.

**NS+NANO6-WeP14 A Novel Low Temperature Synthesis Route for Silicon Nanowires, S. Sharma, M. Sunkara, University of Louisville; E.C. Dickey, University of Kentucky; R. Miranda, University of Louisville**

In this paper, we present a novel synthesis route for growing silicon nanowires at temperatures lower than the temperatures required for traditional vapor-liquid-solid (VLS) approaches that employ transition metals as catalysts. In the present work, gallium, which has low melting temperature (~30 °C) and broad temperature range for the melt phase (30-2400 °C at 1 atm), was used as a catalytic media for the low-temperature synthesis of silicon nanowires. Growth of silicon fibers was observed when silicon substrates covered with a thin film of liquid gallium, were exposed to a mixture of nitrogen and hydrogen in a microwave-generated plasma. The resulting silicon wires ranged from several microns to less than ten (10) nanometers in diameter. The observed growth rates were on the order of 100 microns/hr. Results indicate that this technique is capable of producing oriented rods, whiskers and with reasonable size distribution. We will present results showing the crystallinity, composition, patternability, and role of gas phase composition, obtained when using this technique. The growth mechanism in this method is hypothesized to be similar to that in other VLS processes, i.e., rapid dissolution of silicon hydrides in gallium melt, which catalyzes subsequent precipitation of silicon in one dimension in the form of wires. We believe that this technique offers several advantages over the conventional VLS technique using silicon-gold eutectic for catalyzed growth. In this technique, there is no need to supply silicon through the gas phase. Secondly, this technique in principle can operate at very low temperatures (<400 °C) thus allowing easier integration with other processing techniques and materials involved in electronics and optoelectronic device fabrication. Nanometer scale one-dimensional silicon structures such as nanowires and nanowhiskers are expected to be critically important in the future mesoscopic electronic and optical device applications.

**NS+NANO6-WeP15 Fabrication of a Nanosize Oxide Aperture Array Coated with Thin Metal Films, J.W. Lee, J.W. Kim, M.Y. Jung, D.W. Kim, S.S. Choi, Sun Moon University, Korea**

There have been considerable interests about the fabrication of the nanosize hole due to the potential application of the near field optical sensor or liquid metal ion source. The 2 micron size dot array was initially patterned. After formation of the V-groove shape by anisotropic etching, dry oxidation was followed. In this procedure, the orientation dependent oxide growth was performed to have an etch-mask for dry etching. The reactive ion etching by the inductively coupled plasma system was performed in order to etch 90 nm silicon dioxide layer at the bottom of the V-groove and to etch the Si at the bottom. The negative ion energy on the bottom substrate would enhance the anisotropic etching by the Cl<sub>2</sub> gas. After etching, the remaining thickness of the oxide on the Si(111) surface was measured to be 130 nm by scanning electron microscopy. After bulk micromachining from the backside Si wafer, the etched oxide aperture will be coated with metal thin film in order to improve the light transmittance efficiency.

**NS+NANO6-WeP16 Resolution Enhancement in Kelvin Probe Force Microscopy, R. Shikler, O. Kozael, Y. Rosenwaks, Tel-Aviv University, Israel**

Kelvin probe force microscopy (KPFM) has become in recent years a valuable tool for characterizing and analyzing semiconductor surface electronic properties with nanometer resolution. The Kelvin probe force microscope measures the semiconductor work function by nullifying the electrostatic force between a vibrating tip and the semiconductor surface. It is accepted that the finite tip size in scanning probe microscopy can have a profound effect on the obtained topography image. This phenomenon is enhanced in KPFM measurements because the electrostatic force is long range, hence introducing topographic artifacts into the contact potential difference image. We propose an algorithm that improves the lateral resolution and reduces the topographic artifacts of the contact potential image by taking into account the full tip shape and the semiconductor surface. By using the real tip shape and sample topography (obtained using conventional blind tip estimation and dilation algorithms), the true contact potential difference image can be deconvoluted. A comparison of measured and deconvoluted KPFM images is presented, and the limits of the method are discussed.

**NS+NANO6-WeP17 Effect of Beam Parameters in Electron Beam Induced Deposition of Rhodium from a Carbon Free Precursor: A Systematic Study, F. Cicoira, I. Utke, P. Hoffmann, B. Dwir, K. Leifer, E. Kapon, D. Laub, H.J. Mathieu, Swiss Federal Institute of Technology Lausanne, Switzerland; P. Doppelt, Ecole Superieure de Physique et Chimie Industrielle, Switzerland**

Electron-beam induced deposition (EBID) offers unique advantages over classical resist-based processes, like the capability of depositing in situ conducting or dielectric materials, high aspect ratio supertips, air-bridges and other three dimensional structures. In our deposition system, based on a Cambridge S100 SEM with a thermionic tungsten filament, EBID is obtained by decomposing with the focused electrons a metal precursor directed to the sample surface by an internal needle. Carbon-free precursors are used to improve the purity of the EBID metal containing deposits. The inorganic precursor [RhCl(PF<sub>6</sub>)<sub>3</sub>·2H<sub>2</sub>O] allowed the deposition of nanocrystalline Rh containing supertips and nanowires with diameters down to 200 nm. Auger Electron Spectroscopy (AES) measurements show that the Rh deposits contain up to 60% of Rh. The rest of the deposit consists in P, Cl, N and O; no carbon could be detected after removal of the contamination layer. These results are confirmed by TEM investigation, revealing that the deposits have a crystalline structure and are covered by an amorphous 10-20 nm thick shell. TEM images allow also the determination of the Rh cluster size, which depends on the beam parameters used to induce the deposition and show that crystalline lines and tips with high Rh content can be obtained even when low deposition currents (some pA) are applied. In this work, we present a systematic study of the effect of the beam parameters on the properties of the deposits. Different two and three-dimensional structures have been deposited from the same precursor varying the electron energy, the electron current and the scanning speed of the electron beam. The obtained deposits have been characterized by Auger Electron Spectroscopy and Transmission Electron Spectroscopy to determine the chemical composition and the nanostructure for every set of parameters.

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6-WeA

### Nanoscale Modification of Materials

**Moderator:** R.E. Palmer, The University of Birmingham, U.K.

2:00pm **NS+NANO6-WeA1 Selective-Area Chemical Vapor Deposition Using AFM-Patterned Silicon Nitride Growth Mask, S. Gwo, S.-W. Lin, Y.-C. Chou, T.T. Chen**, National Tsing-Hua University, Taiwan; **T. Yasuda, S. Yamasaki**, Joint Research Center for Atom Technology (JRCAT), Japan; **T.-S. Chao**, National Nano Device Laboratory, Taiwan

Silicon nitride (Si@sub 3@N@sub 4@) is a very robust material against oxidation and etching and is typically used as an oxidation mask. Here we report atomic force microscope (AFM)-based local oxidation of Si@sub 3@N@sub 4@ and its applications in nanofabrication. Owing to very large etch and growth selectivities among Si@sub 3@N@sub 4@, SiO@sub 2@, and Si, AFM nanolithography using Si@sub 3@N@sub 4@-mask could be used to both "subtractive" (selective-area anisotropic etching of underlying substrate) and "additive" (selective-area chemical vapor deposition) fabrication processes of nanostructures through the openings on the Si@sub 3@N@sub 4@ resist mask. Using this AFM-patterning method combined with a novel design of bilayer growth mask, which is entirely compatible with the existing microelectronic processes, synthesis of ultrahigh packing density and ordered nanostructure becomes readily achievable.

2:20pm **NS+NANO6-WeA2 Fabrication of Nanoscale Templates by Chemical Lithography, A. Götzhäuser, W. Geyer, V. Stadler, W. Eck, M. Grunze**, Universität Heidelberg, Germany; **T. Weimann, P. Hinze**, Physikalisches Technische Bundesanstalt, Germany; **K. Edinger**, University of Maryland

Nanostructure fabrication requires precise lithographic tools and smart materials that can be modified in a controlled manner. Recently, we discovered that self-assembled monolayers of biphenylthiols are crosslinked by electrons and can be utilized as negative resists. Crosslinked biphenyls can also be applied as stabilizing spacer groups during modifications via chemical lithography, specifically the conversion of NO@sub 2@ to NH@sub 2@ end groups. Based on these findings, we fabricated templates by high resolution electron beam lithography. We used a Leica LION LV 1 system at a beam energy of 2.5 keV and doses between 10 and 50 mC/cm@super 2@. We also applied a simple and versatile projection technique using FIB structured stencil masks and low energy (50eV) electrons. The templates were characterized by contact and lateral force AFM. The finest structures have lateral dimensions of ~20nm. We show that they can be used for a laterally controlled molecular deposition. @FootnoteText@ @footnote 1@ W. Geyer, V. Stadler, W. Eck, M. Zharnikov, A. Götzhäuser, M. Grunze, Appl. Phys. Lett. 75, 2401 (1999). @footnote 2@ W. Eck, V. Stadler, W. Geyer, M. Zharnikov, A. Götzhäuser, M. Grunze, Adv. Materials, in press.

2:40pm **NS+NANO6-WeA3 National Nanotechnology Initiative: Overview, M. Roco**<sup>1</sup>, The National Science Foundation  
**INVITED**  
PLEASE SEND US AN ABSTRACT. Thank you.

3:20pm **NS+NANO6-WeA5 Electronic Conductivity and Thermoelectric Measurements of Bismuth Nanoline Structures, K. Miki, A. Yamamoto, K. Sakamoto**, Electrotechnical Laboratory, Japan

Using the newly discovered characteristic structure of Bi perfect lines in Si(001) terraces, we have fabricated two new nanostructures of bismuth in silicon epitaxial layers and measured both their electronic and thermoelectric properties. Atomically perfect bismuth lines form in flat Si(001) surfaces around the temperature at which most of the bismuth desorbs from bismuth epitaxial layers. The lines are 1 nm wide and can be hundreds of nm long without kinks or other defects. In order to form multiple layers containing lines, segregation of the bismuth during subsequent silicon deposition was suppressed by further deposition of bismuth. In this way we fabricated three layers of Bi lines with undoped Si spacer layers 62 nm thick on an SOI (silicon on insulator) substrate, then capped it with 20 ML of silicon. We also fabricated a Bi delta doped structure: we intentionally destroyed most of the whole line structure by incompletely covering the surface with Bi and then heating the sample around 1100 degree C for 20 minutes. In this way we made five delta

doped layers, separated by undoped Si spacer layers 20 nm thick. The layered structures were patterned into 1 micron x 100 micron bar-shaped devices through reactive ion etching, and metal contacts were made for transport measurements. By isolating the device on silicon oxide we eliminated electric leakage. The electronic measurements show that the delta doped structure is n type whereas the buried Bi line structure has metallic conductivity. We applied a temperature gradient to the buried Bi line structure along sample length of 100 micrometres, and observed a thermoelectric voltage linearly proportional to the temperature difference, of the order of 100 microvolts.

3:40pm **NS+NANO6-WeA6 Quantum-wire Arrays Fabricated by a High-pressure High-temperature Injection Process, T.E. Huber**, Howard University; **M.J. Graf**, Boston College; **C.A. Foss Jr.**, Georgetown University

Three-dimensional arrays of metals and semiconductor ultrafine wires can be synthesized by injecting its liquid melt into a porous anodic aluminium oxide (PAAO) template. This is a highly effective technique with a resolution that exceeds 10 nm in many cases. Nanowire arrays are attracting a great deal of attention because of their potential applications in electronics and optics and promise for studying quantum confinement effects. The semimetal Bi is unique because, due to its extremely small effective mass, its Fermi wavelength is long, 25 nm. Therefore, quantum confinement effects should be readily observed in nanowires whose diameter are below ca. 25 nm. Large area (2 mm x 2 mm) arrays of parallel wires of Bi with diameter as small as 20 nm, lengths of 30 - 50 mm, and packing density as high as 10@super 11@ cm@super -2@ have been fabricated. The nanowires are essentially single crystalline and oriented. We have found a resistivity enhancement and a very large positive magnetoresistance. For small diameter wires a resistance maximum is observed. The nanowires contact resistance and doping effects are discussed. The experimental results are discussed in terms of the semimetal-to-semiconductor transition, classical size effects and mesoscopic phenomena. Bulk Bi and Bi-Sb alloys are the best thermoelectric materials at 77 K; the nanowire array composites, especially the 1D Bi quantum wire systems, are expected to show improved properties.

4:00pm **NS+NANO6-WeA7 Transport Study of Single Bismuth Nanowire Fabricated by Silver and Silicon Nanowire Shadowmask, S. Choi, M. Leung, G. Stupian, N. Presser**, The Aerospace Corporation; **S. Chung, J. Heath, A. Khitun**, University of California, Los Angeles; **A. Balandin**, University of California, Riverside

We have carried out measurements of the electrical conductivity of a single bismuth nanowire fabricated by low energy electron beam lithography using silver/silicon nanowire shadowmasks. The nanowires examined have characteristic dimensions slightly below the critical diameter (about 50nm) at which a semimetal to semiconductor phase transition was predicted to occur. Our results reveal a semiconductor-like temperature dependence of the electrical conductivity of a bismuth nanowire which is strikingly different from the bulk dependence. We have also developed a theoretical model which adequately describes the dependence of the electrical conductivity and energy band gap on the diameter of bismuth nanowires and other parameters. The experimental data presented may be crucial for suggested thermoelectric application of bismuth nanowires.

4:20pm **NS+NANO6-WeA8 Conductance Anisotropy in a Mesoscopic Array of Atomic Wires: Ga/Si(112)@footnote 1@, K.J. Yoo, S. Tang**, University of Tennessee; **P.T. Sprunger**, Louisiana State University; **H.H. Weitering**, University of Tennessee

In recent years, researchers at the Naval Research Laboratory have used the Si(112) surface as a template for the fabrication of a mesoscopic array of single-atom-wide gallium wires or quantum wires. @footnote 2@ The structural uniformity of the wire array appears far superior to those created by nanolithographic methods. We have characterized this Ga/Si(112) hetero-structure with Scanning Tunneling Microscopy, Angle-Resolved Photoelectron Spectroscopy (ARPES), and Si 2p core-level spectroscopy. The electrical conductivity of the wire-array was measured as a function of temperature in ultrahigh vacuum using the four-point-probe technique parallel and perpendicular to the quantum wires. The parallel conductivity has a temperature-dependence characteristic of a semiconductor. In contrast, the conductivity perpendicular to the wires appears metallic. This unexpected result can be understood on the basis of first-principles calculations by Flores and coworkers in Madrid @footnote 3@, which indicate that the band effective mass in the perpendicular direction is close to the free electron mass ( $m^* = 1.3 m_{\text{e}}$ ) whereas the effective mass in the parallel direction is very large ( $m^* > 5.5 m_{\text{e}}$ )

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e@). The main features of the theoretical surface-state band structure have been confirmed with ARPES. Contributions to the electrical conductivity by the space charge layer beneath the surface have been calculated based on core level measurements of band bending. These contributions were subtracted to determine the electrical conductance solely through the quantum wires. @FootnoteText@ @footnote 1@Work supported by National Science Foundation(DMR-9705246). @footnote 2@A. A. Baski, S. C. Erwin, and L. J. Whitman, J. Vac. Sci., B14(2), 992(1996). @footnote 3@F. Flores, in private communication.

4:40pm **NS+NANO6-WeA9 Time Evolution of Ag Nanowires Grown on Ag/GaAs(110) Surfaces**, *H.-B. Yu, C.-S. Jiang, X.-D. Wang, C.-K. Shih*, University of Texas at Austin

We have studied an unusual 'shrink-expand' behavior of Ag nanowires grown on Ag films using scanning tunneling microscope (STM). Ag films were prepared using low temperature deposition onto UHV in-situ cleaved GaAs(110) surfaces, followed by room temperature annealing. The film thus formed, below or around 6 ML, are atomically flat with voids distributed in the film. After the film becomes smooth, atoms inside the voids continue to transfer to the top Ag surface and form nanowires. Some of the nanowires have aspect ratios greater than 100:1. The nanowire length growth was monitored with STM, showing linear growth rate with time, while keeping the same width. After growing to certain length, the wires start to shrink, and at the same time, their widths expand by about 1.4 nm. After this shrinking process, the Ag wires continue to grow linearly in time before it takes on another 'shrink-expand' process. This 'shrink-expand' behavior can be observed on nanowires with no voids surrounded, but also on wires that are approaching the existing void edge, which indicating the repulsive interaction of nanowires with the edge of the voids. Detailed mechanism of this nanowire shape transition will be discussed.

5:00pm **NS+NANO6-WeA10 Ion Implanted Contacts to Nanostructures and Metallic Monolayers on Clean Surfaces**, *J.W. Nolan, B.N. Cotier, M.J. Butcher, P.H. Beton, P. Moriarty, M.R.C. Hunt, A. Neumann*, University of Nottingham, UK; *V.R. Dhanak*, Daresbury Laboratory, UK; *A. Gundlach*, Edinburgh University, UK; *S. Thoms*, University of Glasgow, UK

Ion implanted (As) contact tracks with separations of order 200nm and depths ~20nm are formed in p-Si/SiO<sub>2</sub> wafers. These tracks are investigated using scanning tunnelling microscopy (STM) in an ultra-high vacuum environment following removal of the SiO<sub>2</sub> layer using a combination of wet etching and high temperature vacuum annealing in the range 600-1000oC. The ion implanted regions are activated by this anneal and are slightly depressed (by ~5nm) as compared with the surrounding surface but co-exist with the Si(100)-2x1 and Si(111)-7x7 surface. Core level photoemission studies of control samples which have been uniformly implanted show that As desorbs from the near surface region for temperatures >900oC, the temperature at which the oxide layer is thermally desorbed, but this leads to an insignificant change in resistance of the tracks. The ion implanted regions form high impedance reverse biased p/n junctions with a p-type substrate and adjacent implanted tracks may be shorted by the deposition of thin metallic films. The shorting resistance of these structures has been measured using an in-situ electrical probe and results are correlated with the morphology of the metallic layer. For samples annealed below 900oC the oxide layer is not desorbed and a shorting resistance ~20kOhms is observed for a Ag film of thickness 0.5nm. At higher annealing temperatures this shorting resistance is much higher due in part to As desorption and in part the island morphology of the Ag film. The process is compatible with H termination and de-passivation and we report results on metallic adsorption on de-passivated wires ~1-5nm wide.

## Biomaterial Interfaces

### Room 202 - Session BI+NS-ThM

#### Nanoscale Biology

Moderator: J.J. Hickman, Clemson University

#### 8:20am BI+NS-ThM1 Engineering Life into Nanofabricated Systems, C.D. Montemagno, Cornell University

INVITED

Scientists and engineers have anticipated the potential benefits of integrating engineered devices to living systems at the molecular level for many years. Hybrid systems can potentially possess many of the essential properties of life such as the abilities to "intelligently" self-assemble, repair, and evolve. We will present the results of our efforts to incorporate biological energy transduction processes and cell signaling pathways into engineered nanofabricated devices. In particular, we will illustrate our strategy for fueling, controlling and integrating a F1-ATPase biomolecular motor with a NEMS to create an engineered hybrid device. Included in the presentation will be the initial results of our efforts to develop and demonstrate an integrated F1-ATPase powered NEMS device that is fueled by light-driven ATP production. ATP is synthesized from light using artificial liposomes comprised of reconstituted FoF1-ATP synthase and bacteriorhodopsin. Subsequently, the ATP provides energy to power a recombinant, thermostable F1-ATPase biomolecular motor that is coupled to a NEMS device. We will also present our technique for integrating nanomechanical structures to biomolecular motors with a precision of 40 nm. This work capitalizes on a core feature of living systems: the capability of transforming diverse sources of energy into a generic energy currency that can be universally used. The integration of a synthetic photosynthetic system with NEMS establishes a new mechanism for fueling the next generation of nanoelectromechanical devices. Light is used to produce ATP from ADP and P, the ATP is used by the F1-ATPase biomolecular motor to produce work with ADP and P as waste products. Ultimately, we anticipate that this chemically closed system will be used to pump fluids, open and close microvalves, provide locomotion, generate electricity, and make way for "Smart Dust" applications such as long-lived microscopic intelligence and environmental sensors.

#### 9:00am BI+NS-ThM3 Powering Molecular Shuttles through an Artificial Photosynthetic System, V. Vogel, H. Hess, University of Washington; K. Jardine, Arizona State University; J. Clemmens, University of Washington; T.A. Moore, A.L. Moore, A. Primak, Arizona State University; J. Howard, University of Washington; D. Gust, Arizona State University

The ultimate goal for bioengineers is to be able to engineer systems on a nanoscale as perfect as nature does in cells. Great progress has been made in recent years in biochemistry and biophysics, supplying us with information about the construction principles as well as the details of many cellular subsystems. This information is matched by recent advances in nanotechnology, allowing control of the arrangement of biomolecules on a sub-micron scale. From an engineering point of view the construction of artificial systems, performing different tasks related to the cellular environment, becomes possible. Examples of this approach are the construction of artificial photosystems, consisting of vesicles doped with antenna molecules, proton pumps and the enzyme ATPase, and the construction of "molecular shuttles", microtubules moved by motor proteins on a patterned surface. The arising challenge is to combine these subsystems into a larger, more complex system with extended functionality. Here we present a proof-of-principle experiment demonstrating the integration of a transport system (the "molecular shuttles") with a system providing chemical energy from light (the above mentioned artificial photosystem). In the integrated system we can therefore nonintrusively control the motion of the microtubules through light. The experimental setup consists of a flow cell mounted on an epi-fluorescence optical microscope and illuminated by a laser diode. The surface of the flow cell was patterned with parallel grooves spaced between 30 nm and 1  $\mu$ m apart by shear-deposition of a teflon film. The motor protein kinesin adsorbed preferentially along the grooves providing "tracks" for the motion of the microtubules. The microtubules were fluorescently labeled and bound to the motor proteins in the absence of ATP. The ATP-generating vesicles floated freely in the buffer solution. Illumination of the sample with light absorbed by the vesicles as followed by motion of the microtubules. The motion was mainly directed along the direction of shear of the underlying teflon film. This experiment thus demonstrated that in an integrated system, multiple self-assembled entities cooperate functionally all the way from light harvesting through charge separation across a lipid membrane and ATP-synthesis driven by a proton gradient to ATP-fueled

conformational changes of kinesin leading to directed motion of microtubules on uniaxially aligned kinesin tracks. @FootnoteText@ @footnote 1@ Gust, D., T.A. Moore, and A.L. Moore, Mimicking bacterial photosynthesis. *Pure & Appl. Chem.*, 1998. 70(11): p. 2189-2200. @footnote 2@ Steinberg-Yfrach, G., et al., Light-driven production of ATP catalysed by FOF1-ATP synthase in an artificial photosynthetic membrane. *Nature*, 1998. 392(6675): p. 479-82. @footnote 3@ Dennis, J.R., J. Howard, and V. Vogel, Molecular shuttles: directing the motion of microtubules on nanoscale kinesin tracks. *Nanotechnology*, 1999: p. 232-236. @footnote 4@ Service, R.F., Borrowing from biology to power the petite: nanotechnology researchers are harvesting molecular motors from cells in hopes of using them to drive nano-scale devices. *Science*, 1999. 283: p. 27-28. @footnote 5@ Wittmann, J.C. and P. Smith, Highly oriented thin films of poly(tetrafluoroethylene) as a substrate for oriented growth of materials. *Nature*, 1991. 352: p. 414-417. @footnote 6@ Howard, J., A.J. Hudspeth, and R.D. Vale, Movement of microtubules by single kinesin molecules. *Nature*, 1989. 342: p. 154-158.

#### 9:20am BI+NS-ThM4 Unbinding Process of Adsorbed Proteins under External Stress Studied by AFM Force Spectroscopy, C. Gergely, J. Voegel, INSERM, France; P. Schaaf, Institut Charles Sadron (CNRS) Strasbourg, France; B. Senger, INSERM, France; J.K.H. Horber, EMBL Heidelberg, Germany; J. Hemmerle, INSERM, France

We report the study of the unbinding process under a force load  $f$  of adsorbed proteins (fibrinogen) on a solid surface (hydrophilic silica) by means of AFM force spectroscopy. By varying the loading rate  $r$ , defined by  $f=r \cdot t$ ,  $t$  being the time, we find that, as for specific interactions, the mean rupture force increases with  $r$ . This unbinding process is analysed in the framework of the widely used Bell model. Thus typical dissociation rate at zero force entering in the model lies between 0.02 and 0.6 1/s. Each measured rupture is characterized by a force  $f_0$  which appears to be quantized in integer multiples of 180-200 pN.

#### 9:40am BI+NS-ThM5 Single-Molecule Protein-Ligand Bond-Rupture Forces Measured Using the Poisson Atomic Force Method, Y.-S. Lo, Y.J. Zhu, J.D. McBride, T.P. Beebe, Jr., University of Utah

INVITED

It is known that bond strength is a dynamic property that is dependent upon the force loading rate applied during the rupturing of a bond. For biotin-avidin and biotin-streptavidin systems, dynamic force spectra, which are plots of bond strength vs.  $\ln(\text{loading rate})$ , have been acquired in a recent biomembrane force probe (BFP) study [Merkel et al., *Nature* 397 (1999) 50] at force loading rates in the range of 0.05 to 60,000 pN/s. In the present study, the dynamic force spectrum of the biotin-streptavidin bond strength in solution was extended from loading rates of  $\sim 10$  to  $\sim 10^9$  pN/s with the atomic force microscope (AFM). The Poisson AFM statistical analysis method was applied to extract the magnitude of individual bond-rupture forces and non-specific long-range interactions from the AFM force-distance curve measurements. In addition, surface characterization methods for the analysis of protein-coated surfaces and AFM tips, both imaging and spectroscopic x-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) will be discussed. The AFM bond strengths were found to scale linearly with the logarithm of the loading rate in two regimes with two different slopes, consistent with the view that multiple energy barriers are present along the unbinding coordinate of the biotin-streptavidin complex. In contrast, the non-specific interactions, which can be separately measured and characterized apart from the specific bond-rupture forces in this method, did not exhibit a measurable dependence on loading rate. The dynamic force spectra acquired here with the AFM combined well with BFP measurements by others, and demonstrated that unbinding forces measured by different techniques are in agreement and can be used together to obtain a dynamic force spectrum covering 11 orders of magnitude in loading rate.

#### 10:40am BI+NS-ThM8 Measuring the Mechanical Properties of Soft Samples by Atomic Force Microscopy, M. Radmacher, Universität Tübingen, Germany

INVITED

The Atomic Force Microscope combines in a very unique way a very high sensitivity in detecting and applying forces (of up to a few  $10^5$  of piconewton), a high accuracy in positioning a sharp tip relative to the sample in all three dimensions (of up to a few Angstrom), and the possibility to be operated under physiological conditions. This combination allows experiments not possible before, particularly in the field of biophysics and soft materials. One example is the mapping of mechanical properties with high spatial resolution of polymeric films and living cells. In living cells it is

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possible to probe the mechanics during dynamic processes like cell migration and cell division.

11:20am **BI+NS-ThM10 Protein Adsorption and Monocyte Activation on Ge Nanopyramids**, **B. M@um u@ller**, ETH Z@um u@rich, Switzerland; **M. Riedel**, ProBioGen, Germany; **R. Hofer**, **E. Wintermantel**, ETH Z@um u@rich, Switzerland

The performance of an implant material depends crucially on its surface architecture or morphology. The significance of topographic features with micrometer size on cell shape and function has been clearly demonstrated. The power of features on the nanometer scale is still under discussion. In order to get an insight into the responds of monocytes onto a well-defined substrate nanostructure, we have grown germanium nanopyramids on Si(100) in a natural way by epitaxial growth, i.e. without any lithographic technique. The density of the pyramids (hut and dome cluster) is adjusted varying the substrate temperature during deposition. The morphology of the oxidized pyramids is quantified by ex situ atomic force microscopy. To characterize the nanostructure roughness further, contact angles of water under dynamic conditions are measured in comparison with the bare Si wafer and flat Ge films on Si. The receding angles show a significant increase with pyramid density. The amount of the selected proteins g-globulin and albumin adsorbed on the nanostructures is determined spectroscopically with labeled proteins. It raises with pyramid density. The impact of nanostructuring on the biological activity of adsorbed g-globulin is addressed by immunosorption with an anti-g-globulin antibody. These data reveal that the amount of active g-globulin does not scale with the adsorbed one. Nanoscale roughness even decreases the activity. The in vitro assays with monocytes that allow studying specific aspects of inflammatory reactions of the body - an important aspect of the biocompatibility, are based on the monocyte-like cell line U937. After 5 days in vitro, the cell performance is characterized microscopically and by the secreted cytokines IL-1b, IL-1ra and TNF-a. For the CVD grown samples, a roughness increase leads to reduced cytokine expression. Consequently, implants with nanoscale roughness gives rise to less inflammatory reactions.

## Magnetic Interfaces and Nanostructures Room 206 - Session MI+NS+NANO 6-ThM

### Nanomagnetism

**Moderator:** P.N. First, Georgia Institute of Technology

8:20am **MI+NS+NANO 6-ThM1 New Directions for Semiconductors**, **D.D. Awschalom**<sup>1</sup>, UC Santa Barbara **INVITED**  
PLEASE SEND US AN ABSTRACT. Thank you.

9:00am **MI+NS+NANO 6-ThM3 Electron Spin Relaxation at Nanometer Length Scales Near a Ferromagnet**, **B.C. Stipe**, **D. Rugar**, **H.J. Mamin**, **C.S. Yannoni**, IBM Almaden Research Center; **T.D. Stowe**, **T.W. Kenny**, Stanford University

Long spin relaxation times will be important to the success of many proposed solid-state quantum computing devices, spintronic devices, and to the detection of single spins by magnetic resonance force microscopy (MRFM). However, spin relaxation may be strongly influenced by thermal magnetic fluctuations in nearby materials such as conductors and ferromagnets. We have employed MRFM with 100 spin sensitivity and 20 nm spatial resolution to study the behavior of E' centers in SiO@sub 2@ near a micron-size ferromagnetic PrFeB particle tip. Magnetic resonance was induced within a 1 nm thick selective slice at 6 GHz and 3 Kelvin in a field gradient of 1 Gauss/nm. For detection, spins were manipulated by adiabatic inversion to produce oscillatory forces on the magnetic particle mounted on a sensitive cantilever. A typical spin ensemble consisted of 2000 spins with a net polarization of 100 μ@sub B@. T@sub 1@ was measured as a function of distance from the tip and was found to systematically decrease from 13 seconds when the spins were far from the tip to about 2 seconds within 500 nm of the tip. We interpret our results in terms of magnetic noise at the spin due to small-angle, thermal magnetic moment fluctuations in the particle. No relaxation effect due to proximity to the sample surface was found for depths greater than 50 nm. This work is supported, in part, by the Office of Naval Research.

9:20am **MI+NS+NANO 6-ThM4 Ferromagnetic Resonance of Monodisperse Co Particles**, **M. Farle**, **U. Wiedwald**, Technische Universitaet Braunschweig, Germany; **M. Hilgendorff**, **M. Giersig**, Hahn-Meitner-Institut Berlin, Germany

Quasi- twodimensional regular arrays of monodisperse 6 nm diameter Co particles can be produced on Carbon substrates by a monophoretic deposition technique from colloidal suspensions. Transmission electron microscopy reveals hexagonal ordering on micrometer scales for deposition at 0.8 Tesla and a bcc crystalline structure of the particles.@footnote1@ Ferromagnetic resonance (FMR) spectra at 296 K show a weak angular dependence near the paramagnetic resonance field with an easy in-plane magnetization axis . This shows a preferential alignment of the superparamagnetic particles in the film plane. A symmetric lineshape and linewidth dH < 0.1 Tesla is observed which indicates the high monodispersity in magnetic and geometric properties of the individual particles. Characteristic differences of the FMR spectra for different substrates and deposition parameters are observed and will be discussed in terms of simple dipolar coupling models. Supported through EC - grant no. HPRN-CT-1999-00150. @FootnoteText@ @footnote 1@ M. Giersig and M. Hilgendorff, J. Phys. D: Appl. Phys. 32 (1999) L111.

9:40am **MI+NS+NANO 6-ThM5 Magnetic Mirages**, **H.C. Manoharan**, **C.P. Lutz**, **D.M. Eigler**, IBM Almaden Research Center **INVITED**

While the correlated electron physics underlying the diverse manifestations of magnetism and spin have long been studied via macroscopic behavior, only recently have novel local probes opened the door to a new class of studies on the nanometer length scale. On top of these technological advances, the advent of controlled atomic and molecular manipulation provides a unique opportunity not only to detect spin phenomena at atomic length scales, but to manipulate spins as well. This talk will detail new results that exploit these techniques using low-temperature scanning tunneling microscopy.@footnote 1@ We have directly imaged the electronic perturbation arising from the spin-compensation cloud formed around isolated magnetic moments on a metal surface. Utilizing the detection of this many-body state, known as the Kondo resonance, we demonstrate that the spectroscopic signature of an atom may be sampled and projected to a remote location by means of a surrounding two-dimensional electron gas confined in an engineered nanostructure. The "quantum mirage" thus cast by a single magnetic atom can be coherently refocused at a distinct point where it is detected as a phantom atom around which the electronic structure mimics that at the real atom. Once materialized, this phantom can interact with real matter in intriguing ways. We have also been developing a novel communication method based on this effect. @FootnoteText@ @footnote 1@ H. C. Manoharan et al., Nature 403, 512 (2000).

10:20am **MI+NS+NANO 6-ThM7 Correlation of Structural and Magnetic Properties of Ultra-Thin Fe-Films on W(110) by Spin-Polarized STM/STS**, **A. Kubetzka**, **O. Pietzsch**, **M. Bode**, **R. Wiesendanger**, University of Hamburg, Germany

To investigate magnetism at the nm-scale and to improve the understanding of its underlying principles, a magnetic imaging technique with ultra-high resolution is of vital importance. Recently, spin-polarized scanning tunneling microscopy/spectroscopy has been developed to a reliable tool to allow such investigations down to the atomic level.@footnote 1,2@ We investigated Fe-films on W(110) at T = 15 K in a coverage range of 1 to 2 ML. In this regime Fe grows as double layer islands interconnected by a closed ML, where the island size can be tuned by the amount of evaporated Fe. By using ferromagnetically coated tips with a magnetization direction along the tip axes, we are sensitive to the out-of-plane component of the sample magnetization. Our measurements reveal that above a critical width of about 2.5 nm along the [1-10] direction, the islands are magnetized perpendicularly to the film plane. Below this width we do not observe a magnetic contrast which we attribute to a reorientation of magnetization to in-plane. Whereas the small perpendicularly magnetized islands are in a single domain state, we observe domain walls above a coverage of 1.5 ML, with wall widths of w = 7±1 nm. @FootnoteText@ @footnote 1@ Pietzsch et al., Phys. Rev. Lett. 84, (2000). @footnote 2@ Heinze et al., Science (accepted).

10:40am **MI+NS+NANO 6-ThM8 Current-driven Magnetization Reversal in Nanopillars**, **F.J. Albert**, Cornell University; **J.A. Katine**, IBM Almaden; **R.A. Buhrman**, Cornell University; **R.H. Koch**, IBM Research Division; **E.B. Myers**, **D.C. Ralph**, Cornell University

As reported elsewhere, we have successfully fabricated functional F/N/F thin film nanopillar devices with lateral dimensions down to 60 nm, and

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with one ferromagnetic layer considerably thicker (magnetically harder) than the other. A substantial shape anisotropy has been introduced by patterning a 2 to 1 aspect ratio into these nanopillars. This shape anisotropy is confirmed with the behavior of the measured switching field as a function of angle to the elongated axis. The resistance of these devices shows abrupt, single-domain-like switching when the spin-polarized current flowing through the nanopillar exceeds a critical value and forces the two F layers either into parallel or anti-parallel alignment, depending on the current direction. Here we will report on the results of detailed studies of this spin-transfer switching effect as a function of magnetic field, magnetic orientation and nanopillar composition. Also we will report on the spin-transfer switching dynamics of these devices, measured by probing them with extremely short pulses of current. We are also pursuing the thickness dependence of the switching behavior and will report these results.

11:20am **MI+NS+NANO 6-ThM10 Patterning of Co/Pt Multilayers: Topological vs. Magnetic, V. Metlushko, G. Crabtree, V. Vlasko-Vlasov, P. Baldo, L. Rehn, M. Kirk, Argonne National Laboratory; B. Ilic, Cornell University; S. Zhang, S.R.J. Brueck, University of New Mexico; B.D. Terris, IBM Almaden Research Center**

Using magnetron sputtering for Pt and e-beam deposition for Co the [Co<sub>4</sub>/Pt<sub>10</sub>]<sub>n</sub> multilayers were prepared on a Si/SiO<sub>2</sub> substrate. The patterning of submicron periodic arrays were done in two ways, using traditional interference- or e-beam lithography and lift-off which modulates the material composition of the film, and using 30 keV He ion irradiation through a mask which leaves the chemical composition and topography unchanged but reduces the magnetic anisotropy. The results of systematic characterization of arrays for different doses ranging from 1e15 to 5e16 ions/cm<sup>2</sup> with SQUID magnetization to determine the magnetic anisotropy and moment size, with atomic force microscopy (AFM) and magnetic force microscopy (MFM) to determine the topography and the magnetic order in the periodic arrays, and with magneto optical imaging to visualize the moment reversal process during a magnetization cycle will be presented. @FootnoteText@ This work was supported by the U.S. DOE, BES-Materials Sciences, under contract W-31-109-ENG-38 (V.M., G.C.) and by DARPA (S.Z., S.R.J.B.).

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6-ThM

### Nanostructured Materials

Moderator: T. Michalske, Sandia National Laboratories

8:20am **NS+NANO6-ThM1 Implantation and Pinning of Size-selected Ag Nanoclusters at the Graphite Surface, S. Pratontep, S.J. Carroll, M. Streun, P.D. Nellist, R.E. Palmer, The University of Birmingham, UK; S. Hobday, R. Smith, Loughborough University, UK**

Size-selected nanoclusters provide building blocks for creating nanoscale structures. However, the deposition of such clusters on surfaces is often susceptible to surface diffusion and thus aggregation of clusters. We have investigated the deposition of size-selected Ag@sub N@@super +@ clusters (N=20-200) on graphite at elevated impact energies (E=0.25-6 keV). Molecular dynamics simulations establish two energy regimes in which lateral diffusion of the deposited clusters is inhibited.@footnote 1@ At high impact energies (> 20 eV/atom) the clusters are found to penetrate into the surface and come to rest at the bottom of an open tunnel so created. The implantation depth is found to vary linearly as E/N@super 2/3@. This implies a constant decelerating force which is proportional to the cross-sectional area, rather than the volume, of the cluster. At lower impact energies (~10 eV/atom) we have found a size-dependent threshold energy above which the cluster is pinned on the surface at the impact site via the creation of a point defect. This threshold exhibits a linear dependence with the cluster size, N. This prediction is confirmed by scanning tunnelling microscopy (STM) investigations of cluster pinning as a function of cluster size and impact energy. Both the implantation and the pinning behaviour suggest viable methods for the fabrication of well-defined nanostructured surfaces. @FootnoteText@ @footnote 1@ S.J. Carroll, P.D. Nellist, R.E. Palmer, S. Hobday and R. Smith, Phys. Rev. Lett. 84, 2654 (2000).

8:40am **NS+NANO6-ThM2 Self-organization of Pt Nanoparticles on Surfaces in Catalytic Reaction, Q.-H. Hu, B. Kasemo, Chalmers University of Technology, Sweden**

Small metal particles have long been used as catalysts in heterogeneous catalytic reactions. The carrier may act both as a physical support and promoter for the catalytic reactions. Recently it is realized in the Monte Carlo simulation that supported catalyst particles may self-organize through a rather complex interaction with the supported surface and gaseous species in the reaction environment. As consequence, individual particles may attain different shape(adsorbate-induced reshaping or faceting) and combination of facets that strongly influence the reaction kinetics. In addition, particles as a whole may adopt different spatial arrangement, which affects mass-transport in the reaction. We report a technique for preparing SiN-membrane and TEM observations of self-organization of Pt nanoparticles on the membrane in the catalytic reaction. The TEM of as-prepared samples 'Pt deposited on the membranes' showed three types of morphology, continuous granular film at the nominal thickness of above 20 nm, irregular-shaped drops of varied sizes and coverage below 5 nm, and semi-continuous films with varied density of cracks at the thickness in between. The TEM of the samples heat-treated in the ambience of H<sub>2</sub> and O<sub>2</sub> at atmospheric pressure showed significant change in morphology. Thin Pt samples treated at 300 °C and H<sub>2</sub>/O<sub>2</sub>=0.4 self-organized into short-range ordered nanoparticles of less than 5 nm in size with a comparable interparticle spacing. The particle size is proportional to the initial thickness. On the medium thickness samples, multi-connected irregular-shaped islands resulted. The width of the interconnection is about 10 nm and almost equal to the spacing between the islands. Recrystallization of Pt was also observed. A systematic variation of treatment conditions on samples of different thickness, together with the evaluation of the catalytic activity is currently undertaken to realize a controlled shape of the self-organized particles.

9:00am **NS+NANO6-ThM3 Quantum-Coherence and Self-Assembly of Metallic Nanostructures, H. Brune, Ecole Polytechnique Federale de Lausanne, Switzerland**

INVITED

We will discuss several means for the self-assembly of metallic islands of well defined size and mutual distance. The surface states of the (111)-oriented transition metal surfaces have extremely small Fermi vectors leading to long wavelength quantum-interference patterns. For Cu/Cu(111) these Friedel oscillations give rise to long range oscillatory adsorbate-adsorbate interactions which can be used to arrange adatoms or islands in hexagonal patterns. The period of these superlattices is  $\lambda_F/2$  and thus coherently reflects the surface electronic properties. We then present the growth of nanostructure arrays using kinetically controlled growth on dislocation networks serving as templates. The mean size of the Ag, Fe, and Co dots is adjusted by the coverage. Their size distribution is given by the statistics of deposition. It has a half width at half maximum of 12% at 0.10ML, which is competitive with state-of-the-art semiconductor quantum dots, and goes down to 4% at 0.5ML. Small Co islands with well defined sizes can be created through coarsening by cluster diffusion on Pt(111). Since each cluster size has its own onset temperature for diffusion one can deliberately adjust the mean island size by the annealing temperature giving rise to high densities of very small islands ideally suited to investigate size-effects on reactivity and magnetic moment per atom.

9:40am **NS+NANO6-ThM5 Immobilization and Isolation of Molecule Corral Nanostructures Produced on Highly Oriented Pyrolytic Graphite, J.D. McBride, Y.J. Zhu, B. Van Tassel, R.C. Jachmann, T.P. Beebe, Jr., University of Utah**

Gold and silver nanostructures have been produced by condensing evaporated gold onto nanometer-sized etch pit templates (molecule corrals) on the surface of highly oriented pyrolytic graphite (HOPG). By varying the ratio of the thickness of gold deposited to the diameter of the etch pit templates, three distinct types of gold nanostructures can be produced: rings, disks, and mesas. These nanostructures have been formed with their outer diameters ranging from 20 to 150 nm. In order to use these nanostructures in the microelectronics industry, or for potential use as sensor arrays, they must be isolated both from one another as well as from the conductive graphite surface on which they are produced. Two different methods for isolating these nanostructures have been investigated. First, the use of (3-mercaptopropyl)trimethoxysilane (MPS) as a molecular adhesive@footnote 1@ to anchor the gold nanostructures to glass and silicon substrates was evaluated. The degree of gold transfer to the glass and silicon surfaces was examined using x-ray photoelectron spectroscopy (XPS), time-of-flight secondary ion mass spectrometry (ToF-

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SIMS), scanning tunneling microscopy, and atomic force microscopy. The second technique used for isolating gold nanostructures was the immobilization and encapsulation of the nanostructures within a spin-coated fluoropolymer film. These polymer films were examined using both the spectral and imaging modes of XPS and ToF-SIMS. @FootnoteText@ @footnote 1@ Goss, C. A.; Charych, D. H.; Majda, M. Anal. Chem. 1991, 63, 85-88.

10:00am **NS+NANO6-ThM6 Ultraflat Nanosphere Lithography**, *W. Frey, A. Chilkoti*, Duke University

Easy fabrication of periodic features on the submicron scale is needed for many applications ranging from surface-enhanced spectroscopy to quantum structures and nanostructured biofunctional surfaces. For example, biosensors require nanopatterns to be stably bonded to the substrate in solvents, as well as capable of subsequent functionalization by self-assembly chemistry. Additionally, for the study of cell surface-interactions, the nanopatterned surface should have minimal height variation in order to decouple physico-chemical and topographical effects on cell behavior. We have therefore designed a new method, ultraflat nanosphere lithography (UNSL), to create periodic nanopatterned surfaces of well-defined size and minimal topography for different materials that are also stably bonded and are capable of self-assembly chemistry. UNSL is based on nanosphere lithography@footnote 1@ and ultraflat template stripping.@footnote 2@ Nanospheres are self-assembled into close packed hexagonal arrays on mica. Material M1 is evaporated through the mask formed by the self-assembled spheres. After the spheres have been lifted off the substrate, evaporation of material M2 embeds the nanopattern in a much thicker matrix. A silicon or glass substrate is glued to the surface for mechanical support. Finally, the mica is stripped off, revealing an ultraflat pattern of nanotriangles of M1 embedded in M2. In order to demonstrate the feasibility of UNSL, we have chosen pairs of materials M1 (Au, Ag) and M2 (Al, SiO) so that each can be independently functionalized by orthogonal self-assembly, and transparent samples as well as insulator-conductor pairs can be created. We have visualized the nanopatterns fabricated by UNSL using AFM, SEM and Auger electron imaging, and demonstrate that the surface roughness is below 1 nm (rms) over areas of several hundred  $\mu\text{m}^2$ . @FootnoteText@ @footnote 1@Deckman, et.al.: Appl. Phys. Lett. 41, 377 (1982)@footnote 2@Hegner, et.al.: Surf. Sci. 291, 39 (1993)

10:20am **NS+NANO6-ThM7 Controlled Growth of Metal Nanoclusters on Polymers**, *V. Zaporojtchenko, K. Behnke, A. Thran, T. Strunskus, F. Faupel*, University of Kiel, Germany

Metal nanoparticles dispersed on a polymer surface as well within a polymeric matrix offer interesting electronic, optical and catalytic properties due to their small dimensions.@footnote 1@ For metals with moderate reactivity the high cohesive energy of the metal in combination with the weak metal-polymer interaction leads to metal aggregation on the surface. The nucleation and growth of noble metals (Cu, Ag, Au) as well as more reactive metals (Ni, Cr) onto fully cured polymer films with different composition are considered. The metal deposits which were prepared in situ by physical vapor deposition were characterized by combination of transmission electron microscopy (TEM), atom force microscopy (AFM), x-ray photoelectron spectroscopy (XPS) and a novel very sensitive radiotracer technique.@footnote 2@ An extreme variation in metal condensation behavior was observed on the different polymers. It appears to be related to the polymer surface energy and suggests a connection between macroscopic wetting and atomic condensation. The effect of metal-polymer interaction as well as the deposition parameters and surface pretreatment on the nucleation process has been investigated in order to understand how the morphology of the metal deposit can be affected. Influencing the basic processes@footnote 3@ occurring in the initial stages of metal-polymer interface formation it is possible to form 2-d metal nanoparticle-polymer composites with a large variation of the metal cluster size and metal cluster density on the polymer surface. @FootnoteText@ @footnote 1@ Metal Clusters, ed. by W. Ekardt, John Wiley & Sons, Chichester, England 1999. @footnote 2@ A. Thran, M. Kiene, V. Zaporojtchenko and F. Faupel, Phys. Rev. Lett. 82, 1903 (1999). @footnote 3@ V. Zaporojtchenko, T. Strunskus, K. Behnke, M. Kiene, and F. Faupel, J. Adhesion Sci. Technol., 14(3), 467 (2000).

10:40am **NS+NANO6-ThM8 Noble Metal Nanoparticles in Polymeric Thin Films**, *M.K. Chilton, P.G. Van Patten*, Ohio University

Recent results obtained in our laboratory and elsewhere have demonstrated the possibility of using noble metal nanoparticles to enhance the emissive response from phosphor materials by more than an order of

magnitude. We have employed electrostatically-grown polymer-based thin films as model systems for a series of experiments on these light-emitting nanocomposites. The fabrication and characterization of these model thin films will be described in detail along with spectroscopic results from the phosphor films. The enhancement effect has thus far only been observed in trivalent lanthanide ions. Experimental results suggest that the enhancement mechanism is related to the field focussing properties of the nanoparticles. Localized field enhancement apparently leads to the introduction of odd parity terms into the transition matrix element for photoabsorption. Interestingly, the rate of spontaneous emission from the ions are not observed to change as a result of the nanoparticles' presence. A detailed hypothesis for the mechanism will be presented consistent with existing photophysical evidence. The implications of the enhancement phenomenon for device development will also be discussed.

11:00am **NS+NANO6-ThM9 Biomimetic Self-Assembly**, *G. Stucky*, University of California, Santa Barbara  
**INVITED**  
PLEASE SEND US AN ABSTRACT. Thank you.

11:40am **NS+NANO6-ThM11 Cluster Size Effect Observed for Gold and Silver Sulfide Nanoparticles Synthesized by Sol-Gel Technique as Studied by X-ray Photoelectron Spectroscopy**, *S. Shukla, S. Seal*, University of Central Florida; *S. Mishra*, University of Memphis

Gold and Silver Sulfide nanoparticles have been successfully synthesized by a novel sol-gel technique. AFM analysis indicates the formation of @<=12-14 nm and 40-60 nm size Au nanoparticles; while TEM and AFM analysis indicate the formation of Ag@sub 2@S nanoparticles with average size of 26 nm (with broad size distribution ranging from 2.5 nm to 56 nm) and 200-300 nm, for respective H@sub 2@S/not heated and H@sub 2@S/heated samples. XPS analysis of H@sub 2@S/not heated sample, for Au nanoparticles, reveals that the core-level Au 4f@sub 7/2@ B.E. is shifted by +0.3 eV (with increase in the FWHM of 0.2 eV) relative to the bulk value of 84.0 eV. The shift is interpreted in terms of the changes in the electronic structure due to finite cluster size and creation of +ve charge over the surface of cluster during the photoemission process itself. The electronic structure of Au nanoparticles (or clusters) produced via present sol-gel technique is compared with that of Au clusters deposited by evaporation method described in the literature. XPS analysis of Ag@sub 2@S nano and powder particles suggests that the Ag 3d@sub 5/2@ B.E. level is a function of particle size distribution. Moreover, in both the systems, -ve shift in the core-level B.E. is observed, for H@sub 2@S/heated sample, which is attributed to the small size of the particles and their faceted nature. Suitability of the present method to produce silver sulfide ion-selective electrodes is demonstrated by depositing Ag@sub 2@S nanoparticles on the graphite rod and testing its response function.

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## Biomaterial Interfaces

### Room 202 - Session BI+NS-ThA

#### Biosensors

Moderator: A. Chilkoti, Duke University

2:00pm **BI+NS-ThA1 Substrate and Attachment Chemistry Effects on Adsorption and Single-Base Mismatch Discrimination on Immobilized Oligonucleotide Arrays**, *J.E. Forman, L. Gamble, R.S. Gascon, J.I. Henderson, A.D. Suseno, P. Wagner*, Zyomyx, Inc. **INVITED**

Hybridization efficiency and ability to discriminate between perfect match and single-base mismatch target sequences are fundamental to performance of arrays of covalently immobilized nucleic acids (probes). Two factors that significantly contribute to array performance are density and orientation of immobilized probes. Probe density not only controls how much bound target is adsorbed, but also affects the kinetics and thermodynamics of duplex formation. Perturbations to duplex formation occur when the spacing of probes is close enough to force inter-probe association and crowding, phenomena that interfere with target sequence adsorption. However, these perturbations can induce beneficial effects in array performance. For example, crowded nucleic acid surfaces demonstrate lower apparent melting temperatures ( $T_{sub m}$ 's) than the solution phase, but also show discrimination between matched and mismatched sequences under low stringency conditions. The orientation of the probe is highly dependent on the method of immobilization to the surface. Non-optimal attachment can orient the probe in a way that interferes with duplex formation, or such that it becomes buried within the surface and inaccessible to the target sequence. The substrate used for array preparation ultimately controls both density and orientation; optimization of the substrate can enhance array performance in an assay. Especially interesting is a precise control of probe density, where the density effects alter the  $T_{sub m}$ 's of immobilized sequences to allow a broad range of sequence to be analyzed in a single temperature assay. We have been exploring a variety of silane modified substrates for the preparation of oligonucleotide arrays, focusing on immobilization of the probe through amine or thiol moieties. The effect of density and attachment chemistry on target adsorption and single-base mismatch discrimination with single and double stranded oligonucleotide target sequences will be presented.

2:40pm **BI+NS-ThA3 Investigation of DNA Hybridization on Surfaces by Surface Plasmon Fluorescence Spectroscopy (SPFS)**, *T. Neumann, W. Knoll*, Max-Planck Institute for Polymer Research, Germany

The investigation of DNA hybridization on surfaces, and as a consequence, the development of DNA-chips and sensors, has been of increasing interest in recent years, since such technology can be used to investigate the human genome. We report here a study of PNA:DNA and DNA:DNA hybridization on surfaces measured by surface plasmon resonance spectroscopy (SPS) coupled with fluorescence (SPFS) and surface plasmon microscopy (SPM). PNA (peptide nucleic acids) and DNA catchers were immobilized either directly on gold surfaces by thiol linkers or via biotin on streptavidin covered gold surfaces. In order to enhance the detection limit of the SPR, the fluorescence signal of dyes attached to the target strands was detected during the hybridization to the immobilized catchers. To get a deeper insight into the underlying principles of the hybridization process near surfaces, conformational changes in the catcher and target DNA structure were monitored by comparing the hybridization kinetics obtained by having the fluorescent dye attached either to the catcher or the target and varying the length of both types of strands. Furthermore by using two different fluorescent dyes on the DNA strands, we were able to carry out Förster transfer experiments during the hybridization step, which allowed us to monitor distance changes between the catcher and target at the surface.

3:00pm **BI+NS-ThA4 Fluorescence Detection of Surface DNA Hybridization Reactions Based on Surface Structural Changes**, *T.H. Huang, S.J. Stranick, M.J. Tarlov*, National Institute of Standards and Technology

We describe a novel fluorescence method for the detection of surface-immobilized DNA hybridization reactions. Solid phase hybridization reactions form the basis of DNA chip technologies that are used for sequencing and genetic diagnostic applications. In conventional fluorescence-based detection schemes, the "target" DNA is typically labeled with a fluorophore. We report a method where instead, the "probe" DNA is labeled with a fluorophore. Our model surface hybridization system uses a mixed monolayer of fluorescein-tagged, thiol-derivatized, single-stranded DNA probes and 6-mercaptohexanol self-

assembled on Au surfaces. Prior to hybridization, the fluorophore on the probe is in closer proximity to the gold surface, resulting in greater quenching of the fluorescence signal. Upon hybridization, the double-stranded DNA adopts a rod-like structure that extends the fluorophore away from the gold surface. With the fluorophores located further from the gold surface, quenching is reduced and an increase in fluorescence intensity is observed. Parameters affecting fluorescence intensity such as probe surface coverage, probe length, and target concentration will be discussed. In addition, a comparison of probe- and target-labeled fluorescence detection schemes will be made.

3:20pm **BI+NS-ThA5 Silicon Surface Chemistry for DNA Immobilization**, *T. Strother, Z. Lin, W. Cai, L.M. Smith, R.J. Hamers*, University of Wisconsin, Madison

Many emerging areas of biotechnology, such as "gene chips", seek to leverage many aspects of the existing infrastructure in microelectronics and apply it to new areas. While most previous work has focused on the attachment of DNA to surfaces of gold or glass, we have investigated the chemistry involved in covalently bonding DNA to Si(001) and Si(111) surfaces in a way that retains its ability to selectively hybridize with its solution-phase complements. The use of XPS to study the chemical structure at each step in the DNA attachment process has led to the development of new procedures that are both simple and robust. Starting with hydrogen-terminated Si(001) and Si(111) surfaces, photochemical excitation at 254 nm is used to remove the photo-labile hydrogen; the exposed surface is then reactive toward organic molecules containing one or more unsaturated C=C bonds. "Linker" molecules containing a C=C double bond with another functional group (such as an amine or ester group) are then used to provide a dense set of surface-bound functional groups for attachment of DNA. To control the selectivity of the attachment process, however, careful optimization of the molecular structure of the linker and the other processing conditions are required. The density and chemical uniformity of the surface (as judged by XPS) is highly correlated with the intensity and selectivity achieved in subsequent binding of the surface-bound DNA to its fluorescently-labeled complements in solution. The results show that control of surface chemistry indeed leads to significant improvements in the formation of DNA-functionalized silicon surfaces.

3:40pm **BI+NS-ThA6 BARC: A Magnetoresistive Biosensor**<sup>1</sup>, *P.E. Sheehan*, Naval Research Laboratory; *R.L. Edelstein*, Geo-Centers, Inc.; *C.R. Tamanaha, M. Miller*, Naval Research Laboratory; *L. Zhong*, Geo-Centers, Inc.; *R.J. Colton, L.J. Whitman*, Naval Research Laboratory

The Bead ARray Counter (BARC) is a revolutionary biosensor that uses DNA microarrays, magnetic microbeads, and giant magnetoresistive (GMR) magnetic field sensors to detect and identify biological molecules.<sup>2</sup> The current prototype is a table-top instrument with integrated fluidics under development for the detection of biological warfare agents. The core of the sensor is a small, microfabricated chip containing a GMR sensor array for detection of up to eight different pathogens. Oligonucleotide probes complementary to pathogen target sequences are arrayed onto the microfabricated chip directly above the GMR sensors. Specific hybridization is measured and discriminated from non-specific background by addition of functionalized magnetic microbeads that bind to the captured target DNA. The beads tethered to the surface are detected by the GMR sensors, with the intensity and location of the signal indicating the concentration and identity of the target pathogens. A complete assay, including hybridization and detection can be performed in approximately 30 min. Because each GMR sensor is capable of detecting a single magnetic bead, in theory, the BARC biosensor should be capable of detecting a single molecule. With recent advances in GMR technology for computer memory, chips with millions of sensors will soon be commercially available, enabling the development of a BARC sensor capable of detecting thousands of analytes simultaneously. We will discuss the scientific and technical challenges to making such a sensor system a reality.<sup>1</sup> <sup>2</sup>Supported by the Defense Advanced Research Projects Agency. <sup>2</sup>Edelstein et al., *Biosensors & Bioelectronics* 14, 805 (2000).

4:00pm **BI+NS-ThA7 A Biosensor Based on Force Differentiation**<sup>1</sup>, *C. Yanavich, M. Malito*, Nova Research, Inc.; *G.U. Lee, L.J. Whitman, R.J. Colton*, Naval Research Laboratory; *M. Natesan*, Geo-Centers, Inc.

We are developing an array biosensor that uses a magnetic force to differentiate specific ligand-receptor binding from non-specific ligand-surface binding. In this force differentiation assay biosensor, capture antibodies that will bind to specific target analytes within a sample are first

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coated onto a surface. The captured analyte is then sandwiched by a second antibody that is attached to a magnetic microbead. A magnetic force with well-defined magnitude and orientation is applied to remove the non-specifically adsorbed beads, and a semi-automated optical reader then measures the number of the specifically bound beads remaining on the surface (which can be correlated with the analyte concentration). The original prototype used polystyrene microtitre wells for multi-analyte detection. We are now developing a second-generation sensor that employs a filter membrane as the capture surface. The filter facilitates concentration of the antigen on the surface and enhances the antigen-antibody interactions, significantly reducing the assay time (to ~30 min), and increasing the sensitivity by two-to-three orders of magnitude. Several techniques are being investigated to pattern capture antibodies onto the filter surface in order to enable multi-analyte detection on a single disposable filter. They include photo patterning with photo-activated biotin or caged photo-biotin, and imprinting via PDMS masks. We will also discuss our development of alternate techniques aimed at simplifying the bead counting system. @FootnoteText@ @footnote 1@Supported by the Joint Service Technical Panel for Chemical and Biological Defense (JSTPCBD).

**4:20pm BI+NS-ThA8 Encapsulation of Smart Polymers in Silica: Stimuli-Responsive Porous Hybrid Materials That Incorporate Molecular Nano-Valves, G.V. Rama Rao, G.P. Lopez, University of New Mexico; A. Chilkoti, Duke University**

Elastin (a protein) and poly(N-isopropyl acrylamide) (PNIPAAm, a synthetic polymer) are two types of thermo-sensitive, smart polymers which exhibit inverse solubility in water upon heating and undergo the transition from hydrophilic conformations to hydrophobic conformations at a temperature known as lower critical solution temperature (LCST). This interesting property has led to have several applications in biotechnological areas. In this report, we describe the development of molecular switches as nanoscopic actuators that can control the transport of chemical species by encapsulating PNIPAAm and elastin in dense silica gels by sol-gel synthesis. Cycling through the LCST can control molecular permeability through these hybrid materials. The pores resulting from the transition can selectively transport molecular species depending on their size. For example, permeation experiments revealed the LCST behavior of PNIPAAm in silica-polymer membranes and was identified to be 31 °C. DSC studies on bulk gels are in good agreement with the permeation results. Cycling of the membranes between 25 and 40 °C indicates the membranes possess reversible, variable permeability while maintaining good mechanical stability. Importantly, permeation experiments on PNIPAAm-silica membranes with various molecular weights of poly(ethylene glycol) have clearly demonstrated that the membrane is acting as a molecular switch by being impermeable below the LCST, and permeating the lower molecular weights of poly(ethylene glycol) and filtering out higher molecular weight polymers above LCST.

**4:40pm BI+NS-ThA9 Adsorption Behavior and Optical Properties of Surface-Adsorbed Polystyrene Nano Particles, M. Himmelhaus, Universität Heidelberg, Germany; H. Takei, Hitachi Central Research Laboratory, Japan**  
Polystyrene (PS) nano particles have become popular tools in photonics, nano technology, and life science since they have become commercially available in a wide range of sizes with narrow size distribution. While most applications utilizing surface-adsorbed PS nano particles deal with ordered arrays on mesoscopic scale, recently a chemically induced method for adsorption of such particles was introduced to yield random-close-packed (rcp) monolayers of almost arbitrary lateral extension. Such layers can be used as a template for the formation of cap-shaped Gold nano particles that exhibit extraordinary optical properties@footnote 1@ and thus can be developed into a sensitive optical biosensor.@footnote 2@ Here we demonstrate that the chemically induced adsorption method can be combined with alkanethiol chemistry to gain better control of sphere adsorption. Thus, sphere layers of varying density can be fabricated and their optical properties can be studied as a function of coverage. By further utilizing Micro Contact Printing ( $\mu$ CP) of tailgroup modified alkanethiols 2D patterns of rcp PS sphere monolayers with a lateral resolution of a few microns and a total pattern area of  $\sim 1$  cm<sup>2</sup>@super 2@ can be produced. These patterns are a first step to the development of an optical biosensor based on cap-shaped Gold nano particles with massively parallel detection capability. @FootnoteText@ @footnote 1@ H. Takei, J. Vac. Sci. Technol. B 17 (5) 1906, 1999 @footnote 2@ M. Himmelhaus, H. Takei, Sens. Actuators B 63 (1-2) 24, 2000

**5:00pm BI+NS-ThA10 Formation and Patterning of Supported Fluid Lipid Bilayers on a High Refractive Index Substrate, C.M. Ajo, L.C. Kam, S.G. Boxer, Stanford University**

Supported lipid bilayers are a useful model system to probe cellular membrane components and their interactions in a near native environment. Specifically, membrane components reconstituted in supported lipid bilayers create a well-defined two-dimensional system that can be manipulated - and then interrogated with a variety of surface specific and optical techniques. Several of these techniques rely on evanescent fields to probe the region near the solid support-lipid bilayer interface. However, the solid support typically has been a low refractive index material that permits the evanescent wave to penetrate significantly beyond the bilayer (650 Å). Here we report the formation of supported lipid bilayers on lithium niobate (LiNbO<sub>3</sub>), a material with a high refractive index ( $n=2.3$ ). Vesicle fusion onto lithium niobate forms a single uniform supported lipid bilayer that exhibits lateral diffusion properties similar to glass-supported lipid bilayers. By blotting and stamping,@footnote 1@ supported bilayers can be patterned reversibly, and the lipid components reorganize in response to an electric field. The high refractive index of lithium niobate restricts the penetration of an evanescent field to within 160 Å of the solid support-lipid bilayer interface. This provides a method to study the cell-supported lipid bilayer interface, since the relevant distances are on this order. Additionally, the transparency of lithium niobate over a wide range of wavelengths makes it a useful substrate for both visible and infrared studies. @FootnoteText@ @footnote 1@J. S. Hovis and S. G. Boxer, Langmuir 16, 894 (2000).

## Processing at the Nanoscale/NANO 6 Room 302 - Session NS+NANO6+MC-ThA

### Near-field Optics and Photonics

**Moderator:** Y. Kuk, Seoul National University, Korea

**2:00pm NS+NANO6+MC-ThA1 Optical Sieves - How to Get the Light through Subwavelength Holes, T.W. Ebbesen, Louis Pasteur University, France**

**INVITED**

Metallic films perforated with sub-wavelength holes ( $\sim 150$  nm) can transmit the light with an efficiency thousand times larger than what theory predicts for single holes. The efficiency can even be larger than the fractional area of the holes, which means that even the light falling beside the holes emerges on the other side of the sample. This extraordinary transmission is due to the coupling of the incident light with the surface plasmons of the film. The transmission spectrum contains peaks attributed to surface-plasmon modes that depend on both the symmetry and the 2D lattice parameter of the surface corrugation. We have shown that this phenomenon can also be used to tune and enhance the transmission of single subwavelength aperture. These results have broad fundamental and practical implications and show that, with modern fabrication techniques, surface plasmons can be engineered and controlled to yield unique optical properties.

**2:40pm NS+NANO6+MC-ThA3 Near-field Optical Measurements of Two Types of the Super-resolution Near-field Structures, D.P. Tsai, National Taiwan University, Taiwan; W.C. Lin, National Taiwan University**

Two different types of super-resolution structures for the near-field optical storage were studied. Direct experimental observation of the near-field optical properties in the super resolution near-field structures of glass/SiN(170nm)/Sb(15nm)/SiN(20nm) and glass/ZnS-SiO<sub>2</sub>(20nm)/AgOx(15nm)/ZnS-SiO<sub>2</sub>(20nm) have been achieved by using a tapping-mode tuning-fork near-field scanning optical microscope. Both propagating and evanescent field intensities were found at the focused spots of the surfaces of the super resolution structures. The evanescent intensity may result from the localized surface plasmons excited at the antimony or silver oxide. Images of the near-field intensity gradients at different excited laser powers demonstrated that the area had the static evanescent intensity could be stably controlled. Direct observation of the focused spot sizes of the static evanescent field intensity and their changes controlled by the excited laser power have demonstrated the working mechanism of both two types of super-resolution structures.

**3:00pm NS+NANO6+MC-ThA4 Near Field Surface Photovoltage Microscopy, R. Shikler, S. Saraf, Y. Rosenwaks, Tel-Aviv University, Israel**

Surface photovoltage (SPV) is a well-established technique for the characterization of semiconductors, which is based on analyzing illumination-induced changes in the semiconductor surface potential. The

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SPV and other related techniques like surface photovoltage spectroscopy (SPS), has been successfully used to study metal-semiconductor interfaces, surface states, bulk defects, and minority carrier lifetime and diffusion length. To date, all the SPV related techniques have a common significant drawback: they do not have high spatial resolution. With the developments of scanning probe microscopy techniques in recent years, the way is paved to conduct SPV measurements with nanometer lateral resolution. In this talk we describe a novel technique called near-field photovoltage (NFPV) which measures the SPV using near-field optical force sensor. The key feature of the technique is that the excited semiconductor sample is in the optical near-field region of a pulled optical fiber that measures the contact potential difference (CPD) between the fiber and the sample using the Kelvin probe force microscopy (KPFM) method. In such a case the illumination spot size is determined by the diameter of the aperture at the end of the tip and is not limited by diffraction. In addition, the light propagation is evanescent i.e. the intensity of the light falls off exponentially with increase distance from the tip edge (perpendicular to the crystal surface). This in combination with the high spatial resolution of the KPFM makes it possible to obtain depth-sensitive two-dimensional photovoltage images in semiconductors, and other materials. The method is demonstrated by photovoltage measurements conducted on buried p-n junctions of III-V compound semiconductors. When the sample was excited under far-field conditions, a decrease in the PV of the whole structure was observed due to a larger photovoltaic effect (band flattening) in the p-n junction. This is due to the fact that under super-bandgap illumination the band bending of the p-n junction decreases and causes a decrease of the structure work function. On the other hand, when the sample was excited and measured with the near-field optical force sensor, an increase in the PV was observed due to band flattening only in the surface space charge region. Our results demonstrate the large surface sensitivity of the NFPV technique, and opens the way for a variety of ultra-surface sensitive SPV measurements and applications.

**3:20pm NS+NANO6+MC-ThA5 Dual-Wavelength Scanning Near-Field Optical Microscopy, P.R. LeBlanc, M. Gu, P. Grutter, D.G. Gray, McGill University, Canada**

We have developed a Dual-Wavelength Scanning Near-Field Optical Microscope to shed light onto the scanning process and investigate biological samples in air. We couple 442 and 325 nanometer light into a tapered optical fiber glued to a quartz tuning fork which serves as our 'shear-force' sensor. Light transmitted through the sample is detected in a confocal arrangement by two photomultipliers. We have achieved topographical and optical resolutions of 10 and 30 nanometers, respectively. The dual-wavelength nature of our microscope permits the discrimination of topographical and optical cross-talk. It also allows the distinction of far-field artifacts from near-field features. Our primary biological application of the microscopy focuses on the investigation of the lignin distribution in wood fibers. Lignin, a cross-linked phenolic polymer, is of paramount importance in the pulp and paper-making processes. Our instrument permits the discrimination between chemical species density and topographical variations of the sample. The ratio of the two wavelength channels provides a simple and accurate parameter to determine the local concentration of lignin. These studies agree with atomic force microscopy images of wood cells and ultraviolet studies of wood cell walls.

**3:40pm NS+NANO6+MC-ThA6 Novel Scanning Near-field Optical Spectroscopy/Atomic Force Microscope Probes with High Polarisation Single/Double Slit and Cross Aperture Tips, H.P. Zhou, University of Glasgow, UK, United Kingdom; G. Mills, L. Donaldson, J.M.R. Weaver, University of Glasgow, UK**

We have developed novel scanning near-field optical microscopy/atomic force microscope (SNOM/AFM) probes. These probes have high polarisation single/double slit and cross apertures situated at the hollow tip apex of a silicon nitride cantilever. Direct-write electron-beam lithography (EBL) and silicon micromachining are used in a reliable batch process. The apertures are defined by lithographic means, therefore, the size, shape and orientation of apertures are well controlled and reproducible. The integration of a conventional force microscope cantilever with the aperture allows reliable control of the aperture-sample distance. The cross aperture probe has two very narrow orthogonal slits which have a width of much smaller than a wavelength, and a length on the order of a wavelength. We have designed and constructed a SNOM system based on the cross aperture probes. A parallel-polarised (p polarised) beam passes through the vertical slit and illuminates a sample hold in the near field of the aperture. The reflected light from the sample with the incident polarisation

is received by the same slit, while the perpendicular-polarised (s polarised) fluorescent component is received by the horizontal slit. Using this system, we have obtained near-field fluorescence imaging and have demonstrated spectrally resolved photoluminescence imaging with a spatial resolution of 50nm. The single and double slit apertures are vertically or horizontally oriented. The polarisation properties of the optical transmission from the slit aperture probes have been investigated as a function of the polarisation orientation. The polarisation behaviour of the slit probes is linearly polarised. The polarisation ratio ranged from 1:60 to 1:1000 for different ratios of length to width of slits.

**4:00pm NS+NANO6+MC-ThA7 Optical Properties of Strained GaInP/InP Quantum Dots Studied with STM Based Electro-luminescence, M.K.-J. Johansson, U. Hakanson, J. Johansson, M.-E. Pistol, L. Montelius, L. Samuelson, Lund University, Sweden**

The physics of nanometer scale structures has become a rapidly evolving field sparked by considerable interest from both science and technology. In the effort to assess the optical properties of individual nano-structures the use of scanning tunneling microscopy (STM) as the excitation source has received a lot of attention. Compared to more conventional techniques such as photoluminescence and cathodoluminescence the excited volume is very small and a further advantage is that the excitation energy can be greatly varied, from resonant conditions to above the impact ionization threshold. In addition STM provides very detailed information of the electronic structure and thus a manifold of data can be obtained within the same experiment. Here we will present measurements using a variable temperature UHV-STM combined with a scanning electron microscope. Furthermore, the system is equipped with an ex-situ laser source and optical detection system allowing a combined study of STM based electroluminescence, laser induced photoluminescence and cathodoluminescence to be performed without the need to change experimental conditions and set-up. In this report we investigate the optical properties of capped InP quantum dots grown by metalorganic vapor phase epitaxy on a highly doped GaInP layer, lattice matched to GaAs grown on a GaAs(001) substrate. We discuss the mechanisms of inducing luminescence and the dependence on excitation energy using the InP quantum dots as a model system. See for instance, A. Gustafsson et al. J. Appl. Phys. Rev. 88, 1715 (1998) and references therein.

**4:20pm NS+NANO6+MC-ThA8 Silicon Nanostructures via Intense Ultrafast Electronic Excitation, A.V. Hamza, M.W. Newman, University of California, Lawrence Livermore National Laboratory; T. Schenkel, University of California, Lawrence Berkeley National Laboratory; H.W.H. Lee, University of California, Lawrence Livermore National Laboratory; P. Thielen, University of California, Lawrence Livermore National Laboratory; J.W. McDonald, D.H. Schneider, University of California, Lawrence Livermore National Laboratory**

Due to the indirect nature of its band gap, bulk silicon is typically a poor photon emitter upon external excitation. However, as the crystal size approaches nanometer scales, the band gap widens due to quantum confinement and may become direct allowing for more efficient photon emission. Phase transformations induced by intense, ultrafast electronic excitation from slow, highly charged ions have produced nanometer-sized structures in silicon. Beams of highly charged ions of various charge state from 20+ to 69+ and various kinetic energies from 5 to 14 keV times charge have been utilized to induce this phase transformation in clean, silicon surfaces. The new phase is characterized by ex situ photoluminescence from the irradiated area after excitation with laser wavelengths from 379 - 514 nm. Photoluminescence spectra from the exposed areas show emission centered at ~540 nm. This is consistent with emission observed from 1-2 nm silicon nanocrystals. A series of sharp lines at 565, 555, and 548 nm are present in the photoluminescence spectrum from areas exposed to Xe<sup>4+</sup> which are characteristic of an excitonic series in nanometer-sized material.

**4:40pm NS+NANO6+MC-ThA9 Microstructural and Optical Properties of Porous SiC, S. Zangoie, University of Nebraska, Lincoln; H. Arwin, Linköping University, Sweden; J.A. Woollam, University of Nebraska, Lincoln**

Electrochemical etching of crystalline SiC in hydrofluoric acid creates a high surface area material with room-temperature light-emitting properties stronger than those obtained from bulk SiC. The luminescence properties of porous SiC (PSC) open up application possibilities with, e.g., SiC-based integrated electronics. Applications of PSC demand a detailed understanding of microstructure and its relation to different fabrication

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parameters. In this work, ellipsometry and electron microscopy are used for characterization of 4H- and 6H-PSC. The dominant surface morphology consists of branched rosette-like structures surrounding sparsely located and circularly shaped holes with diameters of the order of 20 nm or less. The density of pores descending from the surface does not show clear dependence on the etching conditions. It is likely that pore initiation is favored at certain defect sites. Pores in PSC are found to initially propagate nearly parallel to the surface and gradually change direction and align with the c-axis. Consequently, well-defined columnar pores are formed. Thickness dependent anisotropy in pore propagation influences the etch rate, which varies nonlinearly with anodization time. Etching parameters such as hydrofluoric acid concentration and current density influence the rate of change of propagation direction. Pore sizes are found to increase with depth due to a decrease in acid concentration. A disordered phase is encountered at the interface between crystalline SiC and the pores. Spectroscopic ellipsometry is used to study the microstructure, and we find that a simple effective medium approximation assuming mixtures of crystalline SiC and void does not result in good agreement with experimental data. To obtain good agreement with thickness and porosity, the disordered phase must be considered. Anodization of SiC introduces remarkable changes to the optical properties due to depth-inhomogeneity and particle shape effects.

5:00pm **NS+NANO6+MC-ThA10 Development of a Photon Detection STM for Inelastic Light Emission**, *D. Fujita, W.-L. Deng, T. Ohgi, K. Ishige*, National Research Institute for Metals, Japan

Recently it has been demonstrated that the light emission induced by tunneling electrons can be detected with normal STM operation even with atomic resolution. The possible processes are inelastic tunneling (IET) and hot-electron thermalization (HET). Generally speaking, in the case of IET, the tunneling electrons lose a part of the energy during the tunneling events to excite the state with some radiative decay processes. In the latter case, hot electrons lose the excess energy in the bulk. However, in order to get more detailed understanding of the luminescence process, much more precise measurements of these photons are required. For this purpose, we have developed a novel LT-UHV-STM system for the detection of light emission caused by the tunneling electrons. The emitted photons can be collected by the apex of a conductive and transparent optical fiber coated with a 100nm ITO (Indium-Tin-Oxide) film. Using a cooled photon detection system, single photon counting and spectroscopy with a very low noise level can be achieved. The system has been successfully applied to noble metal surfaces (Ag(111) and Cu(100)), and a direct-gap semiconductor surface (GaAs(110)) in UHV.

## Surface Science

### Room 209 - Session SS2+NS-ThA

#### Tribology and Adhesion

Moderator: J. Krim, North Carolina State University

2:00pm **SS2+NS-ThA1 Friction at High Speeds**@footnote 1@, *J.E. Hammerberg, T.C. Germann, B.L. Holian, P.S. Lomdahl*, Los Alamos National Laboratory **INVITED**

The mechanisms of dissipation at dry sliding metal-metal interfaces are complex, nonlinear, and involve a range of length and time scales. Depending upon the magnitude of the relative sliding velocity and the material pressure, phenomena described by phonons, dislocation generation and dynamics, microstructure formation, and mechanical mixing of materials are important. We discuss the variety of these phenomena as seen in extensive very large-scale molecular dynamics simulations for copper and Lennard-Jones systems in two and three dimensions. These simulations suggest universal behavior at large sliding speeds and we discuss some of the theoretical descriptions of these phenomena and recent high speed experimental results. @FootnoteText@ @footnote 1@Work performed under auspices of U.S. DOE at LANL under contract W-7405-Eng-36.

2:40pm **SS2+NS-ThA3 Energy Dissipation Thresholds During Interfacial Shear**, *N.D. Shinn, R.W. Cernosek, S.J. Martin*, Sandia National Laboratories

By measuring the power spectra of clean and surface-modified quartz crystal microbalance resonators in contact with nanometer thick fluid layers, we can detect and quantify energetic thresholds for interfacial slip and other dissipation mechanisms at interfaces under dynamic shear. Understanding fundamental dissipation mechanisms is a necessary prerequisite to mitigating friction via interfacial lubricants or surface

coatings. Using an amplified network analyzer, we vary the QCM peak-to-peak potentials, and hence the shear acceleration, over a wide range (0.002-30V) and detect any resultant changes in resonant frequency or resonance damping. This approach allows us to access shear forces otherwise unavailable using traditional QCM oscillator circuits [1] or standard network analyzer systems and therefore to probe stronger interfacial adhesive forces. Initial experiments for water adsorption on polycrystalline Au(111) electrode surfaces with and without a hydrophobic (methyl-terminated) alkanethiol self-assembled monolayer reveal no new dissipation thresholds. This suggests that interfacial slip is not occurring because the nanometer-scale surface roughness is sufficient to entrain the water layer or the energetic barrier for lateral motion remains insurmountable. Experiments are in progress to distinguish interfacial slip thresholds from mechanical entrapment effects by preparing atomically flat microcrystalline domains, contrasting polar and non-polar fluids, and modifying the electrode surfaces with other functionalized monolayers. Research 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@ @footnote 1@J. Krim and A. Widom, Phys. Rev. B38, 12184 (1988).

3:00pm **SS2+NS-ThA4 Chemical and Mechanical Contributions to the Friction In Self-Assembled Monolayers on Au Surfaces**, *H.I. Kim, J.E. Houston*, Sandia National Laboratories

By studying various combinations of chemically distinct end groups on alkanethiol molecules self-assembled on Au probe/substrate surfaces, and by varying the odd-even length of the chains, we are able to delineate the contributions of chemical and mechanical effects to interfacial friction. Because it is stable and quantitative throughout the entire range of the interfacial interactions (including the attractive load regime), we use the interfacial force microscope (IFM) to measure the friction force as a function of applied load. For even-length molecular chains, the friction forces progressively increase in the order CH<sub>3</sub>-CH<sub>3</sub>, CH<sub>3</sub>-COOH, COOH-COOH, while the interfacial adhesive force remains virtually identical. In contrast, for odd chain lengths, friction and adhesion are low for both CH<sub>3</sub>-CH<sub>3</sub> and CH<sub>3</sub>-COOH combinations, but much higher for COOH-COOH. By systematically correlating the interfacial adhesion and friction, we conclude that energy dissipation giving rise to sliding friction has distinct chemical and mechanical components. The making and breaking of hydrogen bonds, due to either intra- or inter-film interactions, gives rise to the "chemical" dissipation while simply disturbing the structure of the film under interfacial sliding invokes the "mechanical" component. This work was supported by the US Department of Energy under Contract DE-AC04-94AL85000. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed-Martin Company, for the US Department of Energy.

3:20pm **SS2+NS-ThA5 Atomic and Molecular Level Insight into Discrete Frictional Events**, *S.S. Perry, T.R. Lee, S.H. Lee, K.I. Kim, R. Colorado, P. Cao, Y.S. Shon, M. Graupe*, University of Houston **INVITED**

Technological advances in a number of industries has driven the need to understand interfacial friction on an atomic and molecular level. This level of understanding requires the careful design of model interfaces and experimental approaches sensitive to atomic and molecular scale events. This has been achieved in our studies by combining two recently developed techniques that operate at the subnanometer scale: control of the interfacial composition through molecular self-assembly or chemical modification under controlled UHV conditions and tribological measurements performed with the atomic force microscope. With this approach, we are able to control or measure the specific chemical composition and structure of interfaces and to correlate changes in composition and structure to the frictional properties of a single asperity contact. Examples will be drawn from both molecularly-thin organic film systems and composite coating systems to illustrate the origins of interfacial friction in terms of the composition and structure of the interface.

4:00pm **SS2+NS-ThA7 Molecular Tribology of Highly Ordered Monolayers**, *D. Gourdon*, University of California, Santa Barbara; *C. Duschl*, Swiss Federal Institute of Technology; *N.A. Burnham*, Worcester Polytechnic Institute

In order to investigate friction at a fundamental level, atomic force microscopy (AFM) in the wearless regime was performed on a model system - a highly ordered thiolipid monolayer on mica. In the monolayer, condensed domains with long-range orientational order were present. These domains revealed strong friction anisotropies as well as non-

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negligible asymmetries in the quasistatic friction loops. The directionality of these two effects appeared to correlate well with the tilt direction of the molecules (more specifically of their terminal alkyl chains) in the monolayer. The friction was measured as a function of applied load. The measurements versus load revealed two or three different frictional regimes (depending on the load range), that correlated well with a systematic stepwise behavior of the height of the domain as measured simultaneously. These discrete effects were attributed to molecular gauche defects created under the stress applied by the tip. Other studies include the friction as a function of sliding velocity, chemical preparation of the tip, alternative molecules, and nature of the substrate. Our work suggests that friction on this system is primarily a mechanical phenomenon.

4:20pm **SS2+NS-ThA8 The Effect of C60 on Interfacial Friction and Wetting of Toluene**, *T.S. Coffey, M. Abdelmaksoud, J. Krim*, North Carolina State University

Experimental investigations of friction, lubrication, and adhesion at nanometer length scales have traditionally been performed using atomic force microscopy (AFM), surface forces apparatus (SFA), or quartz crystal microbalance (QCM) techniques. While collectively these techniques yielded useful information, their results have never been cross-referenced. In order to achieve a cross referencing, we investigated the sliding friction of C60 using AFM, QCM, and macroscopic contact angle measurements. (Recent studies<sup>1</sup> suggest that a larger contact angle implies a more slippery interface.) C60 has been previously studied by S.E. Campbell et al. with SFA<sup>2</sup> whereby it was reported that C60 at a toluene/mica interface resulted in a full-slip boundary condition, and C60 was recommended as a possible lubricant. Our contact angle measurements reveal that the contact angle of C60/toluene solutions on mica is greater than the contact angle of toluene alone and therefore support Campbell's result. Using QCM, we have also studied the system toluene/Ag(111) with and without C60 deposited on the silver. These studies indicate that toluene on the C60/silver surface is less slippery than the toluene on the silver alone, and that the contact angle for toluene on C60/silver surface is less than the contact angle for the toluene on the silver. This indicates that C60 is not a good lubricant for a silver surface. We believe that this seeming contradiction with Campbell's result is due to the manner in which the C60 is bound to the mica surface vs. the silver surface. We complete our cross referencing of C60/toluene solutions on these substrates by employing AFM.<sup>3</sup> <sup>1</sup> J.L. Barrat and L. Bocquet, Physical Review Letters, vol. 82, p. 4671 (1999). <sup>2</sup> S.E. Campbell, G. Luengo, V.I. Srdanov, F. Wudl, and J.I. Israelachvili, Nature, vol. 382, p. 520 (1996). <sup>3</sup> This work is supported by NSF, AFOSR, and a GAANN fellowship.

4:40pm **SS2+NS-ThA9 Mechanical Behaviour of Confined Films: An In-situ Study of Silane Monolayers by Second Harmonic Generation (SHG)**, *M.T. Strobel, J. Blümmel, W. Eck, M. Buck, M. Grunze*, University of Heidelberg, Germany

Understanding the tribological properties of ultrathin organic films is of vital interest for their application in micro electro-mechanical systems (MEMS). To elucidate structural aspects of layers confined between two solids SHG was applied to trace force-induced changes of the polar and azimuthal orientation of molecular entities. As a model system monolayers of a silane bearing an SHG-active moiety were adsorbed on a glass-substrate. Mechanical loading and shearing of the film was achieved by a lens pressing against or rolling over the substrate. Mapping the contact area reveals pronounced, site dependent changes of both the tilt angle and the azimuthal alignment of the SHG active moiety upon loading. Whereas the azimuthal alignment persists after unloading and can be controlled by the rolling direction of the lens, the tilt angle is largely reversible for a loading unloading cycle. A quantitative evaluation of the data shows that the degree of the alignment is significantly larger than expected from simple models of contact mechanics.

5:00pm **SS2+NS-ThA10 New Aspects of Friction Force Microscopy in Ultra-high Vacuum**, *R. Bennewitz, E. Gnecco, T. Gyalog, O. Pfeiffer, Ch. Loppacher, M. Guggisberg, E. Meyer*, University of Basel, Switzerland

Recent experimental results of Friction Force Microscopy in ultra-high vacuum on well defined surfaces give new insight into the atomic processes of friction. On Cu(111), atomic stick-slip behaviour was found for the first time on a metal surface. Lateral stiffness of the contact and its I(V)-characteristic indicate that a copper neck between tip and sample is dragged over the surface.<sup>1</sup> The velocity dependence of atomic stick-slip processes was studied on Cu(111) and on NaCl(100) revealing a logarithmic increase of the friction with increasing scan

velocity.<sup>2</sup> This dependence can be explained by introducing the effects of thermal activation into the Tomlinson model for atomic friction. A new experimental approach to study dissipation processes on atomic scale is the measurement of the damping of vertical and horizontal tip oscillations in the non-contact mode, where power losses of the order of 100 meV per tip oscillation can be detected. <sup>1</sup> R. Bennewitz et al., Phys. Rev. B60 (1999) R11301 <sup>2</sup> E. Gnecco et al., Phys. Rev. Lett. 84 (2000) 1172.

## Material Characterization

### Room 207 - Session MC+NS-FrM

#### Characterization of Interfaces and Thin Films

**Moderator:** L.A. Giannuzzi, University of Central Florida

**8:20am MC+NS-FrM1 RBS, AFM, and AES Characterization of Pt Films Deposited by Ion Assisted CVD, B. Rogers, K.A. Telari, H. Fang, L. Shen, R.A. Weller,** Vanderbilt University; **D. Braski,** Oak Ridge National Laboratory

The semiconductor industry has used focussed ion beam (FIB) systems for several years to create site specific transmission electron microscopy (TEM) and secondary electron microscopy (SEM) cross sections, repair lithography masks, and to reroute electrical current by cutting and creating current carrying paths for circuit testing. The FIB systems' capability to both etch and deposit material in defined areas makes them good candidates for use as flexible processing systems for the development of specialized circuits. For example, metal patterns deposited in a FIB system could be used to form electrical connections and sensors on micro-electromechanical systems (MEMS) devices. Properties of the deposited material as well as the process by which it is deposited must be well characterized in order for the material to be successfully used in this type of development program. This work presents results from characterization of the ion assisted chemical vapor deposition of Pt from trimethyl(methylcyclopentadienyl)platinum [C@sub 9@H@sub 16@Pt]. Films were deposited in squares ranging from 50µm on a side to 400 µm on a side. Process parameters, including precursor temperature, dwell time, and ion beam current were varied to investigate their affect on the composition of the deposited films. A specially designed sample holder and Be mask enabled Rutherford backscattering spectrometry (RBS) determination and comparison of the Pt areal densities in these small features. Atomic force microscopy was used to determine the thickness and surface morphology of the deposited films. Scanning Auger electron microscopy was used to analyze the films for oxygen and carbon. Results show that the films contain a large amount of carbon and gallium in addition to the desired platinum. The presentation will conclude with a discussion of the effects of process parameters on the films' composition and possible pathways to maximize the platinum content of the films.

**8:40am MC+NS-FrM2 Material and Interface Characterization of Locally Deposited Dielectrics and Metals Fabricated with a Focused Ion Beam (FIB), H.D. Wanzelboeck, H. Langfischer, E. Bertagnolli, H. Stoeri, M. Gritsch, H. Hutter,** Vienna University of Technology, Austria

A chemical vapor deposition process was performed under vacuum conditions utilizing a focused ion beam (FIB) tool confining the deposition on a selected area in the Åµm down to the deep sub-Åµm range. The direct fabrication by FIB offers a versatile approach for rapid development and instant testing of novel microelectronic devices. The electrical properties of devices are influenced by the bulk attributes of the material. For complex multilayer structures the interface between 2 materials is of crucial importance. A focused Ga ion beam with diameter down to 5 nm was applied to induce decomposition of selected metalorganic species and siliconorganic compounds adsorbed on the sample surface. Deposition of metals was achieved by using metalcarbonyls such as W(CO)6 as precursors. Dielectrics such as siliconoxides were deposited by coadsorption of siloxane and pure oxygen. For siliconoxide the mixture ratio of the volatile precursor gases was found to be decisive for the chemical composition and electrical properties. For material characterization multilayer structures of dielectric and metal have been investigated. Auger-Spectroscopy revealed a correlation between the chemical composition of the FIB-deposited layers their electric characteristics and the process parameters chosen. Images of the X-section of the surface and interface deposited layers showed a homogeneous bulk density and a satisfactory surface roughness. Although the interface between FIB-deposited metal and dielectric layer is clearly visible in the secondary electron image depth profiling by secondary ion mass spectroscopy (SIMS) suggests an altered layer due to atomic mixing by the ion beam in a typical range up to 100 nm. The refined comprehension allowed to optimize the process parameters for improved material properties.

**9:00am MC+NS-FrM3 Three-Dimensional Material Characterization using Focused Ion Beams (FIB), R.J. Young,** FEI Company **INVITED**

The focused ion beam (FIB) system is integral to the manufacture of semiconductor and data storage devices. Systems are now found

throughout the manufacturing process, from the research laboratory to the production line. The ability of the FIB to image at high resolution, and to locally sputter and add material, provides capabilities not obtainable with optical and scanning electron microscope (SEM) techniques alone. FIB techniques include cross-sectioning, gas-assisted etching and deposition, circuit editing, ion channeling imaging, secondary ion mass spectroscopy (SIMS), and transmission electron microscope (TEM) thin film preparation. In addition, combining together the FIB and SEM in a single instrument (a "dual beam") produces an extremely powerful and flexible materials characterization system, providing unique three-dimensional subsurface metrology and analysis capabilities. In particular, the electron beam can be used to image the face of the cross section as it is being milled by the ion beam. This greatly aids in the location of very small defect sites. Further, the use of in-situ delineation etching allows the whole process of sample preparation to final imaging to be carried out in a single instrument. It should also be noted that SEM and FIB images and analysis techniques are in many ways complementary, the different contrast mechanism in each case giving additional information about the sample. Many of the FIB and dual beam techniques used in the analysis and sample preparation of microelectronic devices apply equally to other disciplines. In particular, the ability to locate, expose and then analyze submicron or unique defects has a wide applicability within material and biological sciences. This paper reviews the capabilities of focused ion beam systems, illustrating these with a number of case studies.

**9:40am MC+NS-FrM5 Visualizing Interfacial Structure at Non-Common-Atom Heterojunctions with Cross-Sectional Scanning Tunneling Microscopy@footnote 1@, J. Steinshneider, M. Weimer,** Texas A&M University; **R. Kaspi,** Air Force Research Laboratory; **G.W. Turner,** Lincoln Laboratory, M.I.T.

We describe how scanning tunneling microscopy (STM) may be used to image the interfacial bonding across the nearly-lattice-matched, non-common-atom GaSb / InAs heterojunction with atomic-scale precision. An ideal, compositionally-abrupt GaSb / InAs interface introduces new bonds - either InSb-like or GaAs-like - whose natural lengths differ from those in the surrounding host materials. These bond length differences are readily distinguished following (110) cleavage of the heterojunction, which locally relieves the out-of-plane compressive or tensile strain otherwise accommodated by a lattice-mismatched interface. The method is equally applicable to AlSb / InAs and suggests how it might be possible to determine the interfacial structure of such non-common-atom heterojunctions from atomic-resolution STM data.@FootnoteText@@footnote 1@Work supported by the National Science Foundation, Division of Materials Research, and the Air Force Research Laboratory

**10:00am MC+NS-FrM6 Atomic Resolved Spectroscopic Study of AlGaAs/GaAs/InGaAs by Cross-sectional Scanning Tunneling Microscope, J. Yu, L. Liu, J. Li, J.W. Lyding,** University of Illinois

An atomic resolved cross-sectional STM spectroscopic study has been performed on UHV-cleaved AlGaAs/GaAs/InGaAs heterostructures. Current imaging tunneling spectroscopy (CITS) results demonstrate significant electronic structure contrast between the column III and V sublattices as well as for the different compositional regions. The resulting images reveal surface structures not always evident in topographic images. Using a pattern recognition algorithm derived from the VERI analysis developed at Sandia,@footnote 1@ the image pixels can be grouped into classes according to similar electronic structure. This technique can be applied to automatically identify the spatial distribution of various elements during semiconductor growth. A detailed analysis and physical explanation of the spectra will be presented. @FootnoteText@@footnote 1@Bouchard, A. M., Osbourn, G. C., and Swartzentruber, B. S., Surf. Sci. 321, 276 (1994).

**10:20am MC+NS-FrM7 Scanning Spreading Resistance Microscopy of MOCVD Grown InP Structures, St.J. Dixon-Warren, S. Ingrej,** Nortel Networks, Canada

Scanning spreading resistance microscopy (SSRM) is a new probe microscopy technique that provides localized resistance profiling over a semiconductor surface. The technique, which is based on contact-mode atomic force microscopy (AFM), provides information on the two dimensional distribution of charge carriers and on the position of pn junctions in semiconductor structures. We have used SSRM to examine the cleaved edge of a number of MOCVD grown InP structures, including multi-quantum well layers and buried heterojunction laser structures. Information on the spatial distribution of dopants in the InP epitaxial layers was obtained. The effect of the applied tip voltage was investigated. We

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found that the active region of the laser structure, where the band gap is lowest, could be identified in the voltage dependence of the SSRM images. The effect of scanning rate, surface preparation and tip choice will also be discussed.

10:40am **MC+NS-FrM8 XPS Analysis of Interfacial WO<sub>3</sub> using Linear Least Squares and Standard Curve Fitting Routines, D.K. Fillmore**, Micron Technology, Inc.

X-ray photoelectron spectroscopy (XPS) has proven itself extremely useful in determining the chemical states of various materials. These chemical state identifications are primarily inferred from characteristic peak shapes and energy shifts along with the correlating presence of other elements and a previous knowledge of typical chemical compounds. When several chemical species are present in a particular sample, standard curve fitting routines are employed to extract the individual spectral components from multi-component spectra. This entails significant care in assigning each of the individual component peak energies, relative intensities and characteristic widths. The problem is exacerbated when ion sputtering is employed which results in sputtering-induced decomposition of components. From the curve-resolved data, atomic concentrations of individual components in a multi-component sample may be obtained. By making certain assumptions in specific circumstances, linear least squares (LLS) analysis routines may be able to quickly extract similar atomic concentration data from multi-component spectra without explicitly identifying individual spectral component peaks. This paper will discuss and compare analyses of interfacial WO<sub>3</sub> found between a surface SiO<sub>2</sub> layer and underlying elemental W using both LLS and standard curve fitting analysis routines. Specific advantages and disadvantages of each approach will be covered.

11:00am **MC+NS-FrM9 Investigations into the Chemical Nature of the Interfaces of Cu and Ta with SiN, J.F. Bernard, E. Adem, S. Avanzino, M.-V. Ngo**, Advanced Micro Devices

Surface chemistry is typically related to film properties such as adhesion, wettability, etc. For Cu interconnects the surface mobility of Cu ions can affect electromigration and line-to-line leakage in damascene structures. The surface treatment of Cu after CMP planarization will effect the chemical nature of the interface with the capping material as well as the interface of any liners with the cap. The chemical nature of the interface of Cu with SiN has been determined through XPS and AES compositional and chemical state depth profile analyses. The effect of plasma treatments on the Cu surface and resulting interfaces with the SiN capping layer is detailed. A mechanism for the Cu<sup>0</sup> catalyzed generation of the observed interfacial non-N bonded Si resulting from a silane based SiN deposition is proposed. Additionally, the interface of SiN with Ta is investigated to explore the effects of Cu treatments on the liner/cap interface. The basic outgassing behaviors of Cu and Ta are also discussed relative to the effects observed at the interface with the SiN capping layer. @FootnoteText@ @footnote 1@ Noguchi, J., "TDDB Improvement in Cu Metallization under Bias Stress"; International Reliability Physics Symposium 2000.

11:20am **MC+NS-FrM10 On the Organic Content and Outgassing Behavior of Organometallic-based CVD-TiN Films, J.F. Bernard, E. Adem**, Advanced Micro Devices

The drive to smaller IC geometries has led to an increasing use of organometallic precursor based chemical vapor depositions of TiN barrier films. The CVD-TiN process yields more uniform step coverage in high aspect ratio features. The CVD process also presents challenges in generating a consistently high quality TiN film. At lower temperatures both organometallic and metal halide precursors yield a lower density film with high resistivity and low N contents. A N-based treatment process has been widely employed to reduce the organic component and increase the N content of the films resulting from the use of TDMAT or TDEAT organometallics. The remaining C in the film has been shown to be present as a carbide of Ti. The chemical state of this remaining C has been further examined through the use of depth profiling, FT/IR, and wet chemical etching techniques. Deviations in the plasma treatment or the as deposited thickness can have a significant effect on the film quality. The affinity for O exhibited by uncapped PVD-TiN is higher still for untreated CVD films. The evolved gas profiles for a low resistivity film are dominated by H<sub>2</sub> outgasings. Films with a deviation in plasma conditions exhibited higher resistivity beyond spec limits and outgasings dominated by OH.

## Processing at the Nanoscale/NANO 6

### Room 302 - Session NS+NANO6+MC-FrM

#### Nanoscale Spectroscopy

Moderator: L.J. Whitman, Naval Research Laboratory

8:20am **NS+NANO6+MC-FrM1 Identification of Atom Species on Semiconductor Surface Using Noncontact AFM, Y. Sugawara**, Osaka University, Japan

INVITED

Recently, the noncontact atomic force microscope (AFM) using frequency modulation (FM) technique achieved true atomic resolution imaging. In order to apply the noncontact AFM as a powerful scientific tool for resolving the atomic features in variety of fields such as materials and biological sciences, it is very important to clarify the imaging mechanisms of the noncontact AFM. The imaging mechanism of the noncontact AFM was not established clearly, however the investigation concerning the imaging mechanism has been making progress gradually on various surfaces. In this paper, we experimentally investigated the force interaction between silicon tip and Si(111)-Ag surface by using the noncontact AFM. In case of reactive silicon tip, the AFM images showed the strong distance dependence dominated by the chemical-bonding interaction due to onset of the orbital hybridization very close to surface. On the other hand, in case of the adsorbed silver atom tip, the AFM images showed no distance dependence, and the individual silver atom on the top most layer of the surface could be resolved, corresponding to the true topography. This tip-sample force interaction seems to be dominated by physical bonding interaction such as the van der Waals force between the silver atom on the tip and the silver atom on the surface. These experimental results suggest that the noncontact AFM images drastically change depending on the atom species on the tip apex. It should be emphasized that there is a possibility to identify or recognize atom species on a sample surface using the noncontact AFM, if we can control an atomic species at the tip apex. @FootnoteText@ @footnote 1@ K.Yokoyama, T.Ochi, Y.Sugawara and S.Morita: Phys. Rev. Lett., 83 (24) 5023, 1999.

9:00am **NS+NANO6+MC-FrM3 Dynamic Polarization Imaging using Heterodyned Electrostatic Forces, D.R. Oliver, A. Pu, D.J. Thomson, G.E. Bridges**, University of Manitoba, Canada

Electric fields at the surface of dielectric materials will produce dipoles related to the polarizability of the material. In this paper we will present a technique that measures the polarization induced by a conducting probe near a surface. The images obtained are derived from the electrostatic forces between the probe and surface. In favourable geometries, the spatial resolution approaches the probe diameter. This measurement technique can be extended to frequencies well above the mechanical resonant frequency of the probe cantilever by utilizing amplitude modulation heterodyning. Using heterodyning, both time- and frequency-domain forms of this technique can be realized. We expect this technique to be useable up to frequencies of at least 20 GHz and time resolution of 100 ps. We present calculations of the forces generated assuming simple probe geometries and also thermal noise. Experimental results presented compare favourably with these calculations.

9:20am **NS+NANO6+MC-FrM4 Atomic Scale Chemical Identification on Si(111)-Ag by Atom Probe-Scanning Tunneling Microscope, T. Shimizu, H. Tokumoto**, JRCAT-NAIR, Japan

A scanning probe microscope (SPM) can determine atomic arrangement of various surfaces. It is, however, difficult to identify the chemical species of the atoms and molecules with the SPM. In order to know the atomic arrangement and its chemical species, there are several trials by combining the SPM with chemical analysis technique such as time-of-flight (TOF) mass spectroscopy and XPS. Spence has first demonstrated the possibility of TOF technique for chemical analysis. Then we have shown the usefulness of the TOF combined system in several cases. Furthermore a group in Delft Univ. has just tried to construct a TOF combined system. Nishikawa's group has already constructed a different kind of TOF. Here we shall present our challenge to atom-by-atom chemical analysis on solid surfaces by combining an Atom Probe of single atom sensitivity with the SPM ability of single atom manipulation (AP-STM). We have already demonstrated the following facts: (1) More than hundred Si atoms transferred from Si surfaces to a W-tip are detected; (2) The transferred Si atoms intermix with the tip material and form silicide; (3) Highly doped arsenic can segregate to the surface layers during annealing, form strange triangular step structure on Si(001) and the ratio of Si to As can be quantitatively determined. In this study, we will demonstrate the single atom identification on

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Si(111)@sr@3x@sr@3-Ag, whose atomic structure is supported by the Honeycomb Chained Trimer (HCT) model. The model is as follows. The topmost layer is formed by Ag atoms with the HCT arrangement. Below the Ag HCT layer, there is a Si trimer layer. From this model, it is believed that STM bright spots with sample plus voltage correspond to neither Ag nor Si atoms on the surface, but to the center of the triangle formed by three Ag atoms. To confirm the model directly, we are trying to identify the atomic position of Ag by extracting atoms from the surface and detecting the chemical species of extracted atoms.

9:40am **NS+NANO6+MC-FrM5 Tunneling Spectroscopy of Passivated Gold Nanocrystals**, *R.B. Wyrwas, A.Y. Ogbazghi, T.P. Bigioni, L.E. Harrell*, Georgia Institute of Technology; *T.G. Schaaff*, Oak Ridge National Laboratory; *R.L. Whetten, P.N. First*, Georgia Institute of Technology  
Scanning tunneling spectroscopy (STS) and microscopy (STM) have been used to study the electronic structure of small (< 2 nm diameter) alkanethiol-passivated gold nanocrystals. The nanocrystals were prepared via chemical methods, and their mass distribution was characterized by time-of-flight mass spectrometry. STM/STS measurements were done at room temperature, 77 K, and 8 K, with isolated nanocrystals immobilized on Au(111) surfaces by a xylenedithiol self-assembled monolayer (SAM). A histogram of nanocrystal heights measured by STM shows several peaks. These correlate well with distinct peaks in the mass spectrum. STS dI/dV spectra vary with nanocrystal size, and show prominent features due to their quantized density-of-states. Spectra acquired repeatedly over a single nanocrystal evolve in time slowly, possibly due to configurational changes of the nanocrystal or its passivation layer.

10:00am **NS+NANO6+MC-FrM6 Scanning Probe Microscopy Study of Engineered Ferroelectric Domain Structures**, *G. Rosenman, A. Agronin, Y. Rosenwaks, M. Lesnih, P. Urenski*, Tel-Aviv University, Israel

Ferroelectric domain engineering has recently become an attractive and perspective tool for diverse nonlinear optical applications based on the quasi-phases-matching method (QPM). The QPM occurs by the use of specifically tailored ferroelectric domain configurations with alternate direction of spontaneous polarization. Various types of engineered domain structures (periodic, aperiodic and quasi-periodic) allow an effective generation of coherent light in spectral regions where conventional lasers are not available. In this work we have studied engineered domain configurations (fabricated by electrical poling method in ferroelectric KTiOPO<sub>4</sub> crystals) using different scanning probe techniques like: piezoresponse imaging, Kelvin probe force microscopy, and conventional topography imaging. The different AFM methods image the periodically poled domains and domain walls with high spatial resolution. A detailed electrostatic analysis of the data, and the contrast between ferroelectric domains with opposite polarity is presented.

10:20am **NS+NANO6+MC-FrM7 Imaging of Single Molecules by Low Energy Electron Point Source Microscopy**, *A. Eisele, B. Völkel*, Universität Heidelberg, Germany; *S. Rehbein*, Universität Göttingen, Germany; *B. Jäger*, Universität Heidelberg, Germany; *H.J. Kreuzer*, Dalhousie University, Canada; *A. Götzhäuser, M. Grunze*, Universität Heidelberg, Germany  
Low Energy Electron Point Source Microscopy (LEEPS)@footnote 1,2@ has been used for taking in-line holograms of single DNA strands and carbon nanotubes. These two molecules were chosen to elucidate the extend to which the hologram images are determined by structural and electronic properties of the sample. The holograms were analyzed by reconstruction via the Kirchhoff-Helmholtz integral transformation.@footnote 3@ The resulting image is suggested to represent the scattering centers of the molecule, i.e. yielding its atomic structure. However, various effects can impose a limit to the resolution: vibrations between tip and sample, electronic charge deposition on the sample, non-ideal electron coherence, distortion of the electrical field near the sample molecule. It is discussed to which extend such artefacts are visible in the experimental data and whether they constrain the applicability of the Kirchhoff-Helmholtz transform. A comparison between the transformed images of DNA strands and carbon nanotubes shows differences that can be explained by properties of the molecules. @FootnoteText@ @footnote 1@ H.-W. Fink, W. Stocker and H. Schmid, Phys. Rev. Lett. 65, 1204 (1990). @footnote 2@ A. Götzhäuser, B. Völkel, B. Jäger, M. Zharnikov, H.J. Kreuzer, M. Grunze, J. Vac. Sci. Technol. A16(5), 3025 (1998). @footnote 3@ H. J. Kreuzer, K. Nakamura, A. Wiezbicki, H.-W. Fink, and H. Schmid, Ultramicroscopy 45, 381 (1992).

10:40am **NS+NANO6+MC-FrM8 Atomically-resolved Observation of Single Surface States and Trapped Electrons at Semiconductor Surfaces using Photovoltage Imaging with STM**, *S. Aloni, I. Nevo, G. Haase*, Weizmann Institute, Israel

Atomically-resolved, super- and sub-band-gap photon surface photovoltage (SPV) imaging was used to study surface states and a trapped exciton at semiconductor surfaces. Super-band-gap radiation that flattens the electronic bands gives rise to SPV that reflects the local band bending in the dark. On the other hand, a sub-band-gap photon can excite a localized electron in a surface state into the conduction band, and produce an SPV that reflects the new charge distribution. By studying the effect of the tip on the local band bending, we can learn about the nature of a single localized surface state on an otherwise surface-state-free surface. It turns out that a single adsorbate or a single atom-size defect, can sometimes screen efficiently a strong external electric field. When a filled deep surface state, such as As-vacancy defect on the surface-state-free GaAs(110) surface, was excited by 1064 nm (sub-band-gap) illumination, a trapped electron state was imaged: It's induced SPV feature had a slight positive value at a radius of ~20 Å, and a negative portion 5-50 Å further away, as expected for a localized positive charge with a wide (bias-dependent radius of 25-70 Å) electron-density distribution around it. An observed six-point star shape of the SPV feature is explained by the contribution of the surface atoms to the potential well shape.

## Thin Films

### Room 203 - Session TF+NS-FrM

#### Nanostructured Thin Films

Moderator: A. Hosseini-Tehrani, Florida International University

8:20am **TF+NS-FrM1 Cluster Beam Synthesis of Nanostructured Thin Films**, *P. Milani, P. Piseri, E. Barborini, A. Podesta', C. Lenardi*, Universita' Di Milano, Italy

INVITED

We will present and discuss the use of supersonic cluster beam deposition (SCBD) for the production of nanostructured thin films. With this technique nanostructured films of refractory and semiconductor materials can be produced over large areas on various substrates at room temperature. Elemental building blocks are clusters with a number of atoms ranging from few tens up to few hundreds. These units organize in the film in hierarchical structures from the nanoscale to the mesoscale. By exploiting aerodynamical effects typical of supersonic beams it is possible to obtain very high deposition rates with a control on neutral cluster mass distribution, allowing the deposition of thin films with controlled nanostructure. Due to high deposition rates, high lateral resolution, low temperature processing SCBD can also be used for the micro and nanopatterning of cluster-assembled films when little or no post-growth manipulation or assembly is required. As a practical example we will discuss the case of nanostructured carbon films. Surface morphologies, granularity and atomic structure of nanostructured carbon films grown with different precursors and under different conditions have been investigated by different techniques. The application of nanostructured carbon for the fabrication of supercapacitors and field emission devices will be discussed.

9:00am **TF+NS-FrM3 Growth of Regular Arrays of Pillars and Helices with Repeat Distance Below 100 nm**, *M. Malac*, University of Alberta, Canada; *R. Egerton*, Portland State University

Oblique deposition onto a rotating substrate pre-patterned with suitable nuclei results in the growth of regular arrays of pillars or helices.@footnote 1@ The repeat distance of such arrays can be varied from below 20 nm to over one micrometer. To improve the control over the microstructure it is beneficial to understand the growth mechanism on a microscopic scale. We find that the helices and pillars (within regular or random-nucleated arrays) are composed of many fibers growing simultaneously. The helix-arm diameter is determined by the number of fibers it contains. The distribution of fiber diameters is very narrow and remains constant along the entire helix height. The fiber-diameter, which is characteristic for given material and ratio of substrate and melting point temperatures, is in the order of a few nanometers. The crystalline nature of the growing material has only a minor influence on the fiber diameter. Additionally, we find that the columns formed within a film deposited onto a stationary oblique substrate are also composed of many individual fibers. The helix- and pillar-size distribution is much narrower within a regular unpatterned (randomly nucleated) array. This can be attributed to regularity of the shadowing between structures within a regular array. Anisotropy of shadowing within

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a regular square array arises from the varying distance of shadow-providing neighbors as substrate is rotated. This anisotropy has only a minor influence on the final shape of the pillars or helices. The helices within a random array start growth from a single fiber and exhibit a very narrow size distribution before the onset of bifurcation. This narrow size-distribution was observed when the helix arm diameter (= fiber diameter) is below the lower limit of scaling of the thin-film microstructure. @Footnote 2@ @FootnoteText@ @Footnote 1@Malac M, Egerton RF, Brett MJ, Dick B, J. Vac. Sci. Technol. B 17 (6), 1999 @Footnote 2@Bales GS, Bruinsma R, Eklund EA, Karunasiri RPU, Rudnick J, Zangwill A, Science 249 (1990).

9:20am **TF+NS-FrM4 Plasma Spray Deposition of Nanostructured Materials, H.M. Meyer, III, R. White**, Oak Ridge National Laboratory; *T.T. Meek*, University of Tennessee

Minimum film thickness achieved with any thermal-spray deposition process is limited by the size of powder feedstock materials. However, nanosized feedstock powder materials can be problematic for current plasma spray gun technology because of agglomeration and gun clogging. Some successes with nanopowder feedstock have been achieved, but mainly using special hardware and low quantities of expensive powders. We report the formation of nanostructured (i.e. nanomaterials) thin films using a unique combination of thermal spray coating techniques and liquid feedstock injection. This advanced thermal spray coating technology is a new method of synthesizing materials tailored at the nanoscale level. Injection of liquids into an arc-plasma gun offers the possibility of forming ultra-thin films at high deposition rates, a long-standing technology goal for the plasma spray industry. The formation of alumina thin films via plasma spray deposition using an aqueous precursor is presented. We compare the properties of our films with films formed by conventional thermal spray deposition using standard commercial alumina powders. Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U.S. Dept. of Energy under contract DE-AC05-00OR2272.

9:40am **TF+NS-FrM5 Deposition of Nanoparticles on Metal Surfaces, R.S. Averbach**, University of Illinois at Urbana-Champaign; *J.M. Gibson*, Argonne National Laboratory; *K. Albe, C. Zimmermann, Y. Ashkenazy, M. Yeadon*, University of Illinois at Urbana-Champaign **INVITED**

The deposition of metallic nanoparticles on surfaces has been investigated by a combination of molecular dynamics computer simulations and in situ transmission electron microscopy. It was observed that the reaction kinetics were dominated by a combination of interfacial stresses associated with epitaxy and capillary forces deriving from surface roughness. For Cu and Ag nanoparticles, it was found that the interfacial stresses led to 'contact epitaxy,' whereby the first few layers of the nanoparticles came into registry with the Cu(100) surface. Below a critical size, about 4 nm, the entire nanoparticle became epitaxial. For Co nanoparticles, which are more refractory and do not wet Cu(100) and Ag(100), contact epitaxy was not observed, although for deposition at 500 K, the nanoparticles burrowed into the substrate to achieve smoothing and coherency. Additional work examined how nanoparticles and grains in thin films respond to applied stresses. It will be shown that small particles on surfaces slide with little friction when they form a high energy grain boundaries, but that they quickly reorient during sliding and become locked from additional motion.

10:20am **TF+NS-FrM7 Scanning Probe Investigations of Passivated and Bare Au Nanoclusters on H:Si(111), M.D.R. Taylor, P. Moriarty**, University of Nottingham, UK; *M. Brust*, University of Liverpool, UK

Non-contact atomic force microscopy (NC-AFM) and scanning tunnelling microscopy (STM) have been used to characterise the morphology of thin films of decanethiol-capped 6 nm Au clusters spin-coated onto hydrogen-passivated (HF/NH<sub>4</sub>F treated) Si(111). Prior to cluster deposition, NC-AFM images taken under ambient conditions reveal that the H:Si(111) surface is atomically flat, typically consisting of 30-50 nm wide terraces separated by a mixture of single and multiple atomic steps. Following deposition of passivated clusters onto H:Si(111), a network of branched, dendritic islands (6 nm high) is observed with NC-AFM. The clusters forming these islands are remarkably loosely bound and may be swept aside by the AFM tip to form agglomerates of clusters via minor modification of the non-contact mode feedback parameters. The underlying layers consist of poorly ordered clusters which are stable under STM imaging conditions with tunnel currents up to 5 nA. This stability facilitates photon emission STM investigations of the passivated clusters. The results of comparative studies of STM-induced light emission from passivated and bare clusters (and from evaporated Au films) will be presented.

10:40am **TF+NS-FrM8 Nanophase Metal - Metal Oxide Films Deposited from a High-rate, Nanoparticle Beam, F.K. Urban III, A. Hosseini-Tehrani, P.D. Griffiths, A. Khabari**, Florida International University; *Y.-W. Kim, I. Petrov, L. Wei*, University of Illinois

While interest in nanophase films deposited in vacuum from nanoparticle beams dates back to the early 1970s, development of suitable sources for such beams has been difficult. A rapidly increasing number of different materials are now being used to deposit nanophase films, from a few tenths to a micrometer in thickness. The new method employs DC magnetron sputtering and condensation of a conductive target material into a helium and argon gas mixture in a flow rate ratio of up to 0.12 at total pressures between 0.7-0.8 Torr. A low velocity beam of the gas and nanoparticles is formed as they escape through a 3 mm-diameter converging-diverging exit nozzle consecutively into differentially pumped chambers maintained at pressures less than 10<sup>-3</sup> and less than 10<sup>-4</sup> Torr, respectively. Results to date confirm a typically oxygen-containing nanophase film structure. A set of new metal and doped semiconductor elements including Ag, Al, Au, Co, Cr, Cu, Fe, Mo, Nb, Ni, Pt, Si, Sn, Ta, W, and Zr have just been deposited. To our knowledge, some of these are deposited in vacuum for the first time and details on their nanostructure, composition, and materials properties are just now being learned and will be reported. To date, all films are nanocrystalline with grain sizes ranging from 1 to 20 nm and have a porous structure metallic densities, determined from combined Rutherford backscattering and film thickness measurements, in the range of 15-80% of the bulk densities. The effects of synthesis chamber wall cooling and gas conditions as well as starting material vapor pressure, melting point, and condensation energies will be presented. New findings on material properties will also be presented.

11:00am **TF+NS-FrM9 Thin Film Growth on a Fullerene Molecule, T.P. Martin**, Max Planck Institut KFK, Germany **INVITED**

Since the surface of a fullerene molecule is not unlike that of graphite, it might be expected that they have similar properties when acting as a substrate for thin film growth. The film-substrate interaction is indeed similar. However, the fact that the fullerene surface is curved and closes on itself leads to interesting differences. Both the similarities and the differences will be discussed including wetting, atom-packing, and reaction between film and substrate.

11:40am **TF+NS-FrM11 Sputtered Fabrication of Periodic Sub-Micron Structures, B. Dick, M.J. Brett**, University of Alberta, Canada

It is known that Glancing Angle Deposition (GLAD) utilizing extreme self-shadowing during film growth can produce periodic microstructures on a pre-defined seed layer with electron beam evaporation. @Footnote 1@ This deposition process has been applied to the fabrication of periodic magnetic pillars and has proposed application in optical devices. However, because individual seed elements enforce adatom shadowing on the substrate surface, higher operating pressures, resulting in larger angular flux distributions, can be tolerated in the formation of GLAD microstructures when deposited on a seed lattice. In this presentation, we report the use of low-pressure sputter deposition to fabricate periodic GLAD microstructures of between 500nm and 1.5µm thickness on substrates patterned with a 500nm period seed layer. We have characterized the growth of the microstructures in terms of the target area, deposition pressure, throw-distance, and flux incidence angle. The use of sputtering for periodic GLAD simplifies the process control, and should enable deposition of a broader range of materials for diverse applications including magnetics, optics, and sensors. @FootnoteText@ @Footnote 1@B. Dick, M.J. Brett, T.J. Smy, M.R. Freeman, M. Malac, R.F. Egerton. J. Vac. Sci. Technol. A. 18(4), 2000.

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