

## SYMPOSIUM O

### Scanning-Probe and Other Novel Microscopies of Local Phenomena in Nanostructured Materials

November 29 - December 3, 2004

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\* Invited paper

**8:30 AM O1.1**

**Lock-and-Key Effect in the Surface Diffusion of Large Organic Molecules Probed by STM.** Roberto Otero Martin<sup>1</sup>, Frauke Hummelink<sup>1</sup>, Fernando Sato<sup>2</sup>, Sergio B. Legoas<sup>2</sup>, Peter Thostrup<sup>1</sup>, Erik Laegsgaard<sup>1</sup>, Ivan Stensgaard<sup>1</sup>, Douglas S. Galvao<sup>2</sup> and Flemming Besenbacher<sup>1</sup>; <sup>1</sup>Department of Physics and Astronomy, University of Aarhus, Aarhus, Denmark; <sup>2</sup>Instituto de Fisica Gleb Wataghin, Universidade Estadual de Campinas, Campinas, Brazil.

A nano-scale understanding of the complex dynamics of large molecules at surfaces is essential for the bottom-up design of molecular nanostructures. Although a large number of previous studies has discussed the formation of molecular assemblies in terms of the molecule-molecule and molecule-substrate interactions, thus addressing the thermodynamic aspects of molecular assemblies, very few studies have addressed the dynamical processes that a large organic molecule can undergo when anchored on a solid surface. Here we address how the orientation and shape of large and complex organic molecules influence their dynamics (diffusivity). To this end, we have exploited the capability of the scanning tunneling microscope (STM) (i) to resolve the conformation and orientation of single individual molecules on the atomic scale, (ii) to manipulate the orientation of individual molecules by using the STM tip as a nano-scale tool to gently push molecules adsorbed on surfaces, and finally with the fast-scanning Aarhus STM (iii) to track the surface diffusivity of the different molecular configurations by acquiring dynamic STM movies at low temperatures. In this way, we will show that the diffusion coefficient of the complex organic molecule named the Violet Lander (VL, C108H104) on a Cu(110) surface can be changed by two orders of magnitude by purposefully manipulating the VL molecules, thereby modifying the molecular orientation with respect to the substrate. From an interplay with Molecular Dynamics simulations, we interpret the results within a lock-and-key model similar to the one driving the recognition between biomolecules: the molecule (key) is immobilized only when its orientation is such that the molecular shape fits the atomic lattice of the surface (lock); otherwise the molecule is highly mobile.

**8:45 AM O1.2**

**Biaxial Strain Induced Electrical Inhomogeneities and Phase Separation in the Ferromagnetic Metallic Phase in Thin Films of  $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ : A Scanning Tunneling Potentiometry/Spectroscopy Study.** Mandar Paranjape, Joy Mitra, Achyut Bora and A. K. Raychaudhuri; physics, indian institute of science, bangalore, karnataka, India.

We have investigated the effect of biaxial strain on local electrical/electronic properties in thin films of  $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ . The local electrical properties were investigated primarily in the ferromagnetic (FM) metallic phase of the system, as a function of temperature via scanning tunneling potentiometry (STP) and scanning tunneling spectroscopy (STS). To study the effect of strain in the thin films we chose two 50 nm films of  $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$  deposited on  $\text{SrTiO}_3$  (LCMO/STO) and  $\text{NdGaO}_3$  (LCMO/NGO). Since NGO is ideally lattice matched with  $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ , the LCMO/NGO is practically unstrained while LCMO/STO is under tensile biaxial strain due to significant film-substrate lattice mismatch (-0.6%). The topography images on the films indicate a terrace step pattern, which is characteristic of such epitaxial thin films. The spatially resolved STS measurements, on LCMO/STO, indicate the presence of phase separated (PS) regions that have different local density of states and thus different surface conductance. Such PS is seen to appear just below  $T_c$  with the volume fraction of the coexisting phases maximizing at  $T/T_c = 0.95$ . On further lowering of temperature the high conductance phase is seen to stabilize leading to an overall increase in sample conductance. Similar measurements on LCMO/NGO show the sample to be significantly more homogeneous with no detectable presence of PS. We also find that such PS leads to enhancement of  $1/f$  noise, which we model as arising from the fluctuations at the PS regions. The STP images, that maps the local electrical potential, indicate an inhomogeneous potential distribution and charge transport in the nanometer scale in both samples though being more pronounced for LCMO/STO than LCMO/NGO. The local electric fields calculated on the terraces (ET) and steps (ES) shows a marked difference in the paramagnetic (PM) phase above the FM-PM transition temperature,  $T_c$ . As the temperature is lowered across  $T_c$ , LCMO/STO shows an increased local potential variation though the macroscopic sample conductance decreases below  $T_c$ . This is further reflected in the ratio  $\mathbf{E}_S/\mathbf{E}_T$  which shows a monotonic increase with the lowering of temperature across  $T_c$ . In contrast LCMO/NGO shows a uniform decrease of potential inhomogeneities and of the ratio  $\mathbf{E}_S/\mathbf{E}_T$  as it is cooled below  $T_c$ . This ensures a more homogeneous

potential distribution across the sample. The above results indicate a positive correlation between the lattice mismatch biaxial strain and the local electrical/electronic inhomogeneities observed on the strained sample. This is plausible since the crystal structure of the manganites interfere rather strongly with the magnetic/electronic degrees of freedom. Thus even a small imbalance (biaxial strain) can induce significant changes in the electrical properties of the system. It is rather interesting and important to quantify such strain effects and optimize its role in potential device applications.

**9:00 AM \*O1.3**

**Atom Selective Imaging and Mechanical Atom Manipulation Based on Noncontact-AFM Method.** Seizo Morita<sup>1,2</sup>, Noriaki Oyabu<sup>1</sup>, Yoshiaki Sugimoto<sup>1</sup>, Oscar Custance<sup>2</sup>, Masayuki Abe<sup>1</sup> and Ryuji Nishi<sup>1</sup>; <sup>1</sup>Department of Electronic Engineering, Osaka University, Suita, Osaka, Japan; <sup>2</sup>Handai FRC, Suita, Osaka, Japan.

Recently we found that noncontact atomic force microscope (NC-AFM) based on mechanical method has capabilities of chemical distinction and atom manipulation [1]. For atom manipulation, we used mechanical contact and achieved vertical manipulation such as atom removal and deposition (creation and repair of a missing Si adatom defect) [2]. Very recently, we also achieved lateral manipulation of individual atoms adsorbed on Ge(111)-c(2x8) surface [3]. For distinction of atom species, we used difference of atomic force and achieved the distinction of various atoms such as (1) oxygen and Si atoms on oxygen adsorbed Si(111)7x7 surface [4], (2) Si and Sb atoms on Si(111)5r3x5r3-Sb surface [5], (3) Si and Ge atoms on Si(111)7x7-Ge surface, and (4) Si and Sn atoms on Si(111)7x7-Sn and Si(111)3x3-Sn surfaces [6]. In case of Si(111)5r3x5r3-Sb surface, we compared Si and Sb adsorbed tips and found that modification of tip apex atom can enhance the capability of atom distinction [4]. Besides, we measured the contact potential difference (CPD) on Si(111)5r3x5r3-Sb surface using Kelvin probe force microscope (KPFM) combined with NC-AFM. As a result, we achieved the atomic resolution of CPD and found that atomically resolved CPD could also assist the atom distinction [4]. References [1] S.Morita, R.Wiesendanger and E.Meyer (eds.); Noncontact Atomic Force Microscopy, Springer, NanoScience and Technology (2002) pp.1-439. [2] N.Oyabu, O.Custance, I.Yi, Y.Sugawara, and S.Morita; Mechanical Vertical Manipulation of Selected Single Atoms by Soft Nanoindentation Using Near Contact Atomic Force Microscopy, Phys.Rev.Lett., Vol.90, No.17 (2003), pp. 176102-1-176102-4. [3] N.Oyabu, Y.Sugimoto, O.Custance, M.Abe and S.Morita; Using Noncontact Atomic Force Microscopy for Laterally Manipulating Single Atoms at Semiconductor Surfaces, Abstract of NC-AFM 2004, 12-15 September 2004, Seattle, USA, <http://www.engr.washington.edu/epp/afm/abstracts.html>. [4] R.Nishi, S.Araragi, K.Shirai, Y.Sugawara and S.Morita; Atom Selective Imaging by NC-AFM: Case of Oxygen Adsorbed on a Si(111)7x7 Surface, Appl.Surf.Sci, Vol.210, Nos.1-2 (2003) pp.90-92. [5] K.Okamoto, K.Yoshimoto, Y.Sugawara, and S.Morita; KPFM Imaging of Si(111)5r3x5r3-Sb Surface for Atom Distinction Using NC-AFM, Appl.Surf.Sci, Vol.210, Nos.1-2 (2003) pp.128-133. [6] Y.Sugimoto, M.Abe O.Custance and S.Morita; Surrounding Atom Effect on Atomic Force of the Sn/Si(111) Surface, Abstract of NC-AFM 2004, 12-15 September 2004, Seattle, USA, <http://www.engr.washington.edu/epp/afm/abstracts.html>.

**10:00 AM \*O1.4**

**The Frontiers of Scanning Tunneling Probes in Condensed Matter Physics.** E. Ward Plummer<sup>1,2</sup> and A. V. Balatsky<sup>3</sup>;

<sup>1</sup>Department of Physics and Astronomy, University of Tennessee, Knoxville, Tennessee; <sup>2</sup>Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>3</sup>Theoretical Division, Los Alamos National Laboratory, Los Alamos, New Mexico.

This presentation will describe the state-of-the-art and our vision of the future in the development and utilization of scanning tunneling probes for the elucidation of phenomena in the world of contemporary condensed matter physics. The first examples will be maps of the Fermi contours from Cu to High  $T_c$  materials using Fourier Transform techniques. The use of  $dI/dV$  spectroscopy to map band structure, life-times, and many-body effects above and below the Fermi energy will then be discussed. Many of the phenomena associated with contemporary materials involve some form of spatial inhomogeneity. The use of spatial maps using  $dI/dV$  spectral analysis will be discussed, both in real space and k-space. For example, inhomogeneities in the superconducting gap in High  $T_c$  materials, metal-insulator inhomogeneities in CMR materials, magnetic-nonmagnetic inhomogeneities in artificially structured nanomagnetic materials, etc., will be discussed. Finally, we will discuss the possibility of doing real and k-space maps of excitations using the  $d^2I/dV^2$  mode. All of these techniques will benefit for variable external environments, such as temperature, magnetic field, RF or optical excitation, etc. How does the research enterprise in the US promote the development of such beyond the state-of-the-art

instruments, and more importantly how do we make them available to the materials community. There is a need for national centers for the Advancement of Scanning Probe Technology.

#### 10:30 AM O1.5

**Bromine Islands on Cu{111}: Evidence that Surface State-mediated Interactions are Dominant at High Temperatures.** Sanjini U. Nanayakkara, E. C.H. Sykes, L. C. Fernandez-Torres and Paul S. Weiss; Departments of Chemistry and Physics, The Pennsylvania State University, University Park, Pennsylvania.

A quantitative study of the long-range interactions between bromine islands on Cu{111} mediated by the surface state at elevated temperatures is presented. Randomly distributed bromine adatoms are immobile at 293 K, but when annealed to 600 K the adatoms become mobile and aggregate to form islands. The interaction potential between these islands has been determined by evaluating the distance distribution between the bromine islands from a series of scanning tunneling microscopy images taken at 4 K. By only taking two-body interactions into consideration, we demonstrate that the interaction potential is oscillatory in nature with potential energy minima observed at 12, 26 and 41 Å. This corresponds to a period of half the Fermi wavelength of Cu{111}. Unlike previous quantitative studies of long-range interactions between single metal adatoms on Cu{111} at temperatures up to 21 K,<sup>(1,2)</sup> we do not observe long-range interactions up to 70 Å due to the shorter coherence length of the Cu surface state at elevated temperatures. Our findings indicate the pair potential exists only up to 40 Å, which is in good agreement with the coherence length of 34 Å of the Cu{111} surface state at 600 K.<sup>(3)</sup> In this study we present conclusive evidence that the perturbations due to the surface state are important and relevant in mediating interactions between adsorbates at catalytically relevant temperatures. (1) Repp, J.; Moresco, F.; Meyer, G.; Rieder, K. H.; Hyldegaard, P.; Persson, M. Phys. Rev. Lett. 2000, 85, 2981. (2) Knorr, N.; Brune, H.; Epple, M.; Hirstein, A.; Schneider, M. A.; Kern, K. Phys. Rev. B 2002, 65, 115420. (3) Fujita, D.; Amemiya, K.; Yakabe, T.; Nehoh, H.; Sato, T.; Iwatsuki, M. Phys. Rev. Lett. 1997, 78, 3904.

#### 10:45 AM O1.6

**Pairwise Movement of Iodine on Si(100) at Room Temperature: Direct Observation with Scanning Tunneling Microscopy.** G. J. Xu<sup>1</sup>, A. W. Signor<sup>1</sup>, Abhishek Agrawal<sup>1</sup>, Koji S. Nakayama<sup>1</sup>, B. R. Trenhaile<sup>2</sup> and John H. Weaver<sup>1,2</sup>; <sup>1</sup>Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Physics, University of Illinois at Urbana-Champaign, Urbana, Illinois.

Halogen molecules dissociatively chemisorb on Si(100)-(2x1) and the subsequent mobility of atomic and paired ad-species increases from Cl to Br to I. For Cl, which has the highest barrier for diffusion, scanning tunneling microscopy reveals both single and paired adatoms at 295 K. For Br, the barrier is smaller and single adatoms interrogate the surface until they form pairs, which are then immobile, or are trapped at C-type defects. For I, the barrier is smaller still, C-type defects still act as traps, and pairwise motion is possible and can be quantified at room temperature. Studies over long times reveal pairwise diffusion that is ultimately quenched by the increasing density of C-type defects (dissociated H<sub>2</sub>O). We show that the diffusion of I pairs is thermally activated, the events are random, and they can be frozen-out at 80 K. The diffusivities along and across the dimer row are 0.61 and 0.24 Å<sup>2</sup>/sec at T = 295 K. Their respective energy barriers are estimated to 0.79 and 0.81 eV, assuming an attempt frequency of 10<sup>12</sup> sec<sup>-1</sup>. The effective barrier for pairwise motion is the sum of the isolated atom barrier and the energy needed to dissociate a pair, i.e., the π bond energy for Si(100).

#### 11:00 AM O1.7

**Temperature dependence of molecular diffusion in alkanethiol self-assembled monolayer studied by scanning tunneling microscopy : the effect of melting transition and molecular desorption.** Ung Hwan Pi<sup>1</sup>, Han Young Yu<sup>1</sup>, Chan Woo Park<sup>1</sup>, Sung-Yool Choi<sup>1</sup>, Jun Ho Kim<sup>2</sup>, Yong-Kwan Kim<sup>3</sup> and Jeong Sook Ha<sup>3</sup>; <sup>1</sup>Dept. of future technology, Electronics and Telecommunication Research Institute (ETRI), Daejeon, South Korea; <sup>2</sup>Dept. of Physics, Incheon University, Incheon, South Korea; <sup>3</sup>Dept. of Chemical and Biological Engineering, Korea University, Seoul, South Korea.

We have studied the surface diffusion of the molecules in alkanethiol self-assembled monolayer (SAM) by using scanning tunneling microscopy (STM) with a variation of surface temperature. From the successive STM images obtained at several temperatures, we could visualize the evolution of the melting transition and molecular desorption of alkanethiol SAM. The melting temperature estimated from the STM images was dependent upon the chain length of the alkanethiol, that is, the longer chain alkanethiol SAM has the higher melting transition temperature. Below melting transition

temperature, the molecules diffuse only through narrow channels, i.e., the boundaries of the well ordered lattice domains, resulting in a slow structural change of the surface. Above melting transition temperature, the molecular diffusion shows different behaviors depending upon the chain length of the alkanethiol. For a relatively short octanethiol SAM, the enhancement of the molecular diffusion after melting transition is not outstanding. For dodecanethiol that has a longer chain, however, the diffusion is dramatically enhanced after melting transition, rendering an abrupt structural change of the surface. The measurement of the molecular coverage shows that the desorption temperature is quite near the temperature of melting transition in case of dodecanethiol SAM. The dramatic enhancement of the molecular diffusion observed in dodecanethiol is induced by the molecular desorption. The empty sites formed during the desorption process give much room for the molecular diffusion.

#### 11:15 AM O1.8

**Using ultra-small amplitude AFM for linearized measurements of nanomechanical phenomena.** Peter M. Hoffmann, Shivprasad Patil, George Matei and Atay Tanulku; Physics, Wayne State University, Detroit, Michigan.

Dynamic Atomic Force Microscopy (AFM) is typically performed at amplitudes that are quite large compared to the measured interaction range. This complicates the data interpretation as measurements become highly non-linear. In the last few years we have successfully employed a new dynamic AFM technique in which deliberately ultra-small amplitudes are used (as low as 0.15 Angstrom). This way we have been able to linearize measurements of nanomechanical phenomena, including single atom bonding, atomic-scale dissipation and molecular ordering in liquid layers, including water. We will introduce the technique, its theory, and recent results in vacuum and liquids

#### 11:30 AM O1.9

**Atomically Resolved Imaging of Epitaxial CaF<sub>2</sub> on Si(111) using Non-contact Atomic Force Microscope.** Yoshihide Seino<sup>1</sup>, Masayuki Abe<sup>1,2</sup> and Seizo Morita<sup>1,2</sup>; <sup>1</sup>Handai Frontier Research Center, Osaka University, Suita, Osaka, Japan; <sup>2</sup>Electronic Engineering, Osaka University, Suita, Osaka, Japan.

Non-contact Atomic force microscope (NC-AFM) enables high resolution imaging of surfaces regardless of sample's electric conductivity. It has been accepted the truth that UHV-NC-AFM had already succeeded in the true atomic resolution imaging for various surfaces of metals, semiconductors and insulators. But the imaging mechanism of the atomic contrast is not yet fully understood. NC-AFM involves many kinds of tip-sample interactions, each with different length-scale. Both short- and long-range interactions between them affect the measured forces but only short-range forces give the atomic features. In view of the sensitivity, not only the adatoms but also the rest-atoms on Si(111)-7x7 and Ge(111)-c(2x8) reconstructed surfaces had been imaged with atomic resolution. In CaF<sub>2</sub>(111) surface of cleaved crystal, combined theoretical and experimental investigations have shown the contribution of several inner atomic layers to the atomic-scale contrast. It is consequently considered that UHV-NC-AFM has achieved almost the equivalent resolution of STM, and UHV-NC-AFM comes out to be an appropriate tool for surface study, especially in the observation of insulator surfaces. Besides, CaF<sub>2</sub>/Si(111) is an ideal object of the semiconductor-on-insulator interfaces, and has also been an appropriate for observing the surface structures by NC-AFM. In this work, epitaxial CaF<sub>2</sub> films grown on Si(111) have been investigated the surface structure using NC-AFM. According to the topographic images, both CaF bilayer and CaF<sub>2</sub> islands were reproducibly imaged with atomic resolution including two kinds of atomic corrugation patterns. It is considered that the atomic corrugation must be dominant by the short-range electrostatic interaction between the tip and sample, and the tip polarity (positive terminated or negative) played an important role for two atomic corrugation patterns. These results were consistent with the case of CaF<sub>2</sub>(111) surface of cleaved crystal, which had already been reported. Furthermore, these results will be discussed in conjunction with the images obtained by electrostatic force microscopy, and Kelvin probe force microscopy. Additionally, we have also performed the surface photo-voltage (SPV) measurements with the aim of investigating the local SPV phenomena at atomic-scale by NC-AFM. According to the atomic configuration adjacent of this interface, the CaF bilayer is known to have almost the same geometry as that of the two uppermost atomic layers of bulk CaF<sub>2</sub>, except that the positions of the F atoms below the Ca layer are vacant. The electronic structure has suggested that there is the existence of a pair of two-dimensional interface bands by LDA calculations. It is, therefore, very interesting to investigate composite effects between the interface and photo-induced carrier dynamics in the Si substrate. The result of our work will be also discussed in comparison with those of Si(111) reconstructed surfaces.

#### 11:45 AM O1.10

**Quantitative Surface Potential Tracking of Organic and Ionic Adsorbates.** Christian Loppacher, Stefan Grafstrom, Ulrich Zerweck, Tobias Otto, Sebastian Teich, Elke Beyreuther and Lukas M. Eng; Institute of Applied Photophysics, TU Dresden, Dresden, Saxony, Germany.

We present an improved setup for deducing quantitative surface potential values by means of Kelvin-probe force microscopy (KPFM) [1]. Our technique bases on a recent development by Kitamura and Iwatsku [2] but allows to decouple topography from surface potential mapping by demodulating the two informations in two independent frequency regimes. Both methods [1,2] are sensitive to the electrostatic force gradient rather than the absolute force and therefore provide a higher lateral resolution compared to other methods, and furthermore are not restricted to be used with soft cantilevers only. We show here, under which conditions KPFM delivers quantitative surface potential values. In order to do so, we experimentally deduce the lateral and vertical dependence of the KPFM signal across a surface potential boundary. These experimental surface potential values are in excellent agreement with calculations based on finite element calculations. The latter needs values on the exact tip-sample geometry which we obtain from scanning electron micrographs. From our detailed analysis we conclude that quantitative surface potentials are obtained with KPFM for objects having a lateral extension in the order of the tip size or larger. More specifically, we apply this method to the sub-monolayer coverage of aromatic molecules, C60 molecules, and potassium chloride films on metallic substrates, but equally to octadecylphosphonic acids adsorbed onto ionic or oxidic substrates. Comparison to macroscopic data and simulations will be given. [1] Ch. Loppacher, U. Zerweck, and L.M. Eng, *Nanotechnology* 15, S9 (2004) [2] S. Kitamura and M. Iwatsuki, *Appl. Phys. Lett.* 72, 3154 (1998)

SESSION O2: SPM-Based Lithography,  
Nanofabrication and Atom Manipulation  
Chair: Bryan D. Huey  
Monday Afternoon, November 29, 2004  
Commonwealth (Sheraton)

#### 1:30 PM O2.1

**Fabrication and Characterization of an Inductance Gate in a Superconducting Flux Flow Transistor Using AFM Lithography.** Seokcheol Ko<sup>1</sup>, Byoung-Sung Han<sup>1</sup>, Hyeong-Gon Kang<sup>2</sup> and Haeseong Lee<sup>3</sup>; <sup>1</sup>Division of Electronics and Information Engineering, Chonbuk National University, Jeonju Chonbuk, 664-14, Duckjin-Dong 1Ga, South Korea; <sup>2</sup>Basic Science Research Institute, Chonbuk National University, Jeonju Chonbuk, 664-14, Duckjin-Dong 1Ga, South Korea; <sup>3</sup>Jeonju Branch of Korea Basic Science Institute, Jeonju Chonbuk, 664-14, Duckjin-Dong 1Ga, South Korea.

Local anodization on Si or superconductor layers can be induced by the electric field between the layers and a tip. This process is useful for lithography which enables us to fabricate nano-scale electronic devices. In this study superconducting flux flow transistor (SFFT) with an inductance gate was designed and fabricated by the AFM anodization lithography. In order to improve the transresistance value, the inductance is fabricated as narrow slits of 50  $\mu\text{m}$  width, 25  $\mu\text{m}$  space and 12 turns, the channels are formed as two serial structure with 10  $\mu\text{m}$  width and 5  $\mu\text{m}$  length. The AFM anodization technique is a maskless process and effective method to overcome the difficulty in fabricating a nano-scale channel. This study presents a new fabrication methodology of inductance gate SFFT with two serial channels using the AFM oxidation process. In the structure of the inductance gate, the critical current density was decreased with an applied current in a gate current line. The calculated values from the current-voltage properties curves were compared with measurements.

#### 1:45 PM O2.2

**Dispersive Kinetics in Atomic Force Microscope Assisted Oxidation of Zirconium Nitride.** Jeffrey R. Comer<sup>1</sup>, Natalia Farkas<sup>1</sup>, Guanghai Zhang<sup>1</sup>, Rex D. Ramsier<sup>1</sup>, Edward A. Evans<sup>1</sup> and John A. Dagata<sup>2</sup>; <sup>1</sup>Physics, Chemistry, and Chemical Engineering, The University of Akron, Akron, Ohio; <sup>2</sup>Precision Engineering Division, National Institute of Standards and Technology, Gaithersburg, Maryland.

We report on the local oxidation kinetics of zirconium and zirconium nitride thin-films by atomic force microscope (AFM) assisted lithography. The addition of various proportions of nitrogen to the sputtering plasma during thin-film deposition modifies the crystal structure and electrical properties of the substrates, which we determine through AFM roughness, X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), and four-point probe measurements. By employing a dispersive kinetics model, we investigate the effects of the altered material properties and a range of applied voltages on

ionic diffusion during oxide growth. Our model predicts the existence of power of time dependent rate constants, where the exponent ( $\gamma-1$ ) is related to the defect density of the material. From experimental data, we determine that  $\gamma$  increases from 1.3 to 2.4 for films deposited with nitrogen flowrates from 0.0 sccm to 5.5 sccm. While the local oxidation kinetics of systems such as silicon are highly subdiffusive, with  $\gamma$  ranging from 0.12 to 0.4, and the features developed tend to be a few tens of nanometers high, the zirconium nitride system displays controlled oxide growth to heights of several hundred nanometers. J. A. Dagata, F. Perez-Murano, G. Abadal, K. Morimoto, T. Inoue, J. Itoh, and H. Yokoyama, *Appl. Phys. Lett.* 76, 2710 (2000). N. Farkas, J. C. Tokash, G. Zhang, E. A. Evans, R. D. Ramsier, and J. A. Dagata, *J. Vac. Sci. Technol. A*, in press (2004). N. Farkas, G. Zhang, K. M. Donnelly, E. A. Evans, R. D. Ramsier, and J. A. Dagata, *Thin Solid Films* 447/448, 468 (2004). A. Plonka, *Prog. Reaction Kinetics* 16, 157-333 (1991).

#### 2:00 PM O2.3

**Electrical AFM Based Xerography for Nanofabrication in Liquids.** Nicola Naujoks and Andreas Stemmer; Nanotechnology Group, Swiss Federal Institute of Technology Zurich, Zurich, Switzerland.

The ability to precisely position molecules at predefined locations onto solid substrates is key to the fabrication of molecular sensors and *chemistryplatforms* where they serve as docking sites or scaffolds for further modification steps. Local surface charges have proven useful to guide the deposition of a variety of materials<sup>1</sup>. We will present sub- $\mu\text{m}$  structures fabricated by a guided assembly process, combining the advantages of a lithography-based top-down approach with the bottom-up self-assembly route. In this universal and highly selective process, various kinds of particles and molecules, ranging from SiO<sub>2</sub> beads<sup>2</sup>, over gold nanoparticles, to biomolecules<sup>3</sup>, are directed to different regions on solid substrates using electrostatic interactions. In our Xerography-like method, water droplets act as vehicles to transport the desired molecules or nanoparticles to charge patterns written into the substrate by a conductive AFM tip. The water droplets are emulsified into an insulating perfluoroalkane liquid to prevent discharging and screening of the surface charges due to the electric double layer present in any ionic liquid. The electrostatic field generated by the surface charges causes a net force of Coulomb and/or dipolar nature on the disperse phase of our emulsion. 330 nm SiO<sub>2</sub> beads are attracted by negative charges on Teflon<sup>2</sup> and repelled by positive ones, implicating Coulomb interaction, whereas on PMMA deposition is observed for both polarities, possibly due to the electrostatic field being weakened by a positive background charging of PMMA against the oil. The phenomenological rule of Coehn<sup>4</sup> predicts such behaviour for the dielectric constants involved ( $\epsilon_{PMMA} \approx 3 > \epsilon_{FC-77} = 1.86$ ). Above example shows how the choice of substrate material influences the balance of dipolar and Coulomb forces experienced by the droplets. In addition to properties of the substrate material a number of parameters related to the development conditions are found to have crucial influence on the deposition results such as surface charge amount, emulsion preparation, especially the use of surfactants, and development duration, to name just a few. In our contribution, we will discuss how to adjust above parameters for optimal feature resolution and process selectivity for different deposited materials. With regard to the fabrication of protein arrays we will further show that biotin-modified immunoglobulin G, attached to positively charged dot patterns on PMMA and air-dried, is capable of selectively binding fluorescently labelled Avidin from aqueous solution. Proving site-specific reactions, these results suggest applications in the field of biosensors and patterned cellular arrays. <sup>1</sup>W.M.D. Wright, D.G. Chetwynd, *Nanotechnology* 9 (1998) 133. <sup>2</sup>P. Mesquida, A. Stemmer, *Adv. Mater.* 13 (2001) 1395. <sup>3</sup>N. Naujoks, A. Stemmer, *Microel. Eng.* 67-68 (2003) 736. <sup>4</sup>A. Coehn, U. Raydt, *Annalen der Physik IV Folge* 30 (1909) 777.

#### 2:15 PM O2.4

**Patterning of Nanoscale Metallic Structures Using Automated Scanned Probe Lithography.** James Battaes and Jayne Garno; Surface and Microanalysis Science Division, NIST, Gaithersburg, Maryland.

The ability to precisely construct nanoscale metallic structures has a wide range of applications from the fabrication and testing of molecular electronic devices to the construction of plasmonic waveguides. Wiring molecules into an electrical circuit by connecting each end to a metal conductor is a key requirement for molecule-based electronics. Challenges exist however for the construction of multidimensional contacts and their controlled organization on surfaces. For device applications it is desirable to reproducibly pattern metal structures on self-assembled monolayers (SAMs) to function as nanoscale connections, with tunable gaps ranging from 1 nm to 100 nm. Herein we report a lithographic method for patterning nanosized copper junctions on SAMs using a combination of automated AFM-based nanofabrication and electroless deposition on carboxylic

acid terminated SAMs. Selective metallization by electroless plating was accomplished for nanopatterns of 16-mercaptohexadecanoic acid, constructed via AFM-based lithography. The deposition of copper can be fine-tuned by varying solution chemistry parameters, such as the immersion intervals and the concentration of metal salts. An effective resist was found using 11-mercaptoundecanoic acid, with very high selectivity observed at the nanometer scale. Writing thiols at different densities within nanopatterns provides exquisite 3-D control in directing copper growth, as demonstrated by systematically changing the writing parameters for arrays generated by automated lithography.

### 2:30 PM O2.5

**Spectroscopic EFM Measurements of Nanoparticle Charge Storage.** Michael Gordon, Sebastien Decossas and Thierry Baron; LTM - CNRS, Grenoble, France.

Nanocrystal (NC) memory devices are currently under investigation as a possible solution to the scaling limitations of Flash memory devices used for non-volatile, high density data storage. In this new technology, an array of Si nanocrystals is embedded in an ultra-thin tunnel oxide to act as a storage node that replaces the more conventional floating gate structure. Therefore, it is important to understand the charging/discharging characteristics and charge retention behavior of individual NCs with respect to dot size and tunnel oxide properties. To this end, we have conducted charging experiments of individual Si and Ge NCs on thermal SiO<sub>2</sub> and high-K dielectrics (HfO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>) on Si using electrostatic force microscopy (EFM) in UHV. Nanocrystals were grown via a ULSI friendly CVD process where the dot density (3E8-1E12/cm<sup>2</sup>) and size (5-50 nm) could be carefully controlled. Quantitative charge imaging was accomplished by comparing both the amplitude change (constant frequency mode) and shift in cantilever resonant frequency (constant amplitude mode) after individual NCs were charged in contact mode with electrons or holes. Although constant frequency mode is not traditionally used in vacuum because of the slow response time for imaging (amplitude relaxation time  $\propto \text{cantilever Q factor} / f_{res}$ ), the high cantilever Q-factor in vacuum can provide enhanced amplitude sensitivity to charging related forces. As such, low levels of stored charge as well as fluctuations due to leakage through the oxide barrier can be measured. By changing the tip bias with respect to dot potential for different lift heights before and after the charging step, one can separate the effects of tip-substrate capacitance ( $(V_{surf}-V_{tip})^2$ ) from dipole-charge forces between the tip and charged nanoparticle ( $Q_{stored}*(V_{surf}-V_{tip})$ ). Specifically, we measure the charging requirements and storage characteristics for a range of Si and Ge nanocrystal sizes by writing individual dots using a biased tip to induce tunnelling through the oxide barrier, followed by spectroscopic EFM.

### 2:45 PM O2.6

**Manipulation of Subsurface Hydrogen and Excitation of Surface Hydrogen Atoms through Inelastic Excitation Mechanisms.** Luis C. Fernandez-Torres, E. Charles H. Sykes, Sanjini U. Nanayakkara, Brent A. Mantooth and Paul S. Weiss; Chemistry and Physics, Pennsylvania State University, University Park, Pennsylvania.

We have observed and manipulated hydrogen atoms beneath the surface of a Pd{111} crystal using low-temperature scanning tunneling microscopy (STM). We demonstrate that the subsurface region of Pd can be populated with hydrogen atoms from the bulk by applying voltage pulses from a STM tip. We present topographic and local electronic data characterizing subsurface hydrogen in these stable sites. We discuss our ability to selectively populate subsurface sites with hydrogen and present a full STM characterization of this state. We explain this phenomenon with an inelastic excitation mechanism, whereby hydrogen atoms in the bulk are excited by tunneling electrons and are promoted to more stable sites in the subsurface region. We also induced the motion of surface hydrogen atoms at 4 K using the STM tip. The motion has been ascribed to inelastic tunneling of electrons; inelastic electron tunneling spectroscopy (IETS) corroborates this assignment, and the surface hydrogen atom onset for motion has been determined.

SESSION O3: Near and Far-Field Spectroscopy of Quantum Structures  
Chair: Lukas Novotny  
Monday Afternoon, November 29, 2004  
Commonwealth (Sheraton)

### 3:30 PM O3.1

**Near field optical scanning microscopy of phase separation effects in diluted nitride alloys.** Alexander Mintairov<sup>1</sup>, James Merz<sup>1</sup>, Kai Sun<sup>1</sup>, Thomas Kosel<sup>1</sup>, Victor Ustinov<sup>2</sup> and Gregory Peake<sup>3</sup>, <sup>1</sup>University of Notre Dame, Notre Dame, Indiana; <sup>2</sup>Ioffe

Physico-Technical Institute, St. Petersburg, Russian Federation; <sup>3</sup>Sandia National Laboratory, Albuquerque, New Mexico.

Dilute nitride alloys (GaAsN, InGaAsN and GaAsSbN) have recently attracted considerable attention as promising materials for laser diodes in the 1.3 -1.5 micrometers range as well as for more efficient solar cells. These applications exploit their unusual electronic property a giant bowing parameter ( $b \approx 20$  eV), which arises from the large electronegativity and the small size of the nitrogen. The resulting large energy scale for statistical composition fluctuation leads to the creation of intrinsic quantum dots (QDs) in these alloys, which have been observed in near-field photoluminescence (NPL) [1]. Here we present images of this QD emission in GaAsN epi-layers and a GaAsSbN quantum well (QW) using low-temperature near-field scanning optical microscopy (NSOM). We studied GaAsN epilayers (thickness 0.1-0.3 micrometers) and a 6 nm GaAsSbN QW grown on (001) semi-insulating GaAs substrates by solid source molecular beam epitaxy and by metal-organic vapor phase epitaxy, respectively. NPL spectra were taken in collection-illumination mode using uncoated fiber tips providing spatial resolution of 300 nm. The spectra were excited by 20 mW from an Ar ion laser. The NPL signal was integrated over 60 s/tip position-pixel over the whole measured spectral range (40 nm). The NPL images shown in this work were made over a range of 11x8 pixels with 200x200 nm size. We have found that monochromatic images of GaAsN epi-layers consist of a series of bright spots having spatial extent of 300-600 nm. Simultaneous analysis of these images and the near-field spectra show that the spatial resolution of these experiments is 300 nm. The larger images, and their observed elongated shapes, are due to the overlapping of different lines. Using these near-field images we observed a strong lateral inhomogeneity in GaAsN epilayers on the length scale of 1000 nm. For the GaAsSbN QW the images of the individual clusters consist of bright stripes having width 400-600 nm oriented along one of the {110} directions. The separation between the stripes is 300 nm. The length of the stripes is greater than 2000 nm. Plan-view transmission electron microscopy measurements showed the presence of composition modulation along the [110] direction having a wavelength  $\approx 100$  nm. The orientation and the lateral dimensions of the composition modulation stripes observed in TEM agree well with NSOM images. However as the NSOM data are related to the emission of individual clusters, the stripe character of the images can be related to the diffusion of photo-generated carriers along the quantum-wire-like channels created by As and Sb phase separation. The very large spatial extent of the images, exceeding a few micrometers, suggests a long life-time of photogenerated carriers, which can be attributed to II-band alignment in GaAsSb. [1] A. M. Mintairov et al, Phys. Rev. Lett. 87, 244701 (2001).

### 3:45 PM O3.2

**Low-Temperature Spectroscopy of Single CdSe Nanorods: Fine Structure and Polarization Properties.** Nicolas Le Thomas<sup>1</sup>, Erik Herz<sup>1</sup>, Oliver Schoeps<sup>1</sup>, Mikhail Artemyev<sup>2</sup>, Wolfgang Langbein<sup>1</sup> and Ulrike K. Woggon<sup>1</sup>; <sup>1</sup>Physics, University Dortmund, Dortmund, Germany; <sup>2</sup>Institute for Physico-Chemical Problems, Belarussian State University, Minsk, Belarus.

Colloidal CdSe nanorods (NR) are nanoemitters that emit highly polarized light and can be used, e.g., as active optical material in laser devices or photonic structures. For these nanocrystals, the exact knowledge of wave function symmetries, energies and optical transition dipole moments is a crucial problem for future applications in quantum optics, laser devices or as polarization sensitive marker or detector. Fine structure splitting as a result of exchange interaction in quantum confined CdSe nanorods and the consequences for the optical spectra are currently less addressed in literature, mainly because room temperature single rod spectra exhibit a large homogenous broadening which prevents the observation of line shape details. In this work we present low-temperature, polarization-sensitive photoluminescence spectroscopy of single CdSe(ZnS) core-shell nanorods. As experimental method we combine low-temperature confocal microscopy with diffraction-limited spectroscopy using a high-numerical aperture (NA=0.85) microscope optics built-in into a He-bath cryostat allowing us a temperature variation from 5K to 300 K. We investigate CdSe nanorods of radius R 2.5nm and length L 25nm dispersed in a polymer film emitting at 610 nm with an inhomogeneous line broadening of 80 meV. The linear degree of polarization (LDP) is recorded instantaneously during the temporal scans of the single nanorod emission by simultaneous measurement of Iparallel and Iperp using a birefringent calcite plate. Our temperature-dependent single rod emission studies revealed a Non-Lorentzian line profile consisting of a sharp (resolution-limited) zero-phonon line (ZPL) with a line width of 300 micro eV and an asymmetric acoustic-phonon side band. At low temperatures T<15 K, the single NR emission spectrum splits into two peaks separated by 1.4 to 2 meV with different polarization properties: a high LDP>0.95 for the higher-energetic structure and a LDP of 0.85 for the low-energetic peak. This fine structure in the single rod emission

spectra we assign to exchange splitting of a 1D-exciton confined in a cylindrical CdSe nanorod. Using an effective-mass model, the observed degree of polarization as well as the temperature-dependent photoluminescence dynamics can be explained based on the revealed ground state exciton symmetry. We find a radius-dependent change in the symmetry of the 1D-exciton ground state which transforms from a dark state for radii  $R$  above a critical nanorod radius of  $R_{crit} \approx 3.7$  nm into optically allowed states for radii  $R < R_{crit}$ . The calculated 1D-exciton ground states symmetries are supported by the measured degree of linear polarization and the temperature-dependence of the photoluminescence dynamics. The observed high degree of linear polarization ( $LDP > 0.95$ ) and the obtained size/shape dependent PL decay times emphasize the important role of exchange interaction for the understanding of the optical properties of CdSe nanorods.

#### 4:00 PM **O3.3**

**Evidence of Multiexciton Emission from Single CdSe/ZnS Colloidal Nanocrystals.** Brent Fisher, Jean-Michel Caruge and Mounji Bawendi; Chemistry, M.I.T., Cambridge, Massachusetts.

We report the results of measurements that uncover multiexciton photoluminescence from single CdSe/ZnS colloidal nanocrystals. Time-correlated single-photon counting measurements of the photoluminescence lifetime from single quantum dots show two well-separated components to the decay. Single photon correlation measurements in a Hanbury-Brown-Twiss detection scheme reveal a suppression of photon anti-bunching from a single quantum dot, due to cascaded photon emission out of multiexciton states. Finally, single QD emission spectra during the first nanosecond after pulsed excitation using a picosecond-gated CCD system reveal triexciton spectral signatures as compared to the spectrum at longer delay times. Although observation of multiexciton and charged exciton emission is now commonplace for single epitaxially fabricated quantum dots, observation of multiexciton emission from single colloidal nanocrystals was thought to be unlikely because of efficient Auger relaxation pathways. Our measurements show that detection of photons from higher excited states of single nanocrystals is, in fact, feasible. This opens up new avenues of spectroscopy for the study of single nanocrystals, without the blurring effects of an ensemble. Furthermore, this result has relevance for proposed optical applications of colloidal nanocrystals, ranging from their suitability as single photon sources, to their use as nonlinear optical media in laser amplifier devices.

#### 4:15 PM **O3.4**

**Evaluation of the Fine Structures of Isolated ZnO Nanorod Single-Quantum-Well Structures using Near-Field Ultraviolet Photoluminescence Spectroscopy.** Takashi Yatsui<sup>1</sup>, Jungshik Lim<sup>2</sup>, Tadashi Kawazoe<sup>1</sup>, Motoichi Ohtsu<sup>1,2,3</sup>, Sung Jin An<sup>4</sup> and Gyu-Chul Yi<sup>4</sup>; <sup>1</sup>SORST, JST, Machida, Tokyo, Japan; <sup>2</sup>Interdisciplinary Graduate School of Science and Engineering, Tokyo Institute of Technology, Yokohama, Kanagawa, Japan; <sup>3</sup>Faculty of Engineering, the University of Tokyo, Bunkyo-ku, Tokyo, Japan; <sup>4</sup>Department of Materials Science and Engineering, POSTECH, Pohang, Kyungbuk, South Korea.

A nanometer-scale ZnO dot is a promising material for realizing nanometer-scale photonic devices at room temperature, due to its large exciton binding energy. Furthermore, recent demonstration of semiconductor nanorod quantum-well structure enables us to fabricate nanometer-scale electronic and photonic devices on single nanorods. Recently, ZnO/ZnMgO nanorod multiple-quantum-well structures (MQWs) were fabricated and the quantum confinement effect of the MQWs was successfully observed. In addition, further improvement in the fabrication of nanorod heterostructures has resulted in the observation of significant PL intensity, even from nanorod single-quantum-well structures (SQWs). To confirm the promising optical properties of individual ZnO/ZnMgO SQWs for realizing nanometer-scale photonic devices, we measured the PL spectrum using a low temperature (15K) near-field optical microscope. In this measurement, we found fine structures of PL spectra from isolated ZnO SQWs. ZnO/ZnMgO SQWs were fabricated on the ends of ZnO nanorods with a mean diameter of 40 nm using catalyst-free metalorganic vapor phase epitaxy. The average concentration of Mg in the ZnMgO layers used in this study was determined to be 0.2. The ZnO well layer thickness  $L$  investigated in this study were 2.5, 3.8, and 5.0 nm, while the thicknesses of the ZnMgO bottom and top barrier layers in the SQWs were fixed at 60 and 18 nm, respectively. After the growth of ZnO/ZnMgO nanorod SQWs on sapphire (0001) substrate, they were dispersed on the substrate to be isolated. A 325-nm light source was used to excite the ZnO/ZnMgO nanorod SQWs. We used UV fiber probe with an aperture diameter of 30 nm. In the near-field spectra obtained at the ZnO nanorod, the single emission peak was observed at 3.365 eV, which correspond to the neutral-donor bound exciton (DOX). However, at the well layer, the emission from DOX was suppressed, while blue-shifted PL emission peak was emerged at 3.393 ( $L = 2.5$  nm), 3.447 ( $L = 3.8$  nm), and

3.499 eV ( $L = 5.0$  nm). The value of the blue shift was consistent with the theoretical prediction using finite square-well potential of the quantum confinement effect in the ZnO well layer. Their spectral width (3 meV) were much narrower than those of far-field spectra (40 meV). The far-field PL spectra measure an ensemble of SQW with size fluctuations, which resulted in inhomogeneously broadened spectral features. However, since the near-field measurement restricts the isolated SQWs, it had made it possible to observe discrete energy levels. The results shown here provide criteria for realizing nano-scale photonic devices, such as the switching devices confirmed by the authors in CuCl quantum cubes.

#### 4:30 PM **\*O3.5**

**Simultaneous near-field Raman and fluorescence spectroscopy of single-walled carbon nanotubes.** Achim Hartschuh<sup>1</sup>, Alfred J. Meixner<sup>1</sup>, Neil Anderson<sup>2</sup> and Lukas Novotny<sup>2</sup>; <sup>1</sup>Physical Chemistry, University of Siegen, Siegen, Germany; <sup>2</sup>The Institute of Optics, University of Rochester, Rochester, New York.

Raman spectroscopy is a powerful tool used extensively for the structural analysis of single-walled carbon nanotubes (SWNT). We present tip-enhanced Raman spectroscopy of SWNT based on the local field enhancement effect at a laser illuminated metal tip and demonstrate Raman imaging with sub 20 nm resolution. The high-resolution capability of the presented method is used to study localized phonon modes and variations in the Raman spectra caused by structural defects which would be hidden in farfield measurements. The technique opens up new insights into the structural properties of SWNT on the nanoscale [1]. Recently, SWNT were found to fluoresce at distinct energies that are determined by their structural parameters [2,3]. Using local field enhancement, near-field fluorescence and Raman images of the same individual tube can be acquired. Fluorescence images on the nanoscale show the spatial extension of the emitting excited state and allow to distinguish between excitonic and localized trap states. Moreover, SWNT offer the unique possibility to quantify and directly compare the enhancement factors for Raman and fluorescence signals achieved under the same experimental conditions. [1] A. Hartschuh et al., Phys. Rev. Lett. 90, 095503 (2003). [2] S.M. Bachilo et. al, Science 298, 2361 (2002). [3] A. Hartschuh et. al, Science 301, 1354 (2003).

SESSION O4: Poster Session

Chair: Bennett Goldberg

Monday Evening, November 29, 2004

8:00 PM

Exhibition Hall D (Hynes)

#### **O4.1**

**Micro-Raman Investigation for Adsorption of Small Molecules on the Surface of Buckyballs and Carbon Nanotubes.** Mostafa M. El-Ashry<sup>1</sup>, Maher Amer<sup>1</sup> and John F. Maguire<sup>2</sup>; <sup>1</sup>Mechanical and Materials Engineering, Wright State University, Dayton, Ohio; <sup>2</sup>AFRL, Dayton, Ohio.

We studied the adsorption of the pressure transmission medium molecules on the surface of the fullerenes  $C_{60}$  and Single Wall Carbon Nanotubes under high hydrostatic pressure. Different mole fractions of water methanol mixtures as a pressure transmission medium were used in the investigation. The adsorption of the water and/or methanol molecules was monitored using Micro-Raman technique. It has been observed that several layers of adsorbate could be monitored when applying pressure. This behavior is observed for different Raman active peaks. It was found that the adsorption rate depends on the compositions of pressure transmission medium. It was also observed that maintaining the sample under high pressure (5 GPa) for long time (over a month) caused hysteresis in the adsorption desorption behavior.

#### **O4.2**

**Fabrication of a NSOM Cantilever Probe Using Prior Process.** Eun-Kyoung Kim, Sung-Q Lee and Kang-Ho Park; Basic Research Laboratory, Electronics and Telecommunications Research Institute, Daejeon, South Korea.

Near-field recording is one of prominent ways to increase the density of optical data storage device. Silicon based NSOM probe is less fragile than fiber type one and the fabrication procedure can be easily extended to an array type probe using silicon batch process. Cantilever type NSOM probe has been prepared with single SOI wafer using conventional MEMS process. First, a SOI wafer with oxide holes was prepared. The holes with areas of 100  $\mu\text{m}^2$  were patterned in the buried silicon oxide layer previous to bonding the SOI wafer. Next, NSOM tips formed on the top side of the SOI wafer using isotropic silicon dry etching. The tips lay just on the prior fabricated holes in the buried oxide layer. And then, the outside of the tip was oxidized except at the apex of the tip for small aperture. A cantilever pattern

was defined on both topside and backside of the SOI wafer and then silicon was removed by DRIE. Without additional process, silicon oxide tips were hollowed through the pre-etched oxide holes at the end of the DRIE process. The prior etching process of holes in the buried oxide layer was useful to remove the silicon inside oxide NSOM tip. Furthermore, this procedure made simplifying the fabrication process and lowering the cost. The upward apertured tip in this work was proper for approaching the NSOM probe to the sample without collision.

**O4.3**  
**Apertureless Head: A Multipurpose Tool Really Combining Atomic Force Microscopy with Powerful Means for Optical Investigations.** Vasily V. Gavriluk, Serguey A. Saunin and Vladimir V. Zhizhimontov; NT-MDT, Zelenograd, Moscow, Russian Federation.

An original instrument intended to integrate the advantages of scanning probe, near and far field optical microscopy is presented. Recent breakthrough in near field microscopy, specifically the realization of subwavelength resolution based on light field concentration under the specially prepared probe tip [1-3] have challenged the development of the unit providing coupling atomic force microscope with high resolution optical means. The device named Apertureless Head (AH) can work as a standard atomic force scanning microscope. Principal optical scheme of the unit is presented at fig.1. Being equipped with high numerical aperture objective, AH allows illumination and observation of the sample in the proximity of the probe and under the cantilever as well. The working distance of objective is large enough to accommodate the cantilever holder with piezo driver in the space between the frontal meniscus and the sample. Laser system monitoring the cantilever deflections and oscillations is equipped with suitable optics allowing its work through the objective. Light signal caused by the laser induced excitation of the sample surface under the probe tip is collected in the backward direction by the same objective for further analysis. Phase plates based polarization handling system provides radial polarization of annular excitation laser beam. Such an arrangement generates a focal electric field with a predominant Ez component suitable for better light field concentration and excitation of the surface enhanced Raman scattering [4,5]. Beam splitter allows optical observation of the sample during the scanning, providing possibility of exact positioning of scanning microscope probe to the object under investigation regardless non transparency of the probe (see fig.2.). The cantilever is seen not sharply at the bottom of photographs as a blurred triangle. Pieces of dust are seen under the probe as if the cantilever being transparent due to the inclined rays collected by the objective. Apertureless head is supposed to be a powerful tool for high resolution analysis in different fields of investigations such as Material sciences (optical and optoelectronic, magnetic, semi- and superconducting materials), Polymers and Biological sciences (structural biology, molecular and cell biology, microbiology, etc.). The presented device was specially designed to be incorporated to Ntegra Scanning Probe Laboratory, the powerful analytical instrumentation in the nanotechnology field produced by NT-MDT Company, Russia. References 1. E.H. Syngé, Philos. Mag. 6 (1928) 356. 2. E. Betzig, J.K. Trautman, T.D. Harris, J.S. Weiner, R.L. Kostelak, Science 251 (1991) 1468. 3. V.N. Kanopsky, Opt. Comm. 185 (2000) 83. 4. B. Sick, B. Hecht, U.P. Wild, L. Novotny, J. of Microscopy 202(2) (2001) 365. 5. N. Huse, A. Sch?nle, S.W. Hell, J of Biomed. Opt. 6(4) (2001) 480.

**O4.4**  
**Nanoscale Antenna Structures for Field Enhancement and their NSOM Characterization.** Xianfan Xu, Eric X. Jin and Sreemanth M.V. Uppuluri; Purdue University, West Lafayette, Indiana.

In this work, we investigate using nanoscale ridge apertures as nanoscale antenna to concentrate light at visible wavelengths into a nanoscale domain. The optical transmission characteristics of ridge apertures in a metal film are first numerically studied using the finite-difference time-domain (FDTD) method. We show that ridge apertures provide optical transmission enhancement compared to conventional square shape nanoscale apertures and confine the transmitted light in the near field to the gap between the ridges due to the fundamental TE<sub>10</sub> propagation mode. We fabricated various ridge apertures and characterize them using a Near-field Scanning Optical Microscope (NSOM). We developed this NSOM based on a commercial Atomic Force Microscope (AFM). A unique procedure is developed to prepare the AFM/NSOM probes. With the illumination of 633 nm HeNe laser, the optical resolution of NSOM imaging better than 40 nm or  $\lambda/15$  is demonstrated by investigating the near-field optical properties of these nanoscale ridge apertures in the transmission-collection mode. Field concentration of these apertures is also demonstrated in a nano-lithography experiment.

**O4.5**  
**Simulation evidence for lateral excitation transfer in quantum**

**dot arrays.** Harley T. Johnson<sup>1</sup>, Ranojoy Bose<sup>1</sup> and Bennett Goldberg<sup>2</sup>; <sup>1</sup>Mechanical & Industrial Engineering, University of Illinois at Urbana-Champaign, Urbana, Illinois; <sup>2</sup>Physics, Boston University, Boston, Massachusetts.

Semiconductor quantum dot arrays are the subject of much interest for applications in optoelectronics, as well as for potential use in nanoelectronics and quantum computing. Most applications are based on the quantum confining properties of individual nanostructures, but the coupled quantum mechanical behavior of multiple quantum dots is important for many advanced applications. By studying groups of 10-100 III-V quantum dots using NSOM, it was found recently that quantum mechanical coupling between dots leads to resonances in the photoluminescence excitation (PLE) spectra. This talk will describe a parallel effort on modeling the quantum mechanical confining properties of groups of quantum dots of disparate size, shape, and spatial ordering. Results of the model explain a mechanism for inter-dot coupling. By use of a single electron kp Hamiltonian formulation, delocalized electron and hole states are found, where coupling between dots is mediated by the wetting layer between the dots. An analysis of overlap between electron and hole states throughout the quantum dot array, with consideration for exciton binding energies, shows that lateral excitation transfer occurs via hole states spread over groups of 3-5 quantum dots. The energetics of the interdot coupling through delocalized wave functions match well with the energy scale of the PLE resonances. Other analyses of the quantum dot array optical spectra, including strain effects, are also in good agreement with experimental observations.

**O4.6**  
**Near-field nano-Raman imaging of Si device structures.** Robert E. Geer and Jacob Atesang; College of Nanoscale Science and Engineering, University at Albany - SUNY, Albany, New York.

Aperture-based and apertureless-based near-field Raman imaging holds the potential for nanoscale stress metrology in emerging Si devices. Although preliminary application of near-field Raman imaging on Si device structures has demonstrated the potential for stress measurement it is necessary to increase the signal-to-noise ratio of the measurement before it can be widely applied. To that end, we present near-field nano-Raman imaging of Si device structures using a modified near-field optical microscope (NSOM). The nano-Raman system utilizes an off-axis (45 degree) backscattering geometry with free-space collection optics. Initial aperture-based implementation on organic calibration films agree with previously published results and are compared to theoretical models for off-axis scattered power. Application to Si/SiN strip test structures reveals a spatial modulation in Raman scattering as expected. These data are compared to the same measurements using an apertureless approach.

**O4.7**  
**Surface structure and work function of Indium-tin-oxide thin (ITO) films for organic light emitting diodes by conducting atomic force microscopy.** Hyunjung Shin, Chanhyung Kim and Bongki Lee; School of Advanced Materials Engineering, Kookmin University, Seoul, South Korea.

Influence of plasma and chemical treatments on the surface structures of Indium-Tin-Oxide (ITO) thin films has been studied by x-ray photoelectron spectroscopy (XPS) and conducting atomic force microscopy (C-AFM). XPS data show significant changes in core level energies of In 3d<sub>5/2</sub>, Sn 3d<sub>5/2</sub>, and O 1s implying the changes in bonding states of these species after each surface chemical and plasma treatments. Local current mapping of ITO surfaces using C-AFM shows two distinct conducting and nonconducting regions at applied bias of 0.5V. In particular, the nonconducting regions was about 10 40 nm in length. Local I-V measurements in conducting regions show an ohmic behavior; whereas the local I-V from the nonconducting regions show unusual one. It implies the presence of a resistive oxide layer with the breakdown voltage of about 7V. It is known to be formed the stable oxides of In<sub>2</sub>O<sub>3</sub> and SnO in the surfaces.(ref, Hehnan Lin et al., JAP, 89, 3976) We confirmed also the formation of In<sub>2</sub>O<sub>3</sub> and SnO by XPS. Consequently, the stable oxides prevent the formation of an ohmic contact between the ITO surfaces and the conducting AFM tip. In O<sub>2</sub> inductively coupled plasma (ICP) treatment, nonconducting regions are increased due to the formation of the stable oxides. Finally, KFM (Kelvin Force microscopy) was used to reveal the regions of the increase of the work function by the stable oxides with high spatial resolution. Our results are of importance for fabrication of the improved organic light emitting diode (OLED), since the surface treatments of ITO films as hole-injecting layers can be done properly.

**O4.8**  
**Investigation of Corona Charge Deposition for Oxide-Semiconductor Interface Characterization Using Kelvin Probe Force Microscopy.** Bert Lagel, Maria Daniela Ayala and

Rudy Schlaf; Electrical Engineering, University of South Florida, Tampa, Florida.

Scanning probe microscopy based electrical characterization methods with nanometer resolution become increasingly important as the downscaling of device dimensions in integrated circuits reaches the sub-100 nm regime. While various powerful characterization methods have become standard tools for wafer-scale monitoring in semiconductor manufacturing, they are typically limited in their spatial resolution. We have investigated corona charge deposition combined with Kelvin Probe Force Microscopy (KPFM) for non-contact, high lateral resolution electrical characterization of semiconductors. The number of semiconductor parameters that can be obtained, such as surface barrier height, surface doping density, oxide thickness or oxide charge density, reveals the versatility of this characterization method. However, currently available commercial devices based on corona charge characterization employ the traditional macroscopic vibrating Kelvin probe suitable for wafer-scale in line monitoring. In order to examine the feasibility of the corona charge deposition method for nanometer scale device characterization we have determined the surface potential distribution of nanometer sized oxide patterns with different thicknesses on Si wafers using KPFM.

**O4.9**  
**A Scanning Tunneling Microscopy Study: Si/SiO<sub>2</sub> Interface Roughness Induced by Chemical Etching.** Jixin Yu<sup>1,2</sup>, Lequn Liu<sup>2</sup> and Joseph W. Lyding<sup>2</sup>; <sup>1</sup>Electrical Engineering, University of South Carolina, Columbia, South Carolina; <sup>2</sup>Beckman Institute, University of Illinois, Urbana, Illinois.

The Si/SiO<sub>2</sub> interface roughness has received tremendous interest due to its relation to channel mobility degradation and dielectric reliability. We have used ultra-high vacuum scanning tunneling microscopy (STM) to directly examine the Si/SiO<sub>2</sub> interface and study the roughening effect caused by chemical etching. Three Si(100)/SiO<sub>2</sub> samples studied were cut from 8-inch wafers that had gone through different surface treatments before 1nm gate oxide growth at 1000C. The rms-roughness extracted quantitatively from the STM topography was found to be doubled from 0.11nm to 0.232nm by the normal SC-1 treatment, and further increased to 0.285nm for additional etching steps, which is consistent with the conclusion inferred from other experiments. It was also found that there were no regular single steps on the Si(100)/SiO<sub>2</sub> interface, even on the samples without SC-1 treatment. An industry standard shallow trench isolation process flow was examined to find out the process steps that affect the Si/SiO<sub>2</sub> interface roughness. Process improvements were proposed to produce a smooth interface with emphases on the pre-oxidation surface preparation and oxidation, which could be easily incorporated into the current shallow trench isolation process flow.

**O4.10**  
**Charging Effects in Silicon Nanocrystals in SiO<sub>2</sub> Layers Observed by Scanning Capacitance Microscopy.** Euykyu Lee<sup>1</sup>, S. H. Jin<sup>1</sup>, Y. Khang<sup>2</sup>, J. M. Kim<sup>3</sup>, Y. S. Kim<sup>3</sup> and Chi Jung Kang<sup>1</sup>; <sup>1</sup>Physics, Myongji University, Yongin, Kyonggido, South Korea; <sup>2</sup>Samsung Advanced Institute of Technology, Suwon, South Korea; <sup>3</sup>Electrical Engineering, Myongji University, Yongin, South Korea.

Scanning capacitance microscopy (SCM) is used for characterizing the charging effects of silicon nanocrystals. The nanocrystal samples produced by aerosol technique have the size of about 2-10 nm. Trapping phenomena of electrons and holes are observed and analyzed through the SCM images and capacitance spectroscopy. These localized properties of each nanocrystal are compared with C-V, I-V characteristics of the conventional MOS capacitor structure. We also present the detrapping process of trapped charge in nanocrystal with changing temperatures and stress conditions.

**O4.11**  
**Electric Force Microscopy of Silicon-On-Insulator.** Emma Rosamond Tevaarwerk<sup>1,2</sup>, D.G. Keppel<sup>1</sup>, Peter Himpel<sup>1</sup>, M.M. Roberts<sup>2</sup>, P. Rugheimer<sup>2</sup>, D.E. Savage<sup>2</sup>, M.G. Lagally<sup>2</sup> and M.A. Eriksson<sup>1</sup>; <sup>1</sup>Physics, University of Wisconsin-Madison, Madison, Wisconsin; <sup>2</sup>Materials Science, University of Wisconsin-Madison, Madison, Wisconsin.

Silicon-on-insulator (SOI) is fast becoming the material of choice for high performance silicon devices. Evaluation of materials quality before the creation of devices is important to SOI manufacturers, and measurement of low-concentrations (less than  $5 \times 10^{15}$  carriers per cubic centimeter) of free carriers in ultra-thin silicon-on-insulator can be difficult [1]. Voltage-modulated EFM has been previously shown to be sensitive to small concentrations of free carriers [2]. Here, we present a method for measuring small carrier concentrations on minimally processed SOI wafers. Ultra-thin SOI is patterned using e-beam lithography into nanostructures of varying surface area,

volume, and perimeter. Using voltage-modulated EFM, we are able to measure free carrier concentration in SOI nanostructures. Our measurements show that some of these nanostructures contain free charge ( $< 5 \times 10^{14}$  carriers per cubic centimeter), and are not fully depleted as would be predicted by simple calculations of depletion widths. [1] S. Henaux, F. Mondon, F. Gusella, I. Kling, and G. Reinbold, *Doping Measurements in Thin Silicon-on-insulator Films*, J. of Electrochemical Society, 146 (7) 2737-2743 (1999). [2] Emma Tevaarwerk, P. Rugheimer, O.M. Castellini, D.G. Keppel, D. E. Savage, M.G. Lagally, and M.A. Eriksson, *Electrically Isolated SiGe QDs*, Appl. Phys. Lett. 80, 4626 (2002).

**O4.12**  
**Tunneling Microscopy of Boroaluminosilicate Glass Rendered Conductive by Irradiation with Electrons and Ultraviolet Light.** Mark Polking and Christopher Cutler Umbach; Materials Science & Eng., Cornell University, Ithaca, New York.

Both photons and electrons incident on a glass surface can promote charge carriers into trap states with energies that lie within the effective band gap of an amorphous solid such as a silicate glass. The surface conductivity is then enhanced when carriers from these states hop to other trap states or are emitted into extended states. It is possible to use scanning probe microscopy to detect the change in the surface conductivity of glasses associated with the thermally-excited emission of carriers from traps. A 40 mW 325 nm He:Cd laser was used to irradiate the fracture surface of a commercial boroaluminosilicate display glass (Corning Code 1737) maintained in ultra-high vacuum at a temperature of 500 C. After this irradiation process, continued heating of the glass releases carriers from traps and results in a surface conductivity much greater than the bulk ionic conductivity. The conductivity can be determined directly by contacting the surface with an etched Si tip of a scanning probe microscope and measuring the current flowing between the tip and deposited surface electrodes; the conductivity has an activation energy of 0.18 eV. With the proper annealing and irradiation conditions, the conductivity is sufficiently large to allow imaging of the surface in the scanning tunneling microscope mode. A larger and much longer-lived surface conductivity can be achieved by irradiation with 600 eV electrons; morphological changes suggest that a restructuring of the glass surface occurs during electron irradiation.

**O4.13**  
**Comparison of Scanning Capacitance Microscopy and Scanning Electron Microscopy for Semiconductor Reverse Engineering.** St. John Dixon-Warren and Lev Klibanov; Chipworks Inc., Ottawa, Ontario, Canada.

Scanning capacitance microscopy (SCM) is a scanning probe microscopy technique that gives spatially resolved information on the distribution of N and P-type carriers employed in a complex semiconductor structure. Quantitative measurement of the carrier concentration is also possible in favorable cases, provided suitable calibration standards are used. We will compare the application of SCM with conventional scanning electron microscopy (SEM) for the reverse engineering of advance semiconductor devices, such as a CMOS image sensors and bipolar transistors. The SEM provides higher spatial resolution and better delineation of the dielectrics and metals, while the SCM provides greater detail on the structure of the semiconductor device. Our results suggest that a complete understanding of the device under study requires the application of both SCM and SEM.

**O4.14**  
**Simultaneous observation of topography and polarization using non-contact scanning nonlinear dielectric microscopy.** Yasuo Cho and Koya Ohara; R.I.E.C. Tohoku Univ., Sendai, Japan.

Recently, we have proposed and developed Scanning Nonlinear Dielectric Microscopy (SNDM) for observing the polarization distribution on ferroelectric materials [1]. Now, its resolution in measuring the polarization distribution is sub-nanometer order [2]. In conventional SNDM technique, however, it is operated in the contact-mode to measure the linear and nonlinear dielectric properties of materials where the point end of the tip directly contacts the surface of the specimen. Therefore, the point end of tip is easily damaged and the resolution of SNDM becomes degraded. In this paper, we newly developed SNDM operating in non-contact mode (nc-SNDM) to avoid a degradation of tip and specimen and to realize a simultaneous observation of the highly resolved topography and the ferroelectric polarization. In this newly invented technique, we applied technique of higher order nonlinear dielectric microscopy to detect and keep the quite short distance between the point end of a SNDM tip and a specimen [3]. The magnitude of the nonlinear dielectric signal markedly varies (decreases) with the quite small variation (increase) of the gap between the tip and the specimen. As the higher order nonlinear dielectric constant ( $\epsilon_{3333}$ : fourth rank tensor) is much



more sensitive against the gap variation than the lowest order nonlinear dielectric constant ( $\epsilon_{333}$ : third rank tensor) and is completely insensitive to the polarization inversion at all, we monitored this  $\epsilon_{333}$  signal and fed back this signal to the z-axis controller in order to keep the gap constant. Simultaneously, the  $\epsilon_{333}$  signal which contains the polarization information is measured. Thus we can measure the polarization distribution and topography, simultaneously using nc-SNDM. Theoretically, the height sensitivity of nc-SNDM is quite high as much as scanning tunneling microscopy. Experimental evaluation of the height sensitivity of nc-SNDM and the domain imaging taken in non-contact mode are also reported. [1] Y. Cho, A. Kirihaara and T. Saeki: Rev. Sci. Instrum. Vol.67 (1996) 2297. [2] H. Odagawa and Y. Cho: Surf. Sci. Vol.463 (2000) L621. [3] Y. Cho and K. Ohara, Appl. Phys. Lett., Vol.79, No.23,(2001) 3842.

#### **04.15**

##### **Evolution of Isolated Ferroelectric Domains in Nanometer Scale.**

Hyunjung Shin<sup>1</sup>, Bongki Lee<sup>1</sup>, Chanhyung Kim<sup>1</sup> and Seung-Hyun Kim<sup>2</sup>; <sup>1</sup>School of Advanced Materials Engineering, Kookmin University, Seoul, South Korea; <sup>2</sup>Inostek Inc, Seoul, South Korea.

Study of domain structure and its evolution is important in understanding the macroscopic behavior of ferroelectric materials for various applications. In this work, we prepared sol-gel PZT (52/48) thin films with about 30, 40 and 50nm in thickness and fabricated ferroelectric grains with various lateral sizes from sub-100 to 500nm by controlling annealing temperature and time. In order to observe evolution of domains, piezo-response force microscopy, based on the detection of the converse piezoelectric effect of the ferroelectric thin film under electric field, has been employed. As a result, we can observe that ferroelectric domains are formed in pyrochlore matrix containing single domains with one preferred direction (Polarization is upward direction parallel to the surface normal). Further, they are developed into complex domain structures with preferred orientation as grain size increases. Switching of single ferroelectric with sub-100nm in diameter was achieved and piezoelectric hysteresis loops were also recorded. Critical aspect ratio of the lateral size (i.e. diameter) to the thickness for the single ferroelectric domain is investigated.

#### **04.16**

##### **Direct Observation of Polar Nanostructures in PLZT**

**Ceramics for Electrooptic Applications.** A. L. Kholkina<sup>1</sup>, V. V. Shvartsman<sup>1</sup> and A. Sternberg<sup>2</sup>; <sup>1</sup>Ceramics and Glass Engineering, University of Aveiro, Aveiro, Portugal; <sup>2</sup>Institute of Solid State Physics, University of Latvia, Riga, Latvia.

The wide range of unique properties of lead lanthanum zirconate titanate (PLZT) ferroelectric materials makes them suitable for electronic applications including electromechanical actuators, multilayer capacitors, electrooptic modulators, and pyroelectric sensors. The compositions with high concentration of La are especially important because they exhibit low remanent polarization ("slim" hysteresis loops behavior) combined with high dielectric permittivity. These compositions belong to so-called ferroelectric relaxors, where the macroscopic ferroelectric order does not develop in the absence of sufficient external electric field and the high dielectric and electromechanical properties are due to formation of nanosized polar regions (polar nanoclusters) with a short-range order. Even though these compositions were intensively investigated in the past, little attention has been paid to the studies of domain structures and its evolution under the variation of external conditions. The common optical methods have limited resolution and are appropriate only for the observation of "normal" domains having the sizes of the order of microns. Recently, the Piezoresponse Force Microscopy (PFM) has been successfully applied for the investigation of ferroelectric materials at the nanoscale. The advantages of the PFM are extremely high spatial resolution (down to few nanometers) and high sensitivity to local polarization, which make this method well suitable to study PLZT ceramics. We report here our results of the investigation of local piezoelectric properties of transparent electrooptic ceramics  $Pb_{1-y}La_y(Zr_{1-x}Ti_x)_{1-y/4}O_3$  ( $y=0.975$ ,  $x=0.35$ ) prepared by hot pressing. Piezoelectric contrast has been clearly observed in this material indicating spatial distribution of polarization with the typical domain size of the order of  $\sim 100$  nm. Irregular domain patterns of the labyrinth type could be linked to the formation of a glassy state where the random electric and stress fields are responsible for the disruption of long-range ferroelectric order. The evolution of the polar structures under the variation of temperature, dc and ac external electric fields of various frequencies was also investigated. Time relaxation of the ferroelectric state induced by poling was studied that allowed to quantify the dynamics of ferroelectric-relaxor phase transition. The nature of the observed phenomena is discussed based on the current understanding of the relaxor state in ferroelectrics and possible effect of PFM instrumentation

#### **04.17**

**Instability of Surface Charges in Ferroelectric Pb(Zr,Ti)O<sub>3</sub> Nanoparticles on SrRuO<sub>3</sub> Layers Observed by Atomic Force Microscopy.** William Jo<sup>1,2</sup> and J. H. Hur<sup>2</sup>; <sup>1</sup>Department of Physics, Ewha Womans University, Seoul, South Korea; <sup>2</sup>Division of Nanosciences, Ewha Womans University, Seoul, South Korea.

We report results on domain retention in preferentially oriented Pb(Zr,Ti)O<sub>3</sub> nanoparticles on SrRuO<sub>3</sub> electrode layers. Effects of bottom electrodes on domain images and retention properties have been explored by detecting an electrostatic force exerted on the biased conductive probe. Surface morphology and domains in the nanoparticles were observed using atomic force microscopy with the lock-in amplification technique. We observe a direct correlation between domain configurations and microstructural features in the as-grown particles. After poled by an external dc voltage, the surface shows a domain image independent of protrusions at the non-contact mode of electrostatic force microscopy. Moreover, charge retention was controlled by reverse-poling protocol during electrostatic force microscopy measurements. The surface charge density of the Pb(Zr,Ti)O<sub>3</sub> particles was observed as a function of time in a selected area where a region is single-poled and another region is reverse-poled. Retention behaviors of the regions are very different; the single-poled region shows a declined response and the reverse-poled region reveals a retained characteristic. Decay and retention mechanisms are explained by space-charge redistribution and trapping of defects in the nanoparticles.

#### **04.18**

**Abstract Withdrawn**

SESSION O5: Tip-Enhanced Nano-Optics

Chair: Bennett Goldberg

Tuesday Morning, November 30, 2004

Commonwealth (Sheraton)

#### **8:30 AM \*O5.1**

**Single emitters coupled to optical antennas.** Javad N. Farahani, Peter Muehlschlegel, Dieter W. Pohl, Hans J. Eisler and Bert Hecht; Inst Physik, Univ of Basel, Basel, Switzerland.

Single emitters, such as fluorescent molecules or quantum dots, usually are strongly quenched in the proximity of metal (nano) structures. This effect is detrimental for all kinds of scanning near-field optical microscopy techniques that e.g. strive to resolve single emitter labels with metallized optical probes. Coupling of a single emitter to an antenna structure can overcome this problem. We will discuss the properties of antenna structures at optical frequencies and demonstrate their applications in scanning near-field optical microscopy of single quantum dots and ultra sensitive optical sensor applications.

#### **9:00 AM O5.2**

**Optical near-field enhancement around lithographic metallic nanostructures using an azo-dye polymer: direct observation and realization of sub-wavelength complex structures.**

Christophe Hubert, Sergei Kostcheev, Gilles Lerondel, Alexandre Vial, Renaud Bachelot and Pascal Royer; Laboratoire de Nanotechnologie et d'Instrumentation Optique, Université de Technologie de Troyes, CNRS (FRE2761), 12 rue Marie Curie, BP 2060, 10010 Troyes Cedex, Troyes, France.

We recently report on the direct observation of optical near-field enhancement around metallic nanoparticles. This easy to set up approach consists in the uniform irradiation of a photosensitive azo-dye polymer spin-coated on metallic nanostructures and leads to a topographic modification of the polymer film surface. Comparisons between atomic force microscopy (AFM) cross sections and numerical simulations indicate that these topographic modifications can be associated to the near-field intensity distribution around the nano-structures. Characterization of the lightning rod optical effect and lithographic applications to the generation of sub-wavelength complex structures will be here emphasized. More particularly, results obtained using silver and gold nanostructures with different shape and arrangements will be presented. Influence of experimental parameters such as the light polarization direction or the irradiation wavelength is demonstrated and the realization of sub-wavelength complex structures is shown. Based on the photosensitive properties of azo-dye molecules, this so called probeless approach which does not require the use of any probe leads to a better understanding of the material response at a nanometer scale as well as the optical near-field distribution around metallic nanostructures. Full control of this process together with further possible generation of more complex structures could moreover open the way to a new easy-to-set micro and nano-structuring technique.

### 9:15 AM O5.3

#### Subwavelength-Resolution Raman Microscopy of Si Structures Using Metal-Particle-Topped AFM Probe.

Vladimir Poborchii, Tetsuya Tada and Toshihiko Kanayama; MIRAI, Advanced Semiconductor Research Center, National Institute of Advanced Industrial Science and Technology, Tsukuba, Japan.

Performance of Si transistor is strongly affected by mechanical stress in the device area. Miniaturization of the device tends to cause the stress. In particular, the use of shallow trench isolation (STI) and Si-on-insulator (SOI) structures often results in stress in the transistor area. Stress in Si can be monitored by Raman spectroscopy but to meet the device shrinkage requirement, the spatial resolution should be well beyond the diffraction limit of light. Though optical spectroscopy beyond diffraction limit had made remarkable progress in the last decade, the subwavelength-resolution Raman microscopy of solids is still a challenge. We have developed a method that enables us to obtain Raman spectra (RS) of Si with a spatial resolution much better than the wavelength. The method is based on the fact that the light is partly depolarized when it is scattered on a small metal particle (except for highly symmetric shapes). We use forbidden polarization configuration for the 1-st order Raman band of Si at 520 cm<sup>-1</sup> under a conventional microscope to suppress the intensity of this band. Then an Ag particle of 50 to 100 nm size is introduced into the illuminated area, giving rise to the intensity of this band. This signal is associated with the light elastically scattered and depolarized by the Ag particle and then inelastically scattered in small area of Si just around the particle. Another point is utilization of the near-UV light of 364 nm wavelength for the excitation. Penetration depth of the light to Si is only 12 nm making the excitation area highly localized, and yet the Raman cross-section very large owing to the resonance with the direct electron transitions in Si. We have actually realized this scheme using the quartz AFM probe with an Ag particle on top. The probe was immersed into a small droplet of glycerol with refraction index equal to that of quartz minimizing scattering by the probe stem. We applied this method to various Si device structures and demonstrated stress measurements with 100 nm spatial resolution; e.g. observation of compression extending to 1 micron around an STI structure and gradual increase of strain from the edge to the interior part of the strained Si on Si/Ge island.

### 9:30 AM \*O5.4

#### Near-field Optical Interactions and Spectroscopy.

Lukas Novotny, The Institute of Optics, University of Rochester, Rochester, New York.

The field enhancement at a laser-irradiated metal tip defines a strongly confined photon source. We use this localized source for nanoscale spectroscopy. A spectroscopic optical image of a sample is recorded by raster-scanning the sample underneath the laser-irradiated metal tip and detecting the spectroscopic response for every sample pixel. We show that this technique is able to achieve spatial resolutions in the order of 10 nm, the diameter of the metal tip. Many different interaction mechanisms like second-harmonic (SH) generation, two-photon excited fluorescence, or Raman scattering have been investigated and examples of these studies will be presented. We will discuss the possibility of performing nanoscale absorption spectroscopy with single molecule sensitivity by making use of the localized SH generation at the metal tip. Because of thermal and zero-point fluctuations the tip interacts with the sample even in absence of external irradiation. These fluctuations give rise to van der Waals attraction and to friction forces if tip and sample are in relative motion. We show that this 'electromagnetic friction' depends sensitively on the dielectric properties of the sample material and that it can be used for nanoscale subsurface imaging in metals.

### 10:30 AM \*O5.5

**Nanoscale resolved optical microscopy and infrared spectroscopy by light scattering from a tip.** Rainer Hillenbrand, Nano-Photonics Group, Max-Planck-Institute for Biochemie, Martinsried, Germany.

We demonstrate that (apertureless) scattering-type scanning near-field optical microscopy (s-SNOM) allows optical imaging at 10 nm spatial resolution [1]. In our s-SNOM the tip apex of an atomic force microscope (AFM) is illuminated either by a HeNe laser at 633 nm or a CO<sub>2</sub> laser at about 10 micrometer wavelength. The light scattered from the tip in near-field interaction with the sample surface is detected interferometrically and allows material specific imaging at a spatial resolution independent of the wavelength or to visualize the optical eigenfield patterns of plasmon-resonant nanoparticles [2]. Probing a SiC sample with our s-SNOM at mid-infrared wavelengths we observe that the optical near-field interaction between tip and a flat surface can be strongly enhanced due to near-field excitation of lattice vibrations, more precisely by phonon polaritons. Near-field spectroscopy in the range 9.2 - 11.2 micrometer yields a resonant

light-matter near-field interaction of unprecedented dynamic range and spectral sharpness [3]. This phonon-enhanced near-field interaction is not only sensitive to the local chemical composition but also to the local structural properties of a surface and thus allows besides chemical microscopy also mapping of crystal quality at nanoscale resolution. Altogether we envisage applications in optical nanospectroscopy and imaging of physical, chemical and biological nanocomposites. A more general view of surface phonon polaritons in nanostructures suggests phonon photonics, an infrared nanotechnology for manipulating, guiding and controlling infrared light in nanoscale devices. [1] R. Hillenbrand and F. Keilmann, Appl. Phys. Lett. 80, 25 (2002). [2] R. Hillenbrand et.al., Appl. Phys. Lett. 83, 368 (2003). [3] R. Hillenbrand, T. Taubner, and F. Keilmann, Nature 418, 159 (2002).

### 11:00 AM O5.6

**Surface plasmon enhanced luminescence of individual gold and copper nanoparticle aggregates.** Alfred J. Meixner, Achim Hartschuh, Matias Steiner and Christina Debus; Physical Chemistry, University of Siegen, Siegen, Germany.

In the last years a wide range of optical applications came up for noble metal nanoparticles in biology, medicine, chemistry and physics. Luminescence spectra of gold nanoparticle aggregates were recorded and compared with their light scattering spectra. A substantial enhancement of the luminescence by surface plasmon resonance was found. The luminescence enhancement of the aggregates was red-shifted with respect to the bulk and had maxima close to those of the respective plasmon resonances. Enhancement factors reached up to three orders of magnitude. The resonance behaviour of the closely packed almost two dimensional aggregates can be very well described by the plasmon resonance of oblate ellipsoids in the quasi static approximation. Most of the aggregates with strong luminescent enhancement also showed a considerable SERS-activity. Copper nanoparticle aggregates show a similar behaviour with a pronounced red-shift of the emission maximum and a substantial enhancement of the luminescence yield with respect to the bulk material.

### 11:15 AM O5.7

#### Structured Light near microgratings: a numerical study.

Wolfgang S. Bacsá<sup>1</sup>, Benjamin I. Levine<sup>1</sup>, Michel Caumont<sup>1</sup> and Benjamin Dwir<sup>2</sup>; <sup>1</sup>LPST, IRSAMC CNRS, University of Toulouse, 31062 Toulouse, France; <sup>2</sup>Laboratory of Nanostructures, EPFL, 1015 Lausanne, Switzerland.

The optical field variation in the proximity of surfaces depend on their structure and composition. Space and time coherence of the scattered field leads to constructive and destructive interference resulting in considerable local field variations. A detailed examination shows that there are several phenomena taking place side by side which depend on the distance between surface and image plane. We explain the observed phenomena in terms of coherent diffuse scattering, specular reflection, diffraction and self-imaging. We have calculated the local optical field variations in the proximity of microgratings using asymmetric illumination in order to compare them with experimental images recorded with a scanned optical fibre in external illumination mode. We compare the lateral resolution or reproduction of the regular grating structure for the two main orientations of the grating with respect of the incident beam and as a function of angle of incidence. Two distinct zones can be distinguished: one zone where the grating structure is well reproduced and a second zone where diffraction fringes from the finite size of the grating dominate the image. The appearance of the two zones can be explained in terms of isotropic diffuse scattering and displacement of the diffracted image with increasing distance from the surface. The image contrast is reversed as the image height is changed revealing the interferometric nature of the image. The zone which reproduces well the grating structure is not free of diffraction fringes which are visible particularly well on the grating groove edges. The differences of the two zones correspond to the zones outside and inside of the scattering cone defined by the angle and direction of incidence. We will discuss the results of the numerical study as a function of grating constant, angle and direction of incidence, image height and polarisation, and compare them to experimental images.

### 11:30 AM O5.8

#### Far-Field Optical Microscopy with Nanometer-Scale Resolution Based on the In-Plane Image Magnification by Surface Plasmon Polaritons.

Igor I. Smolyaninov<sup>1</sup>, Jill Elliott<sup>2</sup>, Anatoly V. Zayats<sup>2</sup> and Christopher C. Davis<sup>1</sup>; <sup>1</sup>ECE, University of Maryland, College Park, Maryland; <sup>2</sup>School of Mathematics and Physics, The Queen's University of Belfast, Belfast, United Kingdom.

A new far-field optical microscopy approach capable of reaching nanometer-scale resolution is developed using the in-plane image magnification by surface plasmon polaritons. This approach is based on the optical properties of a metal-dielectric interface that may provide extremely large values of the effective refractive index  $n_{eff}$

up to  $10^3$  as seen by surface polaritons and thus the theoretical diffraction limit on resolution  $\lambda/2n_{eff}$ . The experimental realization of the microscope has demonstrated the optical resolution better than 60 nm for 515 nm illumination wavelength. Applications of this technique to bioimaging and sub-100 nm lithography will be discussed.

SESSION O6: SPM of Semiconductor Materials  
Chair: Arthur Baddorf  
Tuesday Afternoon, November 30, 2004  
Commonwealth (Sheraton)

**1:30 PM \*O6.1**

**Sub-Nanometer Resolution Dopant Profiling in Si and Ge-Based Nanoscale Devices.** Wolfgang Vandervorst, Pierre Eyben, David Alvarez and Marc Fouchier; IMEC, Leuven, Belgium.

Dopant profile engineering for sub-45 nm devices has become an increasingly difficult task as simple one-dimensional profiles/simulations are no longer sufficient to describe the actual material interactions. As such, true two-dimensional dopant profiling with high concentration sensitivity and quantification accuracy has become essential in technology development. Scanning spreading resistance microscopy (SSRM) has emerged as one of the sole tools suitable for this purpose as it combines sub-nm spatial resolution with extreme concentration sensitivity over 5-6 orders of magnitude and quantification accuracy. This progress has been achieved through the successful development of dedicated solid diamond tips, pulsed force measurement procedures and a thorough understanding of the tip-semiconductor contact. Despite their apparent bluntness, as they are made with the moulding technique, these diamond tips prove to provide a very high spatial resolution. Dedicated test structures have been developed to probe the electrical tip radius which is as small as 0.5 nm although the mechanical radius is much larger. Finite element simulations confirm that this small electrical tip is indeed only related to a very localized phase transformation in Si limiting the effective electrical tip radius to a region much smaller than the mechanical radius. The latter is one of the main reasons why SSRM outperforms other concepts such as Scanning capacitance microscopy with an order of magnitude in resolution. Due to the high spatial resolution the sampling volume is very limited and a direct conversion from resistance to resistivity can be obtained using a calibration curve. The reproducibility of this quantification procedure is shown to be better than 10-20% across the entire dopant range of interest. Finally it needs to be stressed that SSRM is the sole technique with sufficient dopant sensitivity to probe the very small variations induced by for instance the very important halo-implants and thus also able to study their diffusion and activation in devices. This superior resolution enables detailed analysis of lateral diffusion, halo-implants and SPER-junctions in very small devices with a demonstrated sensitivity to junction position differences of 1-2 nm to various process variations in line with changes in device performance. For applications towards failure, analysis of any transistor becomes possible through the sample preparation scheme based on contacting all the dopant layers with focused ion beam metallization. Recently the application of SSRM has also been extended to the analysis of dopant profiles in Ge-devices as these are emerging as the replacement for Si in the near future. Again examples of the essential support of SSRM towards dopant profile engineering in Ge-devices will be illustrated.

**2:00 PM O6.2**

**Radiation Induced Subsurface Charging in the Buried Oxide Layer in SIMOX.** Marion Stevens-Kalceff, School of Physics, University of New South Wales, Sydney, New South Wales, Australia.

Silicon-on-insulator (SOI) wafers can be fabricated using the Separation by IMplantation of OXYgen (SIMOX) process: Oxygen ions are implanted into the silicon substrate which is then annealed to form a uniform buried silicon dioxide layer with low defect density. SIMOX wafers are typically used in the fabrication of high-performance and low power CMOS and MEMS devices. Insulating silicon dioxide strongly charges when irradiated with an electron beam in a scanning electron microscope. Irradiation of semiconducting silicon under identical conditions results in negligible charging at 295K. Kelvin Probe Microscopy has been used to characterize the magnitude and spatial distribution of residual potential in electron irradiated SIMOX. The SIMOX specimens investigated have a 395nm thick buried oxide layer capped with a 220nm thick silicon layer. The specimens were implanted with a stationary continuous normal incident electron beam for a range of electron beam energies and doses. Focussed electron beam irradiation induces trapped charge within the insulating buried oxide layer which produces highly localized electric fields. The charging processes are dynamic, localized, and dependent on pre-existing and irradiation induced defect concentrations. The reproducible characteristic potentials associated with the charge trapped in the buried oxide

layer have been measured using Kelvin Probe Microscopy in controlled ambient conditions. Potentials of typically several hundred mV are measured; however screening effects limit the determination of absolute potentials. The characteristic experimental surface potential distributions are compared with model surface potential distributions calculated using three-dimensional conformal Finite Element Analysis. This work demonstrates that proximal probe techniques can be used to investigate subsurface properties and gives insight into the subsurface charging processes in buried insulating layers.

**2:15 PM O6.3**

**Measurement of Doping Profile and Visualization of Surface States in Si using Kelvin Force Microscopy (KFM): Optimization for the Fabrication of Resistive Probe.** Hyunjung Shin<sup>1</sup>, Bongki Lee<sup>1</sup>, Hongsik Park<sup>2</sup>, Dong-Ki Min<sup>2</sup>, Juwhan Jung<sup>2</sup>, Sungdong Kim<sup>2</sup> and Seungbum Hong<sup>2</sup>; <sup>1</sup>School of Advanced Materials Engineering, Kookmin University, Seoul, South Korea; <sup>2</sup>Storage Laboratory, Samsung Advanced Institute of Technology, Suwon, South Korea.

In recent, scanning resistive probe microscopy (SRPM)<sup>1</sup>, as a variant of SPM-based techniques, which has a semiconductor resistor at the apex of the tip and can observe surface charges directly, was newly proposed and fabricated. Spatial resolution of SRPM is dependent upon the size of the prepared resistor at the apex. The size of the resistor can be determined by width (or channel) of the SiO<sub>2</sub> implant mask, where both sides of the mask in p-type silicon substrate were opened, implanted with As<sup>+</sup> ions and diffused by activation process at 1000 degrees for 10 to 16 hours. Using Kelvin Force Microscopy (KFM), we investigated the area of the resistor or equivalently underneath of the mask and determined the diffusion length of implanted As<sup>+</sup> ions. As a result, the contact potential difference between implanted n<sup>+</sup> and p (Si substrate) regions, which is equal to the resistive region, was decreased by increasing diffusion time. As annealing time increases from 10 to 16 hours, it caused shrinkage of the width (or channel) due to out-diffusion of the implanted ions. In conclusion, we show the lowering barrier height similar to punch-through effect in bipolar junction occurs in resistive region using the plot of normalized potential across the width (or channel) a function of annealing time. Furthermore, we investigated the influence of surface state on p/n<sup>+</sup> potential contrast dependent upon the existence of thermal oxide, which suggests surface state density causes surface Fermi level to be pinned. The measured p/n<sup>+</sup> potential images are reproducible and semi-quantitative when the surface states were passivated properly. 1. H. Park, J. Jung, D.-K. Min, S. Kim, S. Hong, and H. Shin, "Scanning Resistive Probe Microscopy: Imaging Ferroelectric Domains", *Appl. Phys. Lett.*, 84, 1734-1736 (2004)

**2:30 PM O6.4**

**Novel Filamentary Structures Discovered in the Quantum Hall Liquid using Scanning Charge Accumulation Imaging.** Gary Steele<sup>1</sup>, Ray Ashoori<sup>1</sup>, Loren N. Pfeiffer<sup>2</sup> and Ken West<sup>2</sup>; <sup>1</sup>Physics, MIT, Cambridge, Massachusetts; <sup>2</sup>Lucent Technologies, Murray Hill, New Jersey.

We present Scanning Charge Accumulation (SCA) imaging data measuring novel spatial structure in the Quantum Hall Effect in a GaAs two dimensional electron gas (2DEG) 100 nm beneath the sample surface. SCA measures the local charging of materials using a scanning metallic tip. A small AC excitation in the 10kHz to 2MHz range is applied to the sample and the tip scans 10 nm above the sample surface. Attached to the tip is a charge detector with an extremely high sensitivity (0.01 electrons/rHz). Measurement of the phase and amplitude of the image charge induced on the tip directly reflects charging in the 2DEG, permitting us to extract the local density of states and the local resistivity of the 2DEG. We use the SCA probe to study the local properties of the Quantum Hall Effect (QHE). Under application of large magnetic fields (10 Tesla) at very low temperatures (300 mK), the 2DEG is expected to show rich local structure. In the presence of disorder, the 2DEG will break up spatially into compressible and incompressible regions. Theorists also predict that at certain magnetic fields the 2DEG will spontaneously form ordered bubble and stripe structures. In this talk, we present recent and very striking images obtained using our technique. The images display an unexpected and intricate network of filaments. The filaments appear only in a narrow range of magnetic fields near integer filling factors and evolve continuously as the magnetic field and tip bias are varied. The observed structure is entirely reproducible upon returning to the same experimental parameters. The filamentary pattern is formed when a bias is applied to the tip that pushes the local density under the towards a filled Landau level. This forms a "bubble" of compressible region under the tip that is separated from the rest of the 2DEG by an incompressible strip that acts as a tunnel barrier to charge moving in and out of the bubble. This tunnel barrier slows down the rate at which the enclosed area under the tip can be charged and discharged. This results in an overall darkening of the image at positions where this strip forms. The filaments then appear

as sharp and bright lines penetrating this dark region. The filaments also have an extreme aspect ratio, being often several microns long, but with widths down to the magnetic length (15 nm), the smallest length scale in the physics of the 2DEG. This is much smaller than our expected resolution, which should be limited by the 100 nm depth of the 2DEG below the surface. Equally puzzling is the spatial structure of the filaments. They show no well defined periodicity, but have a strong orientational selectivity, following preferential directions along crystallographic axes of the host GaAs crystal. The data suggest that interactions between the tip and the 2DEG greatly enhance the resolution of the probe. We present ideas for how this occurs and conjectures for the origin of these astonishing features.

SESSION O7: SPM Characterization of Ferroelectrics  
and High-k Materials  
Chair: Lucas M. Eng  
Tuesday Afternoon, November 30, 2004  
Commonwealth (Sheraton)

### 3:15 PM \*O7.1

#### Progress in Nanoscale Studies of Ferroelectrics.

Alexei Gruverman<sup>2</sup>, Brian J. Rodriguez<sup>1</sup>, Angus I. Kingon<sup>2</sup> and Robert J. Nemanich<sup>1</sup>; <sup>1</sup>Physics, North Carolina State University, Raleigh, North Carolina; <sup>2</sup>Materials Science and Engineering, North Carolina State University, Raleigh, North Carolina.

Scanning probe microscopy (SPM) has revolutionized ferroelectric research of the last decade. It provides a unique opportunity to measure local ferroelectric properties, to tailor and engineer these properties and to characterize nanoscale electronic devices. In this paper, recent advances in understanding the physical properties of ferroelectric materials at the nanoscale made possible by the use of SPM techniques will be reviewed. This information has been used as a basis to study the basic mechanisms of polarization reversal as well as the mechanisms of ferroelectric fatigue and imprint in thin films and capacitors. Investigation of the scaling effect on variability of switching parameters has been used to predict the required level of performance reliability. Progress in tailoring electrical, mechanical and optical properties of ferroelectrics with a viewpoint of developing novel nanofabrication approaches and nanoelectronic devices will be discussed. Support provided by the National Science Foundation (Grant No. DMR-0235632).

### 3:45 PM O7.2

#### Domain Growth Scenario and its Implication to High Density Storage Device. Seungbum Hong<sup>1</sup>, Yunseok Kim<sup>2</sup>, Seung-Hyun

Kim<sup>3</sup>, Hongsik Park<sup>1</sup>, Hyoungsoo Ko<sup>1</sup>, Dong-Ki Min<sup>1</sup>, Juhwan Jung<sup>1</sup>, Chulmin Park<sup>1</sup>, Kyounglock Baek<sup>1</sup>, Sungdong Kim<sup>1</sup>, Eunsik Kim<sup>1</sup>, Yong-Su Kim<sup>1</sup>, Kwangsoo No<sup>2</sup> and Noyeol Park<sup>1</sup>; <sup>1</sup>Storage Lab, Samsung Advanced Institute of Technology, Suwon, Kyonggi, South Korea; <sup>2</sup>Department of Materials Science and Engineering, Korea Advanced Institute of Science and Technology, Daejeon, South Korea; <sup>3</sup>INOSTEK, Ansan, South Korea.

We present the ferroelectric material issues for high density probe storage with a focus on domain growth scenario and its implication to the device performance, i.e. ultimate recording density and read/write speed. Grain size effect is coupled with composition variation which makes the analysis difficult, so a qualitative trend will be briefly discussed based on the assumption that composition variation is of minor importance to bit size. The domain scenario where forward domain growth is limiting step for switching is studied on both top electrode and bare ferroelectric thin films by piezoelectric force microscopy. As evidenced by ring structure and domain image evolution as a function of pulse voltage, the scenario is believed to be applicable to our system. This implies that as the bit size and film thickness scale down, the switching speed increases which enhances the competitiveness of ferroelectric thin film as a candidate material for high density probe storage.

### 4:00 PM O7.3

#### Absolute Measurement of Three-dimensional Polarization Direction using Scanning Nonlinear Dielectric Microscopy. Yasuo Cho, Tomoyuki Sugihara and Hiroyuki Odagawa, R.I.E.C. Tohoku Univ., Sendai, Japan.

Recently, ferroelectric materials have attracted a lot of attention in various fields such as FRAM and ultra-high density data storage [1]. Measurement of the ferroelectric polarization distribution is very important subject of research. We have developed a Scanning Nonlinear Dielectric Microscopy (SNDM) which can observe the polarization distribution with sub-nanometer resolution [2]. SNDM can measure the polarization component perpendicular to the surface by applying electric field in the perpendicular direction. Moreover, we have succeeded in observing the polarization component parallel to

the surface by applying electric field in the parallel direction [3]. In this study, we developed new method for measuring the absolute value of three-dimensional polarization direction using SNDM. At first, we developed a new system that makes more exact measurement for an azimuth angle ( $\phi$ ) of polarization component parallel to the surface without an error caused by a residual normal component of electric field. This was achieved by adding a normal component field canceling system to the conventional 3-D type SNDM [3]. Second, we developed new experimental procedure determining the elevation angle ( $\pi/2-\theta$ ) of the polarization using the modified normal polarization component measurement. Thus, combining these two methods, we can determine the absolute three-dimensional polarization direction. Using this new system, the polarization distribution of periodically poled LiNbO<sub>3</sub> (PPLN) in three-dimension was observed and it was successfully demonstrated that the three-dimensional polarization direction was determined precisely. Moreover, we also report nanoscale three-dimensional polarization measurements for several materials obtained by using this new developed 3-D type SNDM. [1] Y. Cho, K. Fujimoto, Y. Hiranaga, Y. Wagatsuma, A. Onoe, K. Terabe and K. Kitamura, Appl. Phys. Lett., Vol.81(2002) 4401. [2] H. Odagawa and Y. Cho, Surf. Sci. Vol.463(2000) L621. [3] H. Odagawa and Y. Cho, Appl. Phys. Lett., Vol.80 (2002) 2159.

### 4:15 PM O7.4

#### Nanoscale Piezoelectric Imaging and Modeling of a Single Antiparallel Ferroelectric Domain Wall. David Scrymgeour and Venkat Gopalan; Materials Research Institute, Penn State University, University Park, Pennsylvania.

Surprising *asymmetry* in the local electromechanical response across a single well-defined antiparallel ferroelectric domain wall is reported in lithium niobate. This material has established itself as a vital material in the field of nonlinear and electro-optics. Precisely patterning these ferroelectric domain structures on small length scales is a key to creating optical devices for laser frequency conversion and electro-optic modulation. However, despite much study, a fundamental understanding of the local structure of domain walls, which underpins device creation, is still elusive. Piezoelectric force microscopy (PFM) is used to investigate both the *in-plane* and two different *out-of-plane* electromechanical signals across a single 180° domain wall in congruent and near-stoichiometric lithium niobate. A combination of three-dimensional electrostatic field simulations and finite element method modeling is used to simulate in detail, the electromechanical response at the wall and reconstruct the PFM images. Excellent agreement between the experiments and simulations is found in both form and magnitude. The observed asymmetry is shown to have a strong correlation to crystal stoichiometry, pointing to the importance of defect-domain wall interactions.

### 4:30 PM O7.5

#### Effect of Nanostructuring on the Piezoelectric Responses of 0.67 Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-0.33 PbTiO<sub>3</sub> Ferroelectric Films Engineered for MEMS Applications. Jun Ouyang<sup>1</sup>, Dong Min

Kim<sup>3</sup>, Zhengkun Ma<sup>1</sup>, V. Nagarajan<sup>4,1</sup>, J. Melngailis<sup>1</sup>, C.-B. Eom<sup>3</sup>, A. L. Roytburd<sup>1</sup> and R. Ramesh<sup>2,1</sup>; <sup>1</sup>Materials Research Science and Engineering Center, Department of Materials Science and Engineering, University of Maryland, College Park, Maryland; <sup>2</sup>Dept. of Materials Science and Engineering, University of California, Berkeley, California; <sup>3</sup>Department of Materials Science and Engineering, University of Wisconsin, Madison, Wisconsin; <sup>4</sup>Institute for Electronic Materials, IFF, Forschungszentrum, Julich, D-42425, Germany.

There are increasing interests in fabricating piezoelectric film-based microelectro-mechanical systems (MEMS), which include the integration of piezoelectric components to silicon and nanostructuring them to devices with semi-micron size. Here we report a systematic study of piezoresponse microscopy on the longitudinal piezoelectric constants ( $d_{33}$ ) of rhombohedral 0.67Pb(Mg<sub>1/3</sub>Nb<sub>2/3</sub>)O<sub>3</sub>-0.33PbTiO<sub>3</sub> ferroelectrics films, which in single crystal form is known for its giant piezoelectric response. These films were engineered on domain patterns by choosing different substrates and film orientations and nanostructured by a focus ion beam process. It was found that as high as 1200pm/V can be realized for (001) oriented films grown on Si, which is the highest ever reported for piezo-active components integrated to Si substrates. This work is supported by the NSF under contract No. DMR-02-10512 and NSF-MRSEC under contract No. DMR-00-80008.

### 4:45 PM O7.6

#### Local Electromechanical Properties of Ferroelectric Materials for Piezoelectric Applications. A. L. Kholkin, V. V. Shvartsman and Igor K. Bdikin; Dept. of Ceramics and Glass Engineering and CICECO, University of Aveiro, Aveiro, Portugal.

Ferroelectric materials are being intensively investigated due to their high dielectric, ferroelectric and piezoelectric properties useful for

various applications. In particular, piezoelectric sensors and actuators utilize strong piezoelectric effect, which is determined by the domain arrangement and its evolution under applied electric field. These domain arrangements have to be investigated with the maximum resolution because of the polycrystalline nature and small grain size of the most frequently used materials. Imaging of domains with nanoscale resolution is becoming also an important issue for microelectromechanical systems (MEMS), which size is approaching to submicron dimensions. Scanning Probe Microscopy has recently proved its usefulness for high-resolution piezoelectric studies. In this presentation, the local piezoelectric properties of Pb-based ferroelectric materials (mainly of the PZT family) will be analyzed and compared with their respective macroscopic behavior studied using conventional techniques (laser interferometry). Several important issues will be addressed including grain size effect, local polarization switching and hysteresis, local non-linearity, self-polarization, and degradation problems such as aging and fatigue. Local variation of piezoelectric properties of ferroelectric films will be linked to their specific texture, composition, and morphological characteristics of individual grains. Based on these observations, the difference between the local and averaged macroscopic properties will be delineated. The effect of local polarization switching by pure mechanical force exerted by the SPM tip will be demonstrated and explained. This effect may be detrimental for the functionality of ferroelectric films in MEMS where mechanical stress is essential. In the second part of the talk, the local piezoelectric properties of ferroelectric relaxor single crystals and ceramics of the  $\text{PbMg}_{1/3}\text{Nb}_{2/3}\text{O}_3$ ,  $\text{PbZn}_{1/3}\text{Nb}_{2/3}\text{O}_3$  and  $\text{Pb}_{1-y}\text{La}_y(\text{Zr}_{1-x}\text{Ti}_x)_{1-y/4}\text{O}_3$  families will be discussed. In these materials, the extraordinary piezoelectric properties are observed for the compositions close to the morphotropic phase boundary. It will be shown that on the nanoscale the properties of relaxors and relaxor-based materials are different from the macroscopic observations and nanoscale domains related to polarization clusters appear on the surface and may play essential role in their high electromechanical performance.

SESSION 08: Nanomechanical Probes  
 Chair: Bryan D. Huey  
 Wednesday Morning, December 1, 2004  
 Commonwealth (Sheraton)

#### 8:30 AM \*O8.1

**Atomic Force Microscopy at Ultrasonic Frequencies, State-of-the-Art and Prospect.** Ute Rabe, Applied Research, Fraunhofer Institute for Nondestructive Testing, IZFP, Saarbruecken, Saarland, Germany.

The combination of ultrasound with atomic force microscopy (AFM) opens the high lateral resolution of scanning probe techniques to ultrasonics. Atomic force acoustic microscopy (AFAM) and ultrasonic friction force microscopy are techniques which use the vibration modes of AFM cantilevers. The resonance frequencies of commercial cantilevers of a few 100 micrometers length are predominantly in the ultrasonic frequency range between 20 kHz and several MHz. In the AFAM-mode the cantilever is vibrating in one of its flexural resonance frequencies while the sensor tip is continuously in contact with the sample surface. With this technique images can be obtained the contrast of which depend on the elasticity of the sample surface. The radius of the tip-sample contact area ranges between several nm's and several 10 nm's. Polycrystalline materials which appear mechanically isotropic on a macroscopic scale are therefore anisotropic on the length scale which is probed by an AFM and the elastic constant which is measured by the tip is the indentation modulus. When ferroelectric ceramics are imaged, the acoustic image reveals a substructure within the grains indicating variations in contact stiffness. In ferroelectrics an important contribution to the contact stiffness results from the electrical polarization of the individual domains. Shear stiffness and friction phenomena can be investigated in ultrasonic friction force microscopy by evaluating the torsional resonances of AFM cantilevers. At low lateral surface vibration amplitudes the sensor tip remains in elastic contact with the sample surface, and the cantilever behaves like a linear oscillator with viscous damping and a certain set of resonance frequencies. If the surface vibration is increased above the critical amplitude, the maximum of the resonance curves does not increase any more and the curves develop a plateau at their highest amplitude. Numerical simulations of a nonlinear oscillator driven by a dry friction element produced curve shapes as in the experiment. This led us to the conclusion that the plateaus in the resonance curves indicate the onset of stick-slip in the relative tip-sample oscillation.

#### 9:00 AM O8.2

**Quantitative Nanomechanical Mapping with Contact-Resonance-Frequency AFM.** D. C. Hurley, A. B. Kos and P. Rice; National Institute of Standards and Technology, Boulder, Colorado.

As critical dimensions shrink below a micrometer, new tools are required to investigate material properties on commensurate scales. In response, we are developing dynamic atomic force microscopy (AFM) techniques to quantitatively image the nanoscale mechanical properties of surfaces, thin films, and nanostructures. We have previously demonstrated how quantitative elastic-property measurements at a single sample position can be obtained with atomic force acoustic microscopy (AFAM). AFAM makes use of multiple resonant frequencies of an AFM cantilever in contact mode to calculate the tip-sample contact stiffness. Local mechanical properties can be determined from the contact stiffness by invoking the appropriate contact-mechanics models. However, fixed-position techniques are typically too slow for practical application to two-dimensional mapping. In this talk, we will describe new methods for quantitative mechanical-property imaging. A key element of our approach is a frequency-tracking circuit to locate the contact-resonance frequencies at each image position. The circuit is based on a digital signal processing architecture that enables rapid data acquisition (typically < 30 minutes for a 256 x 256 image). With a range of operation from approximately 1 to 3000 kHz, the circuit can accommodate a wide variety of different cantilever vibration modes. We will present imaging results obtained with both flexural and torsional modes on several nanostructured materials. By combining information from flexural and torsional images, it may be possible to simultaneously determine both Young's modulus and Poisson's ratio for an isotropic material. Issues related to quantitative image interpretation will also be discussed, such as calibration methods, cantilever selection, tip wear, and choice of contact-mechanics model. Each of these elements plays a significant role in attaining the goal of truly quantitative nanomechanical imaging.

#### 9:15 AM O8.3

**Ultrasonic Force Microscopy Study of Multiwalled Carbon Nanotubes.** Robert E. Geer and Yuegui Zheng; College of Nanoscale Science and Engineering, University at Albany - SUNY, Albany, New York.

Differential ultrasonic force microscopy (d-UFM) has been used to investigate the nanoscale mechanical response of multi-walled carbon nanotubes (MWNT) synthesized via arc-discharge (A-D) and chemical vapor deposition (CVD) techniques. Quantitative analysis of the MWNT d-UFM data utilized Si and sapphire substrates and a high-resolution laser vibrometer system for sample-to-sample calibration. High-resolution transmission electron microscopy (TEM) was used to estimate MWNT wall thickness. Initial investigation of the A-D and CVD MWNTs through d-UFM reveal a surprisingly large radial indentation modulus compared to Si and sapphire. Nanoscale axial nonuniformities in the indentation modulus are also observed and correspond in size and conformation to variations in the MWNT wall thickness observed via TEM. UFM imaging was also carried out on MWNTs following prolonged exposure to a Ga-ion beam. Exposed areas of the nanotube exhibited modified mechanical properties resulting, presumably, from increased defects induced by the ion beam.

#### 9:30 AM O8.4

**Effect of Thickness on Elastic-Property Measurements of Thin Films using Atomic Force Acoustic Microscopy.** Malgorzata Kopycinska-Mueller, Roy H. Geiss, Jens Mueller, Dudley Finch and Donna C. Hurley; Materials Reliability Division, NIST, Boulder, Colorado.

Mechanical-property information is increasingly demanded in order to model and predict a thin film's behavior under different operating conditions. Because thin-film elastic properties often vary from those of the corresponding bulk material, it is critical to measure the properties of the film itself. However, as film thickness decreases, elastic-property measurements become progressively more challenging due to the complexity of the film/substrate system. Here we investigate how the thickness of submicrometer films affects the measured values of elastic properties obtained with atomic force acoustic microscopy (AFAM). AFAM is a dynamic mode of atomic force microscopy (AFM) that involves vibrating the AFM cantilever while the tip remains in contact with a sample. By measuring the resonant frequencies of the AFM cantilever at a fixed sample position, the value of the indentation modulus  $M$  at that point can be calculated. We determined average values of  $M$  for three nickel thin films approximately 50, 200 and 800 nm thick using AFAM. In sharp contrast to other methods such as nanoindentation, the AFAM measurements remained free of any influence from the silicon substrate – even for the 50 nm film. This behavior is understood by realizing that the stress field created in an AFAM experiment extends less than 100 nm into the sample and decreases rapidly due to the small applied static loads (0.4-2.5  $\mu\text{N}$ ) and small radius of contact (< 10-30 nm). The measured values of  $M$  ranged from 210 GPa to 223 GPa, significantly lower than expected from literature values for bulk nickel. X-ray diffraction spectroscopy and scanning electron

microscopy revealed that the thin films were nanocrystalline with an average grain diameter ranging from 16 to 27 nm. Thus grain-boundary or other microstructural effects are likely to be responsible for the observed reduction in  $M$ .

#### 9:45 AM O8.5

##### **Van der Waals and electrostatic force imaging with higher harmonic noncontact scanning force microscopy.**

Scott Russell Crittenden<sup>1</sup>, Arvind Raman<sup>3</sup> and Ron Reifenberger<sup>2</sup>;

<sup>1</sup>Sensors and Electron Devices Directorate, U.S. Army Research Laboratory, Adelphi, Maryland; <sup>2</sup>Physics, Purdue University, West Lafayette, Indiana; <sup>3</sup>Mechanical Engineering, Purdue University, West Lafayette, Indiana.

Conventionally, the cantilevers used in scanning force microscopy (SFM) are treated as simple harmonic oscillators driven sinusoidally at their resonance frequency. Interesting nonlinear behavior lies beyond this approximation when the cantilever is treated as a continuous beam oscillating in a non-quadratic potential well. Any real SFM cantilever can be expected to oscillate at higher harmonics of the driving frequency as well as at a set of frequencies corresponding to higher order bending modes. Recently, attempts to understand these nonlinear effects have provided a better understanding of what information SFM measurements can give about the tip-sample interaction. There are now a number of papers treating both linearized approximations and the full nonlinear system. Higher harmonic signals depend on the details of the force between the cantilever tip and the sample and therefore on such physical properties as the Hamaker constant, Young's Modulus, electrostatic charge, and the work function difference between the tip and sample. The literature on higher harmonic spectroscopy and nonlinear effects has focused on the tapping mode or on the contact mode where the sample is vibrated acoustically. In both modes the tip operates in the repulsive regime of tip-sample interaction potential. Because the repulsive interactions depend on local material elasticity, these works have attempted to measure the local elastic properties of the sample using nonlinear effects with the customary nanoscale resolution of SFM. An as yet unexplored regime lies in the noncontact mode origin of nonlinear cantilever oscillations. Here, the tip-sample forces are dominated by local electrostatic and van der Waals forces; therefore, higher harmonic signals are expected to depend on these parameters. Because the tip never touches the sample surface, any contact mechanics issues, such as tip and sample deformation, etc., do not arise, leading to a simpler system of study. This offers the possibility of characterizing the local van der Waals forces and electrostatic charge which are far superior and more specific indicators of local chemical composition than local elasticity. We will present noncontact higher harmonic amplitude and phase images of, and dynamic force curves (DFC) over, thin biological bilipid membranes deposited on mica. We also show noncontact images and DFCs at a harmonic near the second bending mode of the cantilever and compare these results to those for harmonics far from any bending mode. Finally, higher harmonic imaging under careful control of experimental parameters allows for the minimization of electrostatic effects, leaving van der Waals force differences as the most plausible explanation for the image contrast observed in higher harmonic noncontact images. This work was supported by an Army Research Office MURI grant under contract DAAD19-99-1-1098.

#### 10:30 AM \*O8.6

##### **Probing Nanoscale Tip-Surface Interactions using Dynamic Spectroscopies.** Kathryn J. Wahl, U.S. Naval Research Laboratory, Washington, District of Columbia.

In order to investigate adhesion, mechanical and tribological processes at the nanometer scale, we need to better understand how model asperity contacts respond to surface forces and dissipate energy. Perhaps the most significant complication in the measurement and interpretation of nanoscale tip-sample interactions is the fact that the contact sizes are below the optical limit (1 micron). Macroscopic measurements often rely on optical observations of the contact, and many of the relevant contact mechanics models are formulated around direct measurement of the contact area or radius as a function of experimentally controlled parameters such as load or displacement. Consequently, for studies at the nanoscale to go beyond simple measurements of force and displacement requires approaches that enable indirect measurement of the contact area. Small, oscillatory force or displacement perturbations of the tip or sample can be used to determine the interaction or contact stiffness, and thus provide a measure of the contact size. In this talk, I will review various approaches to making quantitative, dynamic measurements using scanned probe microscopies. I will give examples of how dynamic force and displacement methods can be used to develop quantitative adhesion spectroscopies, mechanics imaging modes, and hybrid contact mechanics experiments.

#### 11:00 AM O8.7

##### **Near-Field Acoustic Holography: Novel High Resolution Sub Surface Imaging for Embedded Features.** Gajendra Shekhawat and Vinayak P. Dravid; Material Science and Engineering, Northwestern University, Evanston, Illinois.

As materials, structures and phenomena continue to shrink, and the micro/nanofabrication paradigms move from planar to 3-D/stacked platforms, there is an acute need to image and analyze surface/sub-surface features and phenomena at ultra-high resolution and sensitivity, coupled with usual ergonomic/economic considerations. Keeping view of these technological challenges and to overcome those, we have developed a turn-key Near-Field Acoustic Holography (NFAH) system. This unique system will address emerging issues in imaging and analysis of diverse embedded nano and microscale structures, and engineered systems. In NFAH, one high frequency acoustic wave is launched from below the specimen, and another one on the scanning cantilever of the scanning probe microscopy (SPM) system, albeit at a slightly different frequency. The resultant beat frequency which forms as a pseudo-standing wave on the specimen surface, acts as a reference lattice. Any perturbation to phase and amplitude of the specimen acoustic wave is then measured with SPM tip as an antenna, and converted in quantitative pictorial map. Thus, internal features, e.g., voids, cracks, phases, which perturb the acoustic wave, can be seen in such images. This technique will fill a critical void in characterization and investigation of the static and dynamic mechanics of nanoscale systems, ranging from engineering systems to biologically active structures, in-vitro. In the presentation, we will report efficacy of NFAH approach, starting with a model subsurface microstructure of buried Au nanoparticles/prisms underneath a polymer film. These embedded features are readily imaged with NFAH providing proof-of-concept of this novel approach. Additional results on practical systems will be presented, such as high resolution sub-surface imaging of copper vias (without doing any cross-sectioning), composite structures and internal features of carbon nanotubes, among others. It will be argued that ramping the acoustic frequency to 100 MHz would enable the extraction of subsurface defects (voids, delamination) with spatial resolution < 5 nm.

#### 11:15 AM O8.8

##### **Photoinduced reversible hardening of the Young's modulus in ZnO nanobelt.** Minhua Zhao<sup>1</sup>, Chuanbin Jiang<sup>2</sup>, Shouxin Li<sup>2</sup>, Lei Lu<sup>2</sup> and Scott X. Mao<sup>1</sup>; <sup>1</sup>Department of Mechanical Engineering, University of Pittsburgh, Pittsburgh, Pennsylvania; <sup>2</sup>Shenyang National Laboratory for Materials Science, Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China.

We report photoinduced reversible hardening of the Young's modulus in ZnO nanobelt. The effective Young's modulus of ZnO nanobelts under over band gap illumination is at least twice as large as that under darkness or below band gap illumination, which is derived by two methods. One is from the unloading part of the load-displacement curve in the nanoindentation depth of 10nm to 80nm using cube corner diamond indenter. The other is from loading part of AFM force-penetration curve below the penetration depth of 5nm using cantilever typed diamond probe. For comparison, similar work is done on (0001) bulk ZnO. We find little difference in the effective Young's modulus of ZnO bulk with and without illumination. The physical mechanism for the observed phenomena is discussed in terms of the electronic strain induced by the photogeneration of free carriers in ZnO. This finding sheds light on realizing nanoscale optical tunable surface acoustic wave (SAW) devices.

#### 11:30 AM O8.9

##### **Fabrication and Mechanical Properties of Suspended One Dimensional Nanostructures: Carbon Nanotubes, Polypyrrole Nanotubes, and Helical Polyacetylene Nanofibers.**

Sang-Wook Lee<sup>1,2</sup>, DongSu Lee<sup>1</sup>, Bio Kim<sup>1</sup>, Eleanor E. B. Campbell<sup>2</sup> and Yung Woo Park<sup>1</sup>; <sup>1</sup>School of Physics, Seoul National University, Seoul, South Korea; <sup>2</sup>School of Physics and Engineering Physics, Gothenburg University and Chalmers University of Technology, Gothenburg, Sweden.

Suspended structures of one dimensional nano materials were fabricated using an acid free method. Poly (methyl methacrylate) that has the same height as Au anchors was fabricated in order to prevent the materials from falling down. The mechanical properties of suspended carbon nanotubes (CNTs), polypyrrole (PPy) nanotubes, and helical polyacetylene (PA) nanofibers were studied by the force-distance measurement with atomic force microscope tip manipulation. Elastic modulus of suspended CNT, PPy nanotube, and helical PA nanofiber were estimated at 0.3 TPa, 3 GPa, and 0.6 GPa respectively.

#### 11:45 AM O8.10

##### **Variation in Atomic-Scale Friction with Substrate Surface Order and Partial Fluorination for Alkanephosphonic Acid**

**Self-Assembled Monolayers on Alumina.** Matthew J. Brukman<sup>1</sup>, Robert W. Carpick<sup>1</sup> and Timothy D. Dunbar<sup>2</sup>; <sup>1</sup>Engineering Physics, University of Wisconsin - Madison, Madison, Wisconsin; <sup>2</sup>Corporate Research Materials Lab, 3M Company, St. Paul, Minnesota.

Self-assembled monolayers (SAMs) are of considerable interest for applications from the macroscopic to the nanometer scale, such as lubrication, corrosion protection, and water repellency. While much attention has been paid to SAMs on gold and silicon, less work has been performed for SAMs that form dense layers on other surfaces such as metal oxides. Here, we compare the wettability and nano-scale friction and adhesion of two phosphonic acid self-assembled monolayers (SAMs):  $\text{CF}_3(\text{CF}_2)_7(\text{CH}_2)_{11}\text{-PO}_3\text{H}_2$  (F8H11 PA) and  $\text{CH}_3(\text{CH}_2)_{17}\text{-PO}_3\text{H}_2$  (H18 PA) deposited on a series of three  $\text{Al}_2\text{O}_3$  surfaces: C- and R- plane single crystal sapphire, and an amorphous alumina layer. The molecules form densely packed hydrophobic layers with strong substrate adhesion. Atomic force microscopy was then used to characterize the nano-scale dependences of adhesion and friction on SAM composition and substrate surface order. We observe consistently greater friction in the F8H11 versus the H18 samples for each substrate. The R-plane sapphire substrate also leads to reduced friction. In addition, partial fluorination generally reduces the wettability of the monolayer by both water and hexadecane. This indicates that substrate-imposed variations in packing density, ordering, and wettability have measurable nanotribological effects.

SESSION O9: Electrical SPM and SPM-Based  
Transport Measurements  
Chair: Wilfried Vander Vorst  
Wednesday Afternoon, December 1, 2004  
Commonwealth (Sheraton)

#### 1:30 PM \*O9.1

##### **Semiconductor Dopant Profile and Dielectric Characterization with Scanning Capacitance Microscopy.**

Joseph J. Kopanski, Semiconductor Electronics Division, National Institute of Standards and Technology, Gaithersburg, Maryland.

Scanning capacitance microscopy (SCM) has been commonly used to image dopant gradients in silicon and other semiconductors. As a mobile, high-resolution (to 10 nm) metal-oxide-semiconductor (MOS) probe, SCM also is a non-destructive, contactless tool with which to examine local variations in dielectric thin film quality and local variations in semiconductor substrate properties. Virtually any MOS qualification measurement that can be made with fabricated metal electrodes can also be made with SCM. Two particular applications being pursued are characterization of high-k dielectric films on silicon for next generation integrated circuits and characterization of native and deposited insulators on wide bandgap semiconductors. Local differential capacitance (dC/dV) versus tip bias (Vtip) measurements can be made with SCM using an ac voltage to generate the differential capacitance signal. These measurements differ from conventional C-V measurements due to the 3-D nature of the SCM tip and the method used to generate the differential capacitance signal. Theoretical predictions and experimental measurements are made of SCM differential capacitance versus dc bias voltage (dC/dV-V) curves for metal-oxide-semiconductor (MOS) capacitors with various levels of fixed and interface traps. The goal of this work is to determine if quantitative interface trap distributions can be measured using SCM and if variations in interface trap density can be observed near defects or device structures. The response of the SCM MOS capacitance measurement to a local electric field stress and optical pumping from the AFM laser will also be discussed.

#### 2:00 PM O9.2

##### **In situ Resistance Measurement of Epitaxial Silicide Nanowires.**

H. Okino<sup>2</sup>, Y. Hosono<sup>2</sup>, Shuji Hasegawa<sup>2</sup> and Peter Bennett<sup>1</sup>; <sup>1</sup>Physics Department, Arizona State University, Tempe, Arizona; <sup>2</sup>Physics Department, University of Tokyo-Hongo, Tokyo, Japan.

We have measured the resistance of self-assembled epitaxial silicide nanowires (NWs) using a multi-tip UHV-STM instrument. NWs were grown by deposition of 1 monolayer cobalt onto a Si(110) substrate at 750 C, producing NWS with a single orientation and average dimensions: 50nm wide by 4 microns long. Resistance is measured in situ by placing two independent STM tips at variable separations along a single NW using an integral SEM to guide the placement. "Leakage" conduction through the substrate is less than 1/(106 ohms), while the NW resistance increases linearly with tip separation, with a nominal value of 500 ohms. Measured resistivity for the NWs is approximately 100 micro-ohm-cm, similar to the value for extended epitaxial films of CoSi2 at 300K. The NW resistance appears to be dominated by interface scattering, even though the NW boundaries are atomically perfect.

#### 2:15 PM O9.3

##### **A Microscopic View of Charge Injection, Transport, and Trapping in Organic Electronic Materials Via High Sensitivity Electric Force Microscopy.** John Aaron Marohn<sup>1</sup>, William R. Silveira<sup>1</sup> and Erik Muller<sup>2</sup>; <sup>1</sup>Chemistry, Cornell University, Ithaca, New York; <sup>2</sup>Physics, Cornell University, Ithaca, New York.

To date, models of charge injection, trapping, and transport have mainly been tested by measuring and modeling the current-voltage characteristics of devices as a function of voltage and temperature. Since organic conductors are often unavoidably disordered, one worries that bulk characterization techniques are poorly suited for testing microscopic theories. We have used electric force microscopy to image the local electrochemical potential and capacitance in films of the hopping conductor triphenyldiamine-doped polystyrene (TPD-PS) on gold, a molecularly doped polymer system used in xerography and a model system for charge injection in LEDs, and in pentacene, a candidate material for organic field effect transistors. In order to get a clearer picture of charge injection in the TPD-PS/Au model system, we have imaged electrochemical potential drops in a working device. We observe a gradual change in the shape of the potential drop and capacitance as the source-drain voltage is increased. This data qualitatively verifies a microscopic picture of hole conduction in which charge transport transitions from ohmic to space charge limited transport. Internal electric field and charge density at the metal-organic interface are inferred from the measured electrochemical potential and the current. We find that the charge density at the TPD-PS/Au interface increases with electric field much faster than is predicted by the commonly assumed diffusion-limited thermionic emission theories. In 20-100 nm thick TPD-PS films created under a wide variety of conditions (varied substrates, host polymer, degree of dipole doping, film thickness, TPD concentration, and temperature) we observe a 60-80 mV-pp variation in electrochemical potential exhibiting a 100-200 nm correlation length. These observations cannot easily be explained using the accepted understanding of charge injection and transport in hopping transport materials. Even in this model system, it seems that charge injection may be a far more complicated process than has been assumed. We have also used electric force microscopy to show that charge traps are spatially inhomogeneous in polycrystalline pentacene devices. These experiments follow changes in local capacitance and contact potential as traps fill in a working field effect transistor, and represent a first step towards measuring single-site trap energies and correlating these energies with structure.

#### 2:30 PM O9.4

##### **Electric Force Microscopy Study on Photo-Induced Charging of Pentacene Monolayer in Dry Nitrogen.** Liwei Chen<sup>1</sup>, Oksana Cherniavskaya<sup>1</sup>, Alex Shalek<sup>1</sup>, Alejandro Schrott<sup>2</sup> and Louis E. Brus<sup>1</sup>; <sup>1</sup>Chemistry, Columbia University, New York, New York; <sup>2</sup>IBM Watson Research Center, Yorktown Heights, New York.

With the recent progress in organic electronic devices such as OLED and OTFT, there is an ever increasing demand in the understanding of organic electronic materials and their interfaces with metal and dielectrics. We recently reported an interfacial dipole between pentacene monolayer and silicon dioxide and pentacene-related in-gap states at the interface in UHV. Here we investigate the pentacene/SiO2 interface in dry nitrogen environment and under photo-excitation at 532nm. Pentacene monolayer is thermally deposited in UHV of 5x10-9 torr onto silicon wafers with a 2nm thick thermal oxide and then put in dry nitrogen box with short exposure to the air. EFM measurements found that first the electric field gradient on pentacene is dependent on the doping of the Si wafer under the 2nm SiO2 dielectrics. Second, it is also dependent on the sample history in ambient and in dry nitrogen. Third, when the pentacene is illuminated with a 532nm (2.3eV) CW laser from a glazing angle, the field gradient increases monotonically, indicating the accumulation of positive charges regardless of the substrate doping and sample history. Time trajectories of individual islands charging and then decay after the laser is turned off were collected and fit to single exponentials. Under strong laser illumination, the monolayer sample undergoes a dramatic morphology change. 5-8 nm size particles appeared in circular regions on the SiO2 surface. The pentacene islands in regions with these small particles are smaller in size compared to samples in dark or weak illumination, and less charged than under weak illumination. We propose a Coulombic explosion mechanism for this phenomenon.

#### 3:15 PM \*O9.5

##### **Exploring Carbon Nanotubes with Scanned Probe Microscopy.** Paul McEuen, Jun Zhu, Markus Brink, Ji-Yong Park, Yuval Yaish, Ethan Minot, Vera Sazonova and Sami Rosenblatt; Laboratory of Atomic and Solid-State Physics, Cornell University, Ithaca, New York.

Carbon nanotubes are an exciting new class of materials with

remarkable electronic and electromechanical properties. These nanometer diameter cylinders offer an unprecedented opportunity to explore solid state physics in a one dimensional system. Electrons in the nanotube occupy one-dimensional subbands that result from the quantization of the electron motion around the circumference of the tube. The tubes can be metals or semiconductors, depending on the detailed structure of the tube. While much is known about nanotubes, many important questions remain about the effects of strain, disorder, and electric and magnetic fields on their properties. This talk will review experiments by our group that use scanned probe measurements to locally probe the electronic and electromechanical properties of nanotubes. In one set of experiments, an AFM is used to look inside carbon nanotube devices. A metallized AFM tip is used to make local, moveable, electrical contact to a nanotube. Using this technique in conjunction with electrical measurements, we determine the resistance of nanotube/metal contacts and also probe intrinsic scattering lengths in both metallic and semiconducting nanotubes. The measurements demonstrate that transport mean free paths can be very long, limited only by electron-phonon scattering. We also use the AFM to explore suspended carbon nanotube devices. The tip can be used to apply tension to the tube and explore how mechanical strain affects the bandgap. These measurements also show that the suspended section of the tube is nearly free of the disorder and threshold voltage shifts characteristic of tubes on oxide surfaces. Finally we use a low-temperature AFM to explore single-electron charging phenomena in nanotube quantum dots. The addition of individual electrons to the dot produces a detectable force on the cantilever, as well as shifting its resonant frequency and producing additional dissipation. All of these can be used to image single electron states of nanotubes, even in tubes without external electrical contacts.

### 3:45 PM O9.6

**Local Probing of Electron Transport Mechanisms in Carbon Nanotubes Using a Scanning Tunneling Microscope in the Transmission Electron Microscope.** Krister Svensson<sup>1</sup>, Hakan Olin<sup>2</sup> and Eva M. Olsson<sup>1</sup>; <sup>1</sup>Experimental Physics, Chalmers University of Technology/Gothenburg University, Gothenburg, Sweden; <sup>2</sup>Engineering, Physics and Mathematics, Mid Sweden University, Sundsvall, Sweden.

Carbon nanotubes are predicted to be ballistic conductors where the electrons travel freely along the carbon layers of the tubes. The conductance is therefore expected to be twice the quantum conductance  $G_0$  ( $G_0 = 2e^2/h \approx 1/13$  (k $\Omega$ )<sup>-1</sup>) for an ideal carbon nanotube with good electrical contacts. The total resistance of a nanotube would then be about 6.5 k $\Omega$  for a nanotube including the contacts and independent on the length of the tube. Several investigations of the conduction mechanisms of carbon nanotubes have been performed. In most cases a diffusive conduction have been reported [1, 2]. Quantum conductance have only been observed at room temperature for suspended multiwalled carbon nanotubes (MWNTs) after repeated dipping into liquid metal [3]. The observed conductivity was of the order of  $G_0$ . The present work concerns a comparison of the conduction mechanisms of MWNTs filled with iron and without. The local properties of the carbon nanotubes are investigated using a scanning tunneling microscope (STM) in the transmission electron microscope (TEM) [4, 5]. The combination of the two microscopies allows simultaneous imaging and measurements of properties thus allowing a direct correlation between atomic structure and properties. Measurements on the iron filled MWNTs showed that the conductance increased with applied bias with a factor of two between low and high bias. The tube diameter and wall thickness also affected the conductivity with a linear dependence on the cross sectional area. The results indicated a diffusive mechanism for the electron transport. References: [1] T.W. Ebbesen, H.J. Lezec, H. Hiura, et al., *Nature* **382**, 54 (1996) [2] A. Bachtold, M.S. Fuhrer, S. Plyasunov, et al., *Phys. Rev. Lett.* **84**, 6082 (2000) [3] P. Poncharal, C. Berger, Y. Yi, et al., *J. Phys. Chem. B* **106**, 12104 (2002) [4] K. Svensson, Y. Jompol, H. Olin and E. Olsson, *Rev. Sci. Instr.* **74**, 4945 (2003) [5] Commercially available from Nanofactory Instruments (<http://www.nanofactory.com/>)

### 4:00 PM O9.7

**Measuring Spatial Variation of Transconductance by Scanning Gate Microscopy.** Julia W. P. Hsu<sup>1</sup>, N. G. Weimann<sup>2</sup>, Michael Manfra<sup>2</sup>, F. F. Schrey<sup>3</sup>, K. W. West<sup>2</sup>, D. V. Lang<sup>2</sup>, O. Mitrofanov<sup>2</sup> and R. J. Molnar<sup>4</sup>; <sup>1</sup>Sandia National Laboratories, Albuquerque, New Mexico; <sup>2</sup>Lucent Technologies, Murray Hill, New Jersey; <sup>3</sup>Technische Universität Wien, Wien, Austria; <sup>4</sup>Lincoln Laboratories, Lexington, Massachusetts.

Scanning gate microscopy (SGM) uses the conducting probe of an atomic force microscope as a local gate to modulate the conductance of the 2DEG between source and drain. To map transconductance variation, a dc bias,  $V_{DS}$ , is applied between drain and source while both a dc and an ac bias,  $V_{GS} V_{ac} \cos(\omega t)$ , are applied between the

tip (gate) and source. The ac component of the current between drain and source,  $I_{DS}$ , is measured using a lock-in amplifier at frequency  $\omega$ . Transconductance is defined as  $\Delta I_{DS}(\omega)/V_{ac}$ . We applied SGM to map the spatial variation of transconductance in AlGaIn/GaN heterostructures, which were grown by molecular beam epitaxy on semi-insulating GaN template prepared by hydride vapor phase epitaxy. The gateless transistor structures used in the SGM experiments are mesas, made next to and with ohmic contacts made in the same processes as conventional HEMTs. The potential drop between the drain and source was mapped using scanning Kelvin force microscopy and found to be linear. A spatial resolution of 250 nm was obtained in the transconductance images. The combination of SGM and scanning Kelvin force microscopy enables us to investigate the role of defects in transistor performance. In particular, when biased near the depletion of the 2DEG, the transconductance map displays a cell structure, with low signal regions correlating with the positions of negatively charged threading dislocations. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. The Lincoln Laboratory portion of this work was sponsored by the ONR under Air Force contract #F19628-00-C-0002. Opinions, interpretations, conclusions and recommendations are those of the authors and not necessarily endorsed by the United States Air Force.

### 4:15 PM O9.8

**Scanning Conductance Microscopy and High Frequency Scanning Gate Microscopy of Carbon Nanotubes and Polyethylene based Nanofibers.** Cristian Staii<sup>1,2</sup>, Rui Shao<sup>3</sup>, Nicholas J. Pinto<sup>4</sup>, Dawn A. Bonnell<sup>3</sup> and Alan T. Johnson<sup>1,2</sup>; <sup>1</sup>Physics and Astronomy, University of Pennsylvania, Philadelphia, Pennsylvania; <sup>2</sup>Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia, Pennsylvania; <sup>3</sup>Material Science and Engineering, University of Pennsylvania, Philadelphia, Pennsylvania; <sup>4</sup>Physics and Electronics, University of Puerto Rico, Humacao, Puerto Rico.

Scanning Conductance Microscopy (SCM), Scanning Gate Microscopy (SGM), and Scanning Impedance Microscopy (SIM) are very powerful approaches for measuring the local electrical properties of nanoscale structures. SCM can be used to determine whether nanotubes, nanowires, and even DNA are insulating or conducting. SGM has been used to image Schottky barriers that develop within carbon nanotube circuits and to quantify the local Fermi energy at gate-susceptible defects along the nanotube length. Here we present two new advances for these microscopies. First, we develop a model for the electrostatic (capacitive) interaction between tip and sample/substrate that makes SCM a more powerful quantitative technique. We show that experimental data from samples of (conducting) carbon nanotubes and (insulating) polyethyleneoxide (PEO) nanofibers are in excellent agreement with the model predictions. As a very important application we use this approach to measure the dielectric constant of PEO nanofibers. We also present results from a new scanning probe technique that extends the frequency range of SGM. This technique is used to image changes in the impedance of a CNFET circuit in the frequency range 40Hz-10MHz induced by a voltage-biased AFM tip that acts as a local gate. We show that our data are consistent with a parallel RC model for the CNFET circuit. In the low frequency range ( $f=40\text{Hz}-100\text{KHz}$ ), the resistance of the SWNT dominates the total impedance of the circuit, while the capacitance of the electrical contacts dominates in the intermediate-high frequency range ( $f=100\text{KHz}-10\text{MHz}$ ).

### 4:30 PM O9.9

**Two-dimensional Carrier Profiling on Operating Si-MOSFET by Scanning Capacitance Force Microscopy.** Kenjiro Kimura<sup>1</sup>, Kei Kobayashi<sup>2</sup>, Hirofumi Yamada<sup>1,4</sup>, Koji Usuda<sup>3</sup> and Kazumi Matsushige<sup>1,2</sup>; <sup>1</sup>Electronic Science Engineering, Kyoto Univ., Kyoto-shi, Japan; <sup>2</sup>International Innovation Center, Kyoto Univ., Kyoto-shi, Japan; <sup>3</sup>Advanced LST Technology Laboratory, Toshiba Corporation, Kawasaki-shi, Japan; <sup>4</sup>Core Research for Evolutional Science and Technology, Japan Science and Technology, Kyoto-shi, Japan.

Investigations of two-dimensional (2D) carrier profiles on nanometer scale are essential for the developments of novel devices to make its fabrication process more optimized. We have recently developed a novel scanning probe technique, scanning capacitance force microscopy (SCFM), which is capable of measuring 2D dopant profile on a semiconducting sample. Its principle is based on the detection of a local electric force proportional to the differential capacitance, which is obtained by measuring the third harmonic components ( $3\omega$ ) in the mechanical oscillation of an AFM cantilever induced by ac electric field between the AFM tip and the sample. The magnitude and the polarity of its component correspond to the carrier concentration and species such as p-type or n-type [1, 2]. Although, SCFM has been a powerful tool for the visualization of local dopant



profiles on cross-sectional Si-MOSFETs in the state of //OFF//, it has not been applied yet to analyze dynamical changes of the carrier profiles near the channel area in the state of //ON//. In this study, we prepared a special Si-MOSFET sample (p-type) for the cross-sectional analysis, and measure the 2D carrier profiles in the //ON// state by both SCFM and SCM. We first cut Si-MOSFET device to have its cross-section, polished the surface and then etched it few nm to remove contaminant and possible mobile charge. Next, we put Tungsten wires on the top side by utilizing Ion beam assist deposition in order to connect the external bias controller to each electrode, source, drain and gate. In the SCFM measurement, we applied an ac bias voltage (36 kHz) between the tip and the sample, and detected the third harmonic component (108 kHz) of the cantilever mechanical oscillation induced by the electrostatic force in vacuum. We found that SCFM contrast in the vicinity of the channel area was changed depending on the gate bias voltage. The contrast change appeared to reflect some changes of the carrier profile related to the inversion layer on the depleted region under the gate. Another possibility is that it is affected by mobile or fixed charge inside the native oxide layer on the sample surface. For the discussion of carrier density more quantitative from the SCFM or SCM data, several issues such as accidental charge injection to the sample from the tip, leak current between the electrodes through the surface, and the optimization of the measurement condition have to be taken into consideration. [1] K. Kobayashi, H. Yamada, and K. Matsushige Appl. Phys. Lett. 81, 2629 (2002). [2] K. Kimura, K. Kobayashi, H. Yamada, and K. Matsushige Appl. Surf. Sci. 210, 93 (2003).

#### 4:45 PM O9.10

##### Measurement of Variations in Electron Transport across Organic/Metal and Metal/Semiconductor Junctions on the Molecular Length Scale using a Scanning Probe.

Regina Ragan<sup>1</sup>, Seun Kim<sup>2</sup>, Douglass A.A. Ohlberg<sup>1</sup> and R. Stanley Williams<sup>1</sup>; <sup>1</sup>Quantum Science Research, Hewlett Packard, Palo Alto, California; <sup>2</sup>Department of Chemistry, Korea Advanced Institute of Science and Technology, Daejeon, South Korea.

As electronic devices size scales down toward the nanometer length scale, the effect of material interfaces and atomic ordering on electron transport must be well characterized. We use scanning tunneling microscopy (STM) to image the molecular arrangement of octanethiol and benzenethiol self-assembled monolayers on Pt(111) and of Pt/rare earth (RE) disilicide nanowire/Si(001) substrates in ultra high vacuum. Using scanning tunneling spectroscopy (STS), we measure variations in electron transport for these material systems with molecular resolution using the scanning probe as the top electrode. On Pt(111), we compare the packing of octanethiol to that of benzenethiol and compare both of these to the well studied system of alkanethiols on Au(111). Both octanethiol and benzenethiol form ordered monolayers on Pt(111) yet have different packing arrangements. Using constant current tunneling spectroscopy combined with STS we image the variations in electron transport as a function of position. Across grain boundaries, a higher current is measured than across ordered domains. In the case of RE disilicide nanowires on Si(001), we measure transport across the Pt-Ir tip/Si interface and the Pt-Ir tip/RE disilicide nanowire/Si junction for both n-type and p-type Si substrates. On highly doped,  $10^{18}/\text{cm}^3$ , n-type Si(001), the IV spectra is consistent with electron transport through a small Schottky barrier. We measure current rectification at the RE disilicide nanowire/p-type Si junction that is consistent with electron transport measurements across Er disilicide thin film/p-type Si junctions. On moderately doped,  $10^{17}/\text{cm}^3$ , p-type Si substrates, a change in the STS spectrum is observed as the measurement is taken with the tip closer to the surface and may arise from a tip induced depletion layer at the surface. Hexagonal RE disilicide nanowires self-assemble during epitaxial growth as one-dimensional metallic nanostructures on Si(001) substrates due to an anisotropic lattice mismatch with Si[110]. Previously we have demonstrated that dense arrays of parallel RE disilicide (RE = Er, Dy, Sm, Gd) nanowires exhibiting high aspect ratios, having lengths exceeding 1 micron and widths less than 5 nm can be grown on vicinal Si(001) substrates with a miscut of 2.5 degrees toward the [110] azimuth. The RE disilicide metallic nanostructures will be used as building blocks for chemical self-assembly of organic molecules to build nanostructures from the bottom up. Molecule/Pt/Er disilicide/p-type Si nanometer scale junctions could have current switching and rectifying behavior and in large arrays have applications as highly dense logic devices utilizing a defect tolerant cross bar architecture. Since decades of research have been focused on silicon based CMOS technology, the combination of organic materials with inorganic nanostructures on the silicon platform holds promise to yield innovative devices while allowing for integration with silicon.

SESSION O10: Poster Session  
Chair: Ute Rabe  
Wednesday Evening, December 1, 2004  
8:00 PM  
Exhibition Hall D (Hynes)

#### O10.1

##### STM Image Simulation: Effect of the Number of Tunneling States and the Isosurface Value.

Juan Radilla<sup>1</sup>, Yolanda Trinidad-Reyes<sup>2</sup>, Nikola Batina<sup>2</sup> and Marcelo Galvan<sup>1</sup>; <sup>1</sup>Quimica. Area de Fiscoquimica Teorica, Universidad Autonoma Metropolitana Iztapalapa, Mexico, D. F., Mexico; <sup>2</sup>Quimica. Area Electroquimica. Laboratorio de Nanotecnologia e Ingenieria Molecular, Universidad Autonoma Metropolitana Iztapalapa, Mexico, D. F., Mexico.

In this work Scanning Tunneling Microscopy (STM) images were simulated for the organic molecule 3,4,9,10 Perylenetetracarboxylic dianhydride (PTCDA) to study the effect of both the number of tunneling states and the isosurface value. Local Density Approximation to the Density Functional Theory (DFT-LDA) calculations were performed to achieve the simulated images under the Tersoff and Hamann approximation. The number of tunneling states has a strong effect in the patterns. The image appearance converges for a certain number of tunneling states. Intermediate contour values for the integrated Local Density Of States (LDOS) produce good resolution and matching in relation to the experimental image. This work was supported in part by Instituto Mexicano del Petroleo (IMP) through the project FIES-98-100-I No. 2115-31786 "Mecanismos de Deposicion de Compuestos Organicos Pesados en Flujo de Petroleo en Ductos", and the Consejo Nacional de Ciencia y Tecnologia (CONACYT) through a Juan Radilla scholarship.

#### O10.2

##### Atomically Resolved STM Images of CVD Grown Carbon Nanotubes.

Daniel (Ching-Shih) Chiang<sup>1</sup>, Philip (Zifeng) Lei<sup>1</sup>, Lifeng Dong<sup>2</sup> and Jun Jiao<sup>2</sup>; <sup>1</sup>Materials and Mechanical Engineering, Washington State University, Vancouver, Washington; <sup>2</sup>Physics, Portland State University, Portland, Oregon.

We have obtained atomically resolved STM images of CVD-grown single-wall carbon nanotubes (CNT) under the air ambient and room temperature. The crystal structure of carbon nanotube surface has been studied intensively since its chirality determines the electronic properties of nanotubes. However most studies focus on the CNTs fabricated by laser ablation or electric arc discharge. And most of the high resolution images were captured by STM at either ultra high vacuum or low temperature. In this research, we studied the chirality and diameter of the single-wall CNTs synthesized by CVD since CVD growth methods are highly promising for scale-up of defect-free nanotube materials, and enable well-aligned CNT growth with better control that is not possible with arc-discharge or laser ablation techniques. The preliminary study shows that the diameters of CNTs are either in the range of 1.2nm or 0.8 nm. The chiral angle is about 0 degree for all nanotubes. We will also discuss the two key factors for reaching capability of atomic resolution at the air ambient and room temperature: the absolute vibration free environment and the sharp probe tips.

#### O10.3

##### Surface Potential Reversibility on Self-Assembled Monolayer : Fermi Memory.

Saito Nagahiro<sup>1,2</sup>, Lee Sun-hyung<sup>2</sup> and Takai Osamu<sup>3,2</sup>; <sup>1</sup>Department of Molecular Design and Engineering, Nagoya University, Nagoya, Japan; <sup>2</sup>Department of Materials, Physics and Energy Engineering, Nagoya University, Nagoya, Japan; <sup>3</sup>EcoTopia Science Institute, Nagoya University, Nagoya, Japan.

Scanning probe microscopy (SPM) has been widely used in order to observe microstructures on self-assembled monolayers (SAMs). Moreover, scanning probe lithography (SPL) have been focused in the field of microstructural fabrication on self-assembled monolayers. The nanochemical conversion is based on electrochemistry in a minute water column formed between the sample and the tip of a SPM, which work as working and counter electrodes, respectively. In order to achieve a reversible chemical nanopatterning by SPM, we need to control both oxidation and reduction reactions. Simultaneously, the chemical conversion of functional group leads to shift of surface potential in nano-scale regions. Surface potential, that is, Fermi level was acquired by Kelvin probe force microscopy (KPFM). Here, we demonstrate that nanochemical conversion of amino-terminated groups in a SAM can be fabricated based on electrochemical reactions and the fabrication can provide us a memory, i.e. Fermi memory. Consequently, we are successfully to create the writing and erasing memory state based on surface potential changes. Memorized dots were fabricated by reducing nitroso groups, i.e. converting to amino groups and were erased by oxidizing amino groups. We have confirmed that these writing and erasing processes can be repeated at least several times.

#### **O10.4**

##### **Low Temperature Ultra High Vacuum STM for local imaging and spectroscopies on Single Magnetic Molecule.**

Messina Giovanni Paolo<sup>1,2</sup>, Dante Gatteschi<sup>1</sup>, Stefano Prato<sup>2</sup>, Daniela Orani<sup>2</sup> and Paolo Sigalotti<sup>2</sup>, <sup>1</sup>INSTM, Sesto fiorentino, Italy; <sup>2</sup>Area Science Park, APEResearch, Trieste, Italy.

We are building a low temperature STM microscope designed to work with magnetic molecular adsorbates. The STM microscope relies on a pendulum mechanism which ensure high vertical stability. The STM Head allows both x-y coarse translation and high stability. The z coarse approach is based on a home made concept. The requirement to do imaging on individual magnetic molecules or on self assembled monolayers calls for the use of high gain and low noise I/V preamplifier. UHV conditions are ensured through a cryopumping conception of the entire machine. The microscope features magnetic fields up to 5T perpendicular to the surface plane. Scanning Tunneling Spectroscopy (STS) is performed both digitally and through an external lockin. The system is particularly suited for fast loading of chemically (ex-situ) prepared surfaces

#### **O10.5**

##### **Electrical AFM Measurements for Evaluation of Nitride Erosion in Shallow Trench Isolation Chemical Mechanical Planarization.** Yordan Stefanov, Tino Ruland, Rama Komaragiri and Udo Schwalke; Institute for Semiconductor Technology, Darmstadt Technical University, Darmstadt, Germany.

This article proposes a new application of tunnelling current Atomic Force Microscopy (AFM) for evaluation of silicon nitride stop-layer erosion in Shallow Trench Isolation (STI) Chemical Mechanical Planarization (CMP). Excessive nitride erosion after the polishing step results in exposed and damaged active area surfaces, thus deteriorating device characteristics and drastically decreasing yield. Standard measurement techniques such as ellipsometry, profilometry, Scanning Electron Microscopy (SEM) and Scanning Tunneling Microscopy (STM) do not provide a simple, non-destructive and efficient means for detecting full nitride erosion on device level. Even pure topographical AFM is not applicable because of the gradual post-CMP transitions between nitride, silicon and silicon dioxide. However, with the help of simultaneous topographical and electrical AFM, positive identification of 'open' silicon surfaces on the nanometer scale can be achieved by detecting enhanced sub-picoampere tunnelling currents in those areas and directly correlating them to surface topography. The experimental results from test wafers prepared at our CMOS processing facility demonstrate clearly the capabilities of this approach. Its advantages over the above-mentioned standard measurement techniques are discussed. The new measurement technique comprises a useful method for automated process control and the comprehensive experimental database obtained by the experiments provides a valuable input for advanced 3D-CMP model verification.

#### **O10.6**

##### **Imaging Charge Traps in Pentacene Devices by High Sensitivity Electric Force Microscopy.** Erik Muller<sup>1</sup> and John Marohn<sup>2</sup>, <sup>1</sup>Physics Department, Cornell University, Ithaca, New York; <sup>2</sup>Department of Chemistry and Chemical Biology, Cornell University, Ithaca, New York.

We are investigating the electronic properties at the semiconductor-dielectric interface in pentacene thin film devices. It is believed that the conduction takes place within the first few monolayers of the organic and is adversely affected by the presence of charge traps. To date, information about charge traps has usually been inferred from analysis of bulk current-voltage measurements - few microscopic studies have been reported. By using a custom variable temperature electric force microscope in high vacuum we can probe the local charge and voltage distribution helping to understand the physics occurring at the interface. Experimental evidence of a spatially inhomogeneous charge traps distribution will be shown. In addition, experiments to determine the role of charge traps in limiting the performance of pentacene devices will also be discussed.

#### **O10.7**

##### **Transport Properties in Carbon Nanotubes Probed by SPM.** Lucile C. Teague<sup>1</sup>, Sarbajit Banerjee<sup>2</sup>, Stanislaus S. Wong<sup>2</sup>, Janice Reutt-Robey<sup>1</sup>, James D. Batteas<sup>3</sup>, David Adebimpe<sup>4</sup> and Marek Zgol<sup>4</sup>, <sup>1</sup>Department of Chemistry and Biochemistry, University of Maryland-College Park, College Park, Maryland; <sup>2</sup>Department of Chemistry, State University of New York at Stony Brook, Stony Brook, New York; <sup>3</sup>Surface and Microanalysis Science Division, NIST, Gaithersburg, Maryland; <sup>4</sup>GMA Industries, Inc., Annapolis, Maryland.

The structural and electronic properties of several types of single-wall

carbon nanotubes have been studied using scanned probe methods. Samples include pristine HiPco tubes, ozonized tubes, and tubes functionalized with CdTe quantum dots, and are observed as deposited on HOPG and Au(111) surfaces. Scanning tunneling spectroscopy (STS) experiments and conductive probe atomic force microscopy allow transport I-V measurements on and along individual tubes and bundles of each type. Here, in this study, we will present the results of these experiments, comparing and contrasting the effects of oxygenation and attached CdTe quantum dots on the transport properties of these single-wall nanotubes.

#### **O10.8**

##### **Microscopic view of charge in semiconducting organic materials.** William Richard Silveira, Tse Nga Ng and John A. Marohn; Chemistry and Chemical Biology, Cornell University, Ithaca, New York.

We use high-sensitivity electric force microscopy to investigate charge transport and homogeneity in thin-films of conducting organic materials. We will present a critical microscopic investigation of the charge injection process in a disordered organic semiconductor, a triarylamine (TPD) dispersed in polystyrene (PS). This study reveals a local observation of microscopic space-charge limited conduction (SCLC), an effect of great importance in many fields, in TPD-PS. We will also discuss the observation of a local variation in the potential of uniform TPD-PS films and the likely causes for this surprising effect.

#### **O10.9**

##### **Simultaneous Measurement of Conductive Property and AFM Force Curves.** Takashi Morii<sup>1</sup>, Rika Mizuno<sup>1</sup>, Kazuhiro Yoneda<sup>1</sup>, Hirotaka Haruta<sup>1</sup>, Takao Okada<sup>1</sup> and Kenshi Hayashi<sup>2</sup>, <sup>1</sup>Research Institute of Biomolecule Metrology, Tsukuba, Ibaraki, Japan; <sup>2</sup>Med. Ins. of Bioreg., Kyushu Univ., Fukuoka, 812-8582, Japan.

Extending our knowledge on an electrical property of DNA molecules is fundamental to developing its application, such as a nano wire in future molecular electronics, even though it was clarified that DNA itself is not a conductor [1]. We are also concerned with the electrical properties of DNA nanostructures and we succeeded in synthesis of the DNA nanostructures, such as I-shaped, Y-shaped, and hexagon [2]. Generally speaking, the measurements of the electrical properties of soft materials are influenced by geometrical arrangement, mechanical deformation, and contact chemistry. We modified an Atomic Force Microscope (AFM) to measure current-voltage characteristics of the sample by means of point contact method at the same time as force curve measurements. In this work, at the beginning, we applied it for a study of the conductive property of a DNA thin film. The DNA molecules adsorbed densely on a gold substrate and formed the film. DNA behaved as an insulator also in our experiment. Two types of breakdown of insulation were observed successfully. One is caused by an electric field and another is done by a mechanical deformation. The result shows that the control of the loading force is essential to the measurement of the electrical property of soft materials. The extension of this method to the study of the electrical property of a single molecule will be discussed. The work was supported by New Energy and Industrial Technology Development Organization (NEDO). [1] C.Gómez-Navarro, F. Moreno-Herero, P. J. de Pablo, J. Colchero, J. Gómez-Herrero and A. Baró, PNAS, **99** 8484-8487 (2002) [2] R. Mizuno, H. Haruta, T. Morii, T. Okada, K. Nakashi, T. Asakawa and K. Hayashi, Transactions of the Materials Research Society of Japan, **29**[2] 439-441 (2004)

#### **O10.10**

##### **Scanning Potentiometric Imaging of Hydride Blister and Inclusion in Irradiated and Non Irradiated Depleted Uranium Samples.** Marilyn E. Hawley, Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, New Mexico.

Scanning potentiometric imaging is sensitive to local variations in electrical properties that can be directly correlated with structure. Uranium, like other metals, is susceptible to the corrosive effects of hydrogen gas, which can readily diffuse into the material because of its small size. In the case of uranium, hydrogen attack results in the formation of blisters that eventually cover the surface. Initially these blisters appear as local circular features, often including an inclusion in the center. The uranium hydride formed as a result of this process has significantly different electrical properties than the base metal that is dramatically revealed in the potentiometric images. In this study, samples were irradiated with positively charged fluoride ions through a grid in order to determine if the irradiation-induced damage and stress would act as initiation sites for the blisters. However, the distribution of blisters was unaffected by irradiation. In contrast the local electrical properties were affected by the irradiation and the blisters appear in the potentiometric images in sharp contrast to the uranium background.

## **O10.11**

### **Charging and Field Emission Properties of Multiwalled Carbon Nanotubes Probed by Electric Force Microscopy.**

Mariusz Zdrojek<sup>1,2</sup>, Thierry Melin<sup>1</sup>, Benoit Jouault<sup>3</sup>, Michal

Wozniak<sup>4</sup>, Wojtek Gebicki<sup>2</sup>, Christophe Boyaval<sup>1</sup>, Andrzej Huczko<sup>5</sup>, Leszek Adamowicz<sup>2</sup> and Didier Stievenard<sup>1</sup>; <sup>1</sup>IEMN-CNRS UMR 8520, Villeneuve d Ascq, France; <sup>2</sup>Faculty of Physics, Warsaw University of Technology, Warsaw, Poland; <sup>3</sup>GES - CNRS UMR 5650, Montpellier, France; <sup>4</sup>Faculty of Materials Science and Engineering, Warsaw University of Technology, Warsaw, Poland; <sup>5</sup>Faculty of Chemistry, University of Warsaw, Warsaw, Poland.

Electrostatic properties of single multiwalled carbon nanotubes (MWCNTs) deposited on a dielectric (SiO<sub>2</sub>) layer have been investigated by charge injection and Electric Force Microscopy (EFM) experiments. We use EFM as a tool to locally inject charges into single MWCNTs in a similar way as the recent work performed on silicon nanocrystals[1], and to detect the amount stored charges, with a sensitivity of 10 electrons, and a lateral resolution 50 nm[2]. We found that upon local injection from the biased EFM tip, charges delocalize over the whole nanotube length (i.e. 1-10 microns), which is consistent with a capacitive charging of the entire MWCNT-substrate capacitance. A drastic change is however observed as for the discharging properties of the MWCNTs. For bigger nanotubes (i.e. with diameter greater than 25-30 nm), stored charges are left floating on the oxide layer, and exhibit retention times greater than ten hours under nitrogen atmosphere. However, for smaller nanotubes, abrupt discharges can occur within a few seconds, in which stored charges are emitted to the EFM tip. Abrupt discharges occur either when scanning the nanotube apex, or at additional "defect" points along the nanotubes. In addition, it is shown that the SiO<sub>2</sub> layer supporting the nanotubes acts as a charge-sensitive plate for electrons emitted from the MWCNTs at low electric fields. This allows to measure to perform by EFM a spatial mapping of MWCNT field emission patterns[3][4]. EFM experiments on single MWCNTs can thus be directly correlated with the MWCNT structural (apex diameter or length) properties. [1] See e.g. T. Melin et al., Appl. Phys. Lett. 81 5054 (2002). [2] T. Melin et al. Phys. Rev. B 69, 035321 (2004); Phys. Rev. Lett. 92, 166101 (2004). [3] Y. Saito, K. Hamaguchi et al., Nature, 389 6651 (1997). [4] See e.g. A. Buldum and J. P. Lu, Phys. Rev. Lett., 91 236801 (2003).

## **O10.12**

### **Electrical Characterization using Scanning Capacitance Microscopy of the Local Electronic properties of Semiconductor Nanostructures.**

Bremond E. Georges<sup>1</sup>, Decossas

Sebastien<sup>1</sup>, Marchand Jean-Jacques<sup>1</sup>, Stoica Tomas<sup>2</sup>, Vescan Lili<sup>2</sup>, Bassani Franck<sup>3</sup>, Karmous Halim<sup>3</sup> and Berbezier Isabelle<sup>3</sup>; <sup>1</sup>Laboratoire de Physique de la Matière, INSA Lyon, Villeurbanne, France; <sup>2</sup>FZ-ISG, Jülich, Germany; <sup>3</sup>CRMCN, Marseille, France.

The development of the nanotechnology field including the use of single semi-conductor nanostructure and nanocrystal needs novel experimental method of probing techniques allowing the measurement of the local electronic properties of devices integrating such objects. In this context the use of conductive tips and specific electrical mode measurements from an atomic force microscopy (AFM) is currently developed in order to probe single nanostructure or nanodevices by acceding to the electrostatic force, the surface potential, the capacitance and the tunneling current through a barrier. This study aims to analyze electrical spectroscopic measurements obtained by scanning capacitance microscopy via atomic force microscopy on Ge nanostructures grown on localized focalized ion beam (FIB) area on Si. A Si/Ge/Si stack has been grown on nanometer scale localized area fabricated by FIB. Molecular beam epitaxy (MBE) and selective chemical vapour epitaxy (CVD) have been used. A low temperature oxidation process has been performed to oxidize the Si around the Ge nanostructure in order to electrically isolate the Ge dot. The samples have been characterized by using electric modes of a D3100 AFM of Digital Instrument as the Scanning Capacitance Microscopy (SCM), the Kelvin Force Microscopy (KFM) and the Tunneling Atomic Force Microscopy (TUNA). SCM allows one to have the local signature of derivative C-V curves (i.e. dC/dV), KFM, the local surface barrier potential and TUNA, the local I-V curves. Nanolithography mode by AFM has also been used to charge the Ge dot. Depending of the measurements conditions, we obtain high contrast in capacitance between area with nanostructure and area without nanostructure. The local dC/dV spectroscopy ( in this case the tip is stopped at a specific location on the sample and the dC/dV signal is recorded while the tip bias is swept) shows a shift of the flat band voltage, that we attribute to the presence of electric charge in the nanostructures. High density sample surfaces can be charged within areas as small as 30nm in diameter. Charged areas are very well defined. Effect of the charging condition ( bias and duration) have been measured on the dC/dV curve and can be discussed in term of retention charge in the Ge nanocrystals or in the oxide. All the results reported points out the possibility to study the electronically

properties of individual semi-conductor nanostructures by electrical mode AFM techniques in order to understand and control them in order to integrate them in nano-devices.

## **O10.13**

### **Mapping of Local Electronic Properties and Spatially Resolved Magneto-resistance of Nanostructured CMR Thin Films by Scanning Tunneling Microscope.**

Sohini Kar<sup>1</sup>, Barnali

Ghosh<sup>1,2</sup>, Loveleen K. Brar<sup>1</sup>, Mandar A. Paranjape<sup>3</sup> and A. K. Raychaudhuri<sup>1,2</sup>; <sup>1</sup>Department of Physics, Indian Institute of Science, Bangalore, Karnataka, India; <sup>2</sup>S. N. Bose National Centre for Basic Sciences, Kolkata, West Bengal, India; <sup>3</sup>Department of Physics, Boston College, Boston, Massachusetts.

We have investigated the local electronic properties and the spatially resolved magneto-resistance of a nanostructured film of a colossal magneto-resistance (CMR) material by Scanning Tunneling Spectroscopy (STS), Scanning Tunneling Potentiometry (STP) and local conductance mapping (LCMAP) using a variable temperature Scanning Tunneling Microscope (STM) operating in a magnetic field. The nanostructured thin films (thickness  $\approx 500$ nm) of the CMR material La<sub>0.67</sub>Sr<sub>0.33</sub>MnO<sub>6</sub> (LSMO) on quartz substrates were prepared using chemical solution deposition (CSD) process. The CSD grown films were imaged by both STM and atomic force microscopy (AFM). Due to the presence of large number of natural grain boundaries (GB) of incoherent nature, the films show non-linear current-voltage (*I-V*) characteristics like CMR films with artificial grain boundary junctions, as well as low field magneto-resistance (LFMR). The measurement of spatially resolved electronic properties reveal the extent of variation of the density of states (DOS) at and close to the Fermi level (*E<sub>F</sub>*) across the grain boundaries and its role in the electrical resistance of the GB. Measurement of these properties as a function of magnetic field as well as temperature reveals that the LFMR occurs at the GB. The main contribution to the resistance as well as the LFMR at the GB occurs due to the variation of the DOS across the GB and its change in the magnetic field. While it was known that LFMR in CMR films originates from the GB, this is the first investigation that maps the local electronic properties at a GB in a magnetic field and establishes the occurrence of the LFMR at the GB and the role of the interface at the GB.

## **O10.14**

### **Elastic Properties of Nanoclusters with AFM Nano-**

**indentation.** Eli Lansey and Fredy R. Zypman; Physics, Yeshiva University, New York, New York.

Atomic Force Microscopy (AFM) can measure mechanical compliance in the nm range. When an AFM is used as a nanoindenter, the incursion of the tip into the sample is measured as a function of applied force. This setup can, in principle, provide the mechanical elastic constants of samples with nN/nm resolution, and concomitantly small surface energies. Here we contribute by developing a theory to extract elastic properties from nanoclusters deposited on silicon substrates. In the absence of the AFM tip, the cluster is assumed to take its minimum energy shape as obtained from Equivalent Crystal Theory, which has already proven appropriate to predict energies of gold clusters as tested by X-Ray absorption fine structure studies. The AFM tip is lowered until it starts interacting with the cluster. Then it is further lowered, thus indenting the cluster. We study the shape of the nanocluster as a function of tip position, by minimizing the total energy of the system. From this we also propose a universal method to solve the inverse problem in which the elastic constants are obtained from the force-separation curve. We are also currently investigating the effects of temperature as a limiting factor in the minimum elastic constant that can be theoretically measured. Work supported by Research Corporation.

## **O10.15 TRANSFERRED TO O8.7**

## **O10.16**

### **Influence of Tip Wear on Atomic Force Acoustic Microscopy Experiments.**

Malgorzata Kopycinska-Mueller, Roy H. Geiss, Paul Rice and Donna C. Hurley; Materials Reliability Division, NIST, Boulder, Colorado.

Techniques based on atomic force microscopy (AFM) can provide qualitative images of surface material properties with high lateral resolution. However, imaging of a sample surface always involves wear of the AFM tip. To better understand and characterize tip wear behavior, we have performed experiments using scanning electron microscopy (SEM) and atomic force acoustic microscopy (AFAM). AFAM utilizes the vibrations of the AFM cantilever while the tip is in contact with a sample to determine the sample's local elastic properties. However, the contact-resonance frequencies also depend on parameters such as the tip radius *R*. Therefore, knowledge of *R* and how it changes with tip wear is essential for accurate determination of elastic properties with AFAM. We performed AFAM experiments

using several different AFM cantilevers on a sample with known elastic properties. Using the Hertz model for the contact mechanics, the value of  $R$  for each measurement was calculated. High-magnification SEM images of the tip were obtained before and after each AFAM measurement. The values of  $R$  measured from the SEM images were compared to the corresponding AFAM values. The first AFAM measurements were done at relatively low static loads (0.4-1.2  $\mu\text{N}$ ). The static load was then successively increased up to several microneutons in an attempt to break and/or plastically deform the tip. Both techniques showed that the tip radius increased with use; however, the AFAM values of  $R$  were consistently smaller than those obtained from the SEM images. In addition to tip wear, we also observed changes in the shape of the tip. For example, side-view SEM images of the very end of the tip showed an increase in the tip width, suggesting that we deformed the tip plastically. The information gained from such experiments will help us to refine our understanding of AFAM contact mechanics beyond the Hertz approximation, in order to improve measurement accuracy and repeatability.

#### **O10.17**

##### **Damping of Atomic Force Microscopy Cantilevers.**

Alan Schilowitz<sup>1</sup>, Dalia Yablon<sup>1</sup> and Fredy Zypman<sup>2</sup>; <sup>1</sup>Corporate Strategic Research, ExxonMobil Research and Engineering Co., Annandale, New Jersey; <sup>2</sup>Department of Physics, Yeshiva University, New York, New York.

When the cantilever of an atomic force microscope (AFM) is immersed in fluid, its vibrational spectrum changes. More specifically, the position of the resonance and the quality factor change depending on fluid properties [1]. Both from a fundamental standpoint, to improve reconstruction algorithms on AFM, and to understand fluid-solid interaction at the nanoscale it is desirable to define rules which explain the impact of fluid properties on the cantilever's frequency spectrum. In this work we present a new theoretical reconstruction algorithm to explain cantilever behavior. The algorithm is based on the continuum model of the cantilever [2] improved by the addition of fluid drag and a new internal friction term. Good agreement is found between theory and experiment. 1. J.W.M. Chon, P. Mulvaney and J.E. Sader, *J. Appl. Phys.* 87(8), 2000,3978 2. L.D. Landau, E.M. Lifshitz, *Theory of Elasticity* (Theoretical Physics, Vol 7) Butterworth-Heinemann; 3rd edition (1986)

#### **O10.18**

**Nanotomography: SPM goes 3D.** Robert Magerle, Physikalisches Chemie II, Universitaet Bayreuth, Bayreuth, Germany.

Nanotomography [1] is a novel procedure for high-resolution volume imaging with scanning probe microscopy (SPM). The method is similar to an excavation on the nanometer scale. With suitable etching or polishing techniques the specimen is eroded step by step and the chemical composition of each freshly exposed surface is imaged with scanning probe microscopy. From the resulting series of images, separated in depth by only a few nanometers, the specimen's three-dimensional microstructure can be reconstructed. I will present current examples of volume imaging with 10 nm resolution the microstructure of block copolymers, semicrystalline polymers, and Ni-based superalloys. With the success of SPM in mind, volume imaging with SPM promises new insights into the physics of materials on the nanometer and even atomic scale. [1] R. Magerle, *Phys. Rev. Lett.* 85, 2749 (2000); EP 1144989; U.S. Patent 6,546,788.

#### **O10.19**

##### **In Situ Monitoring Film Formation of Polyolefin Dispersions Using Atomic Force Microscopy (AFM) Hot Stage.** Jing Li,

Wenbin Liang and Steve Chum; Analytical Sciences Lab, The Dow Chemical Company, Freeport, Texas.

Film formation from polymer latex has been the subject of greatly interest in recent years [1,2] Despite the new advances, formation of void-free film from discrete particles, especially semicrystalline polymer particles, and the mechanisms involved in deformation of small polymeric particles are still the subjects of active research. In particular, particle size of the polymer dispersions has been found to affect the film formation temperature by some investigators, while Sperry and others observed the contrary. This work studies the film formation processes of semicrystalline polyolefin dispersions using AFM technique and attempt to clarify the effect of particle size on film formation mechanism and associated mechanical properties of the resulting films. AFM topography images of ethylene vinyl acetate (EVA) and ethylene octene (EO) copolymers dispersions were obtained by scanning pre-dried samples on a glass slide surface at room temperature. The images show spherical particles with clear boundaries which mean the particles are not diffused with each other at room temperature. The size of the spherical particles of both samples varies widely ranging from 0.03 to 1.5  $\mu\text{m}$  for the EVA sample and 0.1 to 3  $\mu\text{m}$  for the EO dispersion. The wide range of particle size is ideal to investigate the effect of particle size on film formation

temperature. The polymer particle coalescence process was monitored for the first time, to our knowledge, using an atomic force microscopy combined with a miniature hot stage. The result shows the deformation profiles (the reduction of the measurable height of the particles due to softening of particles at elevated temperature) of two target particles as a function of temperature. It is found that both the larger and smaller particles have similar deformation temperature dependence. Smaller particles tend to deform faster than the larger ones which is attributed to its smaller mass and heat transfer. Larger particles may require longer time to completely coalesce at the same temperature. Furthermore, morphology changes associated with the film formation process and mechanical property development are also discussed. Fundamental understanding of the film formation mechanism helps materials selection in practical application of semicrystalline polyolefin dispersions. [1]. Urban, D.; Takamura, K. Ed. *Polymer Dispersions and Their Industrial Applications*. Wiley-VCH Verlag, 2002 [2]. Steward, P.A., Hearn, J., Wilkinson, M.C., *Adv. Colloid Interface Sci.*, 2000, 86, 195-267.

#### **O10.20**

##### **Electrical and Optical Properties of Polythiophene Films**

##### **Employing Current Sensing-AFM and Reflectance**

**Spectroscopy.** Hyo Joong Lee and Su-Moon Park; Chemistry, Center for Integrated Molecular Systems, Pohang University of Science and Technology, Pohang, South Korea.

Recently, we have demonstrated that current-sensing atomic force microscopy (CS-AFM) can be successfully applied to the characterization of conducting polymer films of various doping states. The technique is very useful in studying doping distributions by obtaining two-dimensional current images and nanoscale electrical characteristics by measuring current-voltage curves. We have also shown that the optical properties can be measured at various doping stages together with electrical properties using a reflective absorption mode setup termed the near normal incidence reflectance spectroscopy (NNIRS); both the microscopic electrical properties and their averaged optical properties are correlated well with each other. In this study, electrical and optical properties of poly(3-methylthiophene) (P3MeT) films have been studied using the current-sensing atomic force microscopy (CS-AFM) and the near normal incidence spectroscopic techniques. The P3MeT films were electrochemically deposited onto gold-on-silicon electrodes and their doping levels were controlled by a series of electrochemical reduction. At each doping stage, a two-dimensional current mapping, as well as a number of current-voltage characteristics at selected locations, was obtained with nanometer scale spatial resolution using the CS-AFM, and the corresponding absorption spectrum obtained by the NNIRS technique. As the dedoping was progressed, the magnitude of the current flowing through the P3MeT film was reduced gradually and I-V curves changed from metallic to semiconducting states. Rectifying behaviors were observed from the P3MeT film when its highly doped form was reduced to an appropriate level; the rectifying behaviour appears to result from the unique contacts formed between the polymer film and the tip at this doping stage. To compare the effects of electropolymerization conditions on the electronic states, thiophene and 3-hexylthiophene were also polymerized under the same conditions as those of 3-methylthiophene. The results were significantly different from those shown by P3MeT, indicating their doping behaviours were different from those shown by P3MeT. We have also obtained the electrical and optical properties of chemically synthesized regioregular poly(3-hexylthiophene) (P3HT) and electrodeposited P3HT films. The doping states of P3HT films were controlled by chemical or electrochemical oxidation, and their corresponding 2-dimensional current images, current-voltage characteristics, and absorption spectra were obtained. These films show significantly different properties from each other, which were attributed to different chemical structures and morphologies.

#### **O10.21**

##### **Scanning Tunneling Microscopy Fabrication and Electronic Characterization for Quantum Computing.** Marilyn E. Hawley,

Geoffrey W. Brown and Holger Grube; Materials Science & Technology Division, Los Alamos National Laboratory, Los Alamos, New Mexico.

Quantum computation is a revolutionary new paradigm with tremendous growth since 1994. The quest to build a quantum computer (QC) has been inspired by its recognized formidable computational potential. The long-term goal is to build a large scale, fast, parallel and easily fabricated QC. Although a number of ingenious schemes have been proposed, a silicon-based solid-state architecture, using nuclear or electron spins of dopants such as phosphorus as qubits, are attractive because of their long relaxation times, scalability, and integrability with existing silicon technology. We have been working on such a device based on a proposal by B. Kane (Nature 393, 133 (1998), in which buried P atoms placed 20 nm apart act as quantum bits (qubits) entangled through exchange

interactions. These qubits are atomically placed using scanning tunneling microscopic (STM) lithographic techniques using a monohydrogen layer as a resist. This effort requires dosing the Si(100) surface with phosphine molecules and annealing the phosphorus into the silicon surface. STM-based atomic level lithography provides us with the added capability of characterizing the local electronic environment of the dopants. In this talk, I will describe our effort to fabricate a QC and the charge imaging technique we are using to image buried phosphorus dopants and charged defects that could potentially interfere with the operation of such a QC device and any other nano scale surface device on the silicon (100) surface.

SESSION O11: Novel Concepts in SPM  
Chair: Vincent Meunier  
Thursday Morning, December 2, 2004  
Commonwealth (Sheraton)

#### 8:30 AM O11.1

**Novel Dynamic Scanning Microscope Probe and its Application to Local Electrical Measurement in an Ion Sensitive Field Effect Transistor.** Terunobu Akiyama, Kaspar Suter, Nicolaas F. de Rooij and Urs Staufer; Univ. of Neuchatel, Inst. of Microtechnology, Neuchatel, Switzerland.

We present a unique, self-actuating and self-sensing scanning probe, which is based on a quartz tuning fork and a microfabricated cantilever. The probing tip can be electrically connected to an external source or measure unit. The sensitivity of the drain-source current of an ion sensitive field effect transistor (ISFET) was investigated as function of the probe position in order to assess the potential of the probe and its advantages, e.g. non-optical detection. A plain implementation of the probe has been recently introduced [Akiyama, et al., Rev. Sci. Instrum. 74, 112 (2003)]: a U-shaped microfabricated cantilever, exhibiting a sharp tip is assembled to a commercial quartz tuning fork, as used for wrist watches. The two legs of the cantilever are attached in a symmetrical way to the two prongs of the tuning fork. When exciting the tuning fork in its first resonance mode, the ends of the two prongs are moving in-plane and out of phase, meaning that they approach and withdraw from each other. This motion forces the U-shaped cantilever to start oscillating at the same frequency but in an out-of-plane motion. In a more advanced implementation, two additional, long and soft beams are added for electrically contacting the tip [Suter, et al, AIP Conf. Proc. 696, 227 (2003)]. One end of each of these beams is connected to the U-shaped cantilever and the other end, which is shaped to form a bonding-pad, is fixed on the base of the tuning fork. The tuning fork is used as an oscillatory force sensor similar to a quartz microbalance. Its frequency and amplitude governs that of the tip vibration, while the cantilever determines the spring constant of the whole probe. During dynamic scanning probe microscopy, the resonance frequency of the tuning fork, which is below the eigenfrequency of the cantilever, is tracked by a phase locked loop and kept at a set point by adjusting the sample height-position with a feedback loop. The drain-source current in an ISFET is modulated by the specific adsorption of chemical species above the channel area of the FET. Our new probe with the two contact beams could be used to evaluate the sensitivity of an ISFET to local variations in adsorption. Since ISFETs are also light sensitive, the self-detecting property of the probe was instrumental for this experiment. We have applied a small AC-potential to the tip while scanning it above the channel area of an ISFET, which was driven at a constant current. By doing so, this current was perturbed. These small current variations were registered simultaneously with the topography image of the channel area. The most effective location turned out to be the interface to the source region. It is conceivable that such probes could also be used for interrogating signals in other integrated circuits, e.g. in a prober-station, where a highly accurate positioning of the prober-tips is required.

#### 8:45 AM O11.2

**Ballistic Electron Emission Luminescence Spectroscopy of an InAs Quantum Dot Heterostructure.** Wei Yi<sup>1</sup>, Ian Appelbaum<sup>1</sup>, Kasey J. Russell<sup>1</sup>, Venkatesh Narayanamurti<sup>1</sup>, Micah P. Hanson<sup>2</sup> and Arthur C. Gossard<sup>2</sup>; <sup>1</sup>Gordon McKay Laboratory of Applied Science, Harvard University, Cambridge, Massachusetts; <sup>2</sup>Materials Department, University of California, Santa Barbara, California.

We present ballistic electron emission luminescence (BEEL) spectroscopy measurements of three-terminal hot-electron heterostructure devices embedding optically active InAs quantum dots. As a three-terminal variation of scanning tunneling microscopy (STM), BEEL is a novel probe of carrier transport and spontaneous emission of direct-gap semiconductor heterostructures. To realize both hot-electron injection and efficient radiative recombination, a BEEL device combines a Schottky metal-semiconductor interface with a buried light-emitting p-i-n heterostructure. By applying an emitter voltage higher than the Schottky barrier height, ballistic electrons are

injected through the thin metal base layer into the n-type surface layer of the semiconductor collector. Provided that an external collector voltage bias is applied as required by energy conservation, ballistically injected electrons can recombine radiatively with holes in a buried optically-active layer and emit inter-band luminescence. The principle of BEEL has been previously demonstrated in a GaAs quantum well heterostructure. In the present work, the GaAs quantum well layer is replaced by a layer of InAs self-assembled quantum dots as the optically active region. Complimentary BEEL measurements are performed in the configurations of both solid-state tunnel-junction transistors and STM tip injection. Due to much higher injected current, the planar transistors allow us to detect the wavelength information of the emitted photons, which resolves both quantum dot luminescence peak near 1.34 eV and bulk GaAs luminescence peak at 1.48 eV. This picture is reinforced by the observation of a quantum-confined Stark-shift for the 1.34 eV peak. The wavelength spectroscopy from the solid-state transistor devices facilitates interpretation of collector voltage dependence of the BEEL spectra from STM devices. Furthermore, by freezing out the collector leakage current at low temperatures, consistent collector-current spectra are acquired with both STM and planar transistors. Our results pave the way to a simultaneous imaging of the surface topography, local electron transport, and luminescence of semiconductor heterostructures.

#### 9:00 AM \*O11.3

**Single-Molecule Inelastic Electron Tunneling Spectroscopy.** Hyojune Lee<sup>1</sup> and Wilson Ho<sup>1,2</sup>; <sup>1</sup>Department of Physics & Astronomy, University of California - Irvine, Irvine, California; <sup>2</sup>Department of Chemistry, University of California - Irvine, Irvine, California.

The spatial resolution and versatility of the scanning tunneling microscope (STM) allow us to study single atoms, molecules, and nanostructures in a well-characterized local environment free of uncertainties surrounding the nature of the system under study. The ability to probe individual atoms and molecules has made it possible to reveal properties which would otherwise be masked in the study of an ensemble of atoms and molecules. Combining the imaging, manipulation, and spectroscopic characterization capabilities of the STM, we have probed the electronic and vibrational properties of individual molecules and nanostructures. Spatially-resolved spectroscopy with the STM enables spatial mapping of excitations. The single-molecule studies provide insights into physics and chemistry at the spatial limit.

#### 9:30 AM O11.4

**Applying Nuclear Magnetic Resonance Force Microscopy to Spintronics and Quantum Computing Systems.** Sean R. Garner<sup>2</sup>, Seppe Kuehn<sup>3</sup> and John A. Marohn<sup>3</sup>; <sup>1</sup>Cornell University, Ithaca, New York; <sup>2</sup>Department of Physics, Cornell University, Ithaca, New York; <sup>3</sup>Department of Chemistry and Chemical Biology, Cornell University, Ithaca, New York.

Magnetic resonance force microscopy (MRFM) is a technique which uses cantilevers to detect magnetization in a sample from a resonance-localized volume of spins. If single-nucleus sensitivity can be achieved, possible applications would include atomic-scale imaging in three dimensions, or readout of a spin quantum computer. In pursuit of this goal, we previously introduced a force-gradient-based type of MRFM which we demonstrated at unprecedented sensitivity. Here we will report our recent progress in further improving the sensitivity of nuclear MRFM and our application of the technique to Ga nuclei in GaAs. We will also discuss ongoing efforts to apply MRFM to organic self-assembled monolayer systems, which are being investigated as a framework for spin quantum computing.

#### 9:45 AM O11.5

**Developing Low and Room Temperature Nanoprobes to Address Individual Single Magnetic Molecules.**

Messina Giovanni Paolo<sup>1,2</sup>, Lorenzo Lenci<sup>3</sup>, Stefano Prato<sup>2</sup>, Daniela Orani<sup>2</sup>, Paolo Sigalotti<sup>2</sup> and Dante Gatteschi<sup>1</sup>; <sup>1</sup>INSTM, Sesto Fiorentino, Italy; <sup>2</sup>APEResearch, Trieste, Italy; <sup>3</sup>IPCF-CNR, Pisa, Italy.

We are developing two Scanning Tunneling Microscopes specifically aimed at addressing Single Molecular Magnets. The First instrument is a Low Temperature UHV STM designed to perform STS spectroscopy on Magnetic molecules both self assembled and drop casted on metal surfaces. The major instrumental challenge is the ability to detect Low currents at Low Temperature. The second instrument is a Room Temperature High Vacuum STM able to detect Radio Frequency (RF) components of the Tunneling Current. The instrument is aimed at verify previous results in ESR-STM and extend them on to Magnetic Molecules. In comparisons with earlier experiments this Microscope has the capability to monitor magnetic field, to variate the insitu magnetic field and present a superior RF recovery circuit. First

Results will be presented.

#### 10:30 AM \*O11.6

**Multiple Modulation Scanning Probe Techniques for Defect Mediated Transport Properties.** Dawn Bonnell, Materials Science, University of Pennsylvania, Philadelphia, Pennsylvania.

A number of multiple modulation approaches to scanning probe microscopy (SPM) have been developed recently. Utilizing multiple modulations allows frequency and time domain to be explored and accessing higher order harmonics of response functions opens the window to a wider range of properties. In some cases the goal is to distinguish and separate simultaneous electrostatic and magnetic interactions. In others, new properties are accessed. New approaches include: Multiple Modulation Magnetic Force Microscopy 1 Scanning Impedance Microscopy 2 Nano Impedance Spectroscopy 3 Second Harmonic Piezo Force Spectroscopy 4 Techniques such as these have been applied in ambient, for the most part. This talk will describe these approaches, illustrating the characterization of transport at interfaces, in molecular wires and nanofibers, as well as the domain dynamics in ferroelectric films. Although analysis in UHV allows more systematic control over surface conditions, difficulties arise in implementing these tools in vacuum. We will present a new approach to determining local surface potential in UHV that is based on electrostatic force interactions. This will be demonstrated on an atomically abrupt oxide interface. The ability to generally implementing multiple modulation techniques IHV will be discussed. 1. Alvarez et al APL 2001 2. Kalinin et al APL 2001 3. Shao et al APL 2003 4. Shao et al JJAP 2004

#### 11:00 AM O11.7

**Improve the Accuracy of Scanning Kelvin Probe Microscopy by Elimination of the Cantilever Effect.** Zhitao Yang<sup>1</sup>, Goutam Koley<sup>2</sup> and Michael Spencer<sup>1</sup>, <sup>1</sup>School of Electrical and Computer Engineering, Cornell University, Ithaca, New York; <sup>2</sup>Department of Electrical Engineering, University of South Carolina, Columbia, South Carolina.

Scanning Kelvin probe microscopy (SKPM) is one of the most important tools of device characterization. It can be used as a nano-scale voltmeter to quantitatively measure surface potentials of electronic devices. However, the accuracy of scanning Kelvin probe microscopy is reduced by "cantilever effect", which is due to a large capacitance gradient associated with the cantilever. In conventional SKPM measurements, the finite dimensions of the cantilever result in forces on the tip being affected not only by potentials underneath the tip itself, but also by the potentials underneath the cantilever. Therefore in order for quantitative measurements to be made on structures containing closely spaced potentials, this cantilever effect needs to be eliminated. We have introduced two approaches to eliminate the cantilever effect. One approach is to create an aperture on the device to be scanned by SKPM. This aperture shields and grounds the device, therefore allows the tip to only see the field in the aperture. The aperture can be fabricated on a mobile structure so that it is versatile for different samples. The other approach to eliminate the cantilever effect is to shield the cantilever, which means the conventional SKPM probe need to be modified so that only the tip is exposed to the electric field of the sample, while the cantilever is electrically grounded. In this presentation both approaches are described and compared with fabrication procedures, SKPM scanning results and quantitative analysis.

#### 11:15 AM O11.8

**Non-linear Transport Imaging by Scanning Impedance Microscopy.** Junsoo Shin<sup>1,2</sup>, V. Meunier<sup>3</sup>, A. P. Baddorf<sup>2</sup> and S. V. Kalinin<sup>2</sup>, <sup>1</sup>Department of Physics and Astronomy, The University of Tennessee, Knoxville, Tennessee; <sup>2</sup>Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee; <sup>3</sup>Computer Science and Mathematics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Scanning Probe Microscopy (SPM) techniques such as Scanning Surface Potential Microscopy and Scanning Impedance Microscopy (SIM) have been demonstrated to be powerful tools for quantitative dc and ac transport imaging in semiconductor structures and at grain boundaries. SPM techniques have been exclusively used to access the linear static and frequency dependent lateral electronic transport in materials and devices at the nanoscale, while current-voltage transport properties of most electro-active interfaces are intrinsically non-linear, a highly desired property for device functionality. A novel non-linear SIM technique is proposed to extend the nanoscale transport measurements of intrinsic material properties to the non-linear regime, through detection of frequency harmonics. This technique can be readily transferred to most cantilever-based scanning probe microscopes. As a test for the technique, we used a prototypical metal-semiconductor interface prepared by cross-sectioning a commercial Au-Si Schottky diode and connected in series with two

current limiting resistors. A non-linear SIM signal is shown to originate from both the intrinsic non-linear behavior in the device and from frequency mixing in the tip-surface junction. An approach to differentiate the two is demonstrated. Under certain conditions, the n-th order non-linear SIM signal is shown to be directly related to the corresponding derivative of the I-V curve of the interface. We also demonstrate the use of the first and second resonance of the cantilever to provide resonance amplification for topography and electrostatic measurements simultaneously while avoiding cross-talk between the two. The imaging mechanism, surface-tip contrast transfer, optimal experimental conditions, and potential applications of non-linear SIM are discussed. Although non-linear properties are generally too complex in macroscopic techniques, non-linear SIM allows spatially resolved imaging of non-linear transport properties of individual microstructural elements and provides a new approach for quantitative nanoscale characterization of non-linear transport phenomena. Research was sponsored by the U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the National Science Foundation grant DMR-0072998. Research performed as a Eugene P. Wigner Fellow (SVK).

#### 11:30 AM O11.9

**Terahertz imaging with submicron resolution.** Roland Kersting<sup>1,2</sup>, Chen Hou-Tong<sup>2</sup>, Nicholas Karpowicz<sup>2</sup> and Cho Gyu Cheon<sup>3</sup>, <sup>1</sup>Department of Physics, University of Munich, Munich, Germany; <sup>2</sup>Department of Physics, Rensselaer Polytechnic Institute, Troy, New York; <sup>3</sup>IMRA America, Ann Arbor, Michigan.

The development of the terahertz (THz) frequency band has opened up numerous opportunities for material characterization in the nano-sciences. Terahertz techniques can solve one of the most important questions of nano-electronics, which addresses the decoherence of an electron in an individual quantum dot. At THz frequencies, decoherence processes can be directly observed in the time domain, because THz spectroscopy provides amplitude and phase information about the electronic dynamics. In this contribution, we report on apertureless scanning near-field optical microscopy (SNOM) in the far-infrared. We will present terahertz images of organic and inorganic objects that show spatial resolutions down to 150 nm. In our THz-SNOM the resolution is achieved by scanning the object with a tungsten probe that is held in proximity to the samples surface. The transmission of THz pulses through the microscope head depends on the dipole of the tip-surface system and thus on the dielectric constant beneath the tip. In our experiments, we use tungsten probes that have tip radii between 100 nm and 1 micron. The THz pulses are generated by femtosecond laser excitation of InAs and have a center frequency of about 2.0 THz. After transmission through the microscope head, the THz signal is time-resolved by electro-optic sampling [1]. We apply a novel concept for imaging the sample's permittivity on the nanoscale [2]. In our experiments we achieve an enhanced tip-surface coupling by designing a tip with a resonance at a frequency of about 0.5 THz, which is just below the THz spectrum we use. Changes of the dielectric constant of the surface shift the resonance frequency and thus the diffraction and dissipation of the system. In order to deduce the resolution of the THz-SNOM, we performed experiments on metallic grating structures. Terahertz images show, that lines of 10 micron width can be clearly resolved. Despite an image contrast of less than 1%, a THz image of 60\*60 points can be recorded in less than 20 minutes. Experiments of organic layers of cresol resin showed that the minimum volume, which can be detected is about 10-18 m3. The spatial resolution is found to depend mostly on the tip radius of the metallic probe and the height at which the probe scans over the samples surface. Experiments with tips of 100 nm radius revealed spatial resolutions down to 150 nm. Such resolutions would allow to measure the terahertz response of isolated nanostructures and to investigate the electron dynamics within an individual quantum dot. References: [1] H.-T. Chen, R. Kersting, and G.C. Cho, "Terahertz imaging with nanometer resolution," Appl. Phys. Lett. 83, 3009 (2003). [2] H.-T. Chen, S. Kraatz, R. Kersting, and G.C. Cho, "Identification of a resonant imaging process in near-field microscopy," submitted to Phys. Rev. Lett. (2004).

#### 11:45 AM O11.10

**A Near-Field Microwave Probe for Quantitative Local Characterization of Dielectric Thin Films.** Vladimir V. Talanov, Robert L. Moreland, Andre Scherz and Andrew R. Schwartz; Neocera, Inc., Beltsville, Maryland.

The semiconductor industry is in the midst of a transition from SiO2 to dielectrics with lower dielectric constant k, the so-called 'low-k' materials, to reduce the delay times on interconnect wires, and to minimize the crosstalk between such wires. Design of advanced interconnect structures requires measurement of the microwave dielectric constant of these low-k materials to characterize the effects of clock harmonics (5x to 10x clock frequency) and crosstalk. The desired metrology should be non-destructive, non-contaminating, and provide real time/rapid data collection and analysis. We have

developed a novel technique based on a scanning near-field microwave probe. The technique is non-contact, requires no sample preparation, and can be used for both porous and non-porous low-k dielectrics. The probe has a few-micron sampling spot-size, so the measurement could be performed over a test region in the scribe line on a device wafer. For dielectric films with  $k < 7$  and thickness down to 200 nm the probe provides precision and accuracy better than 1% and 5%, respectively. We will present quantitative dielectric constant measurements on a variety of SOD and CVD low-k films and show excellent correlation with mercury probe measurements. The design of our probe is based on a balanced microwave transmission line tapered down to a one-micron tip aperture. Unlike the apertureless STM- or AFM-based schemes that have been previously employed, our "apertured" approach allows for truly quantitative measurements on a few-micron length scale where the result is insensitive to the material property outside this probing volume. The balanced line geometry virtually eliminates stray fields and reduces the amount of power radiated from the tip by a few orders of magnitude compared to the unbalanced coaxial geometry. A modified shear-force approach is employed to actively control the tip-sample separation. When the probe is placed in close proximity to the thin film sample the fringe capacitance is governed by the film and the substrate permittivities, the film thickness, the tip geometry, and the tip-sample separation. A microwave resonator operating at 4 GHz is employed to measure this capacitance with resolution of 30 zF. Extraction of the film dielectric constant is based on an original theory describing the tip-sample interaction, which provides for direct removal of the substrate contribution. Bulk Si and a set of variable thickness thermal oxide films are employed to calibrate the probe. There is no need to know the absolute value of the tip-sample separation for either measurement or calibration procedures; this separation must only be kept nominally the same for both measurements by the virtually material independent shear-force distance control. Application of this technique for quantitative measurements of the sheet resistance of thin metallic barriers and ultra-shallow implants will be discussed as well.

SESSION O12: SPM of Low-Dimensional Systems  
 Chair: Alexei Gruverman  
 Thursday Afternoon, December 2, 2004  
 Commonwealth (Sheraton)

**1:30 PM \*O12.1**

**Theory of Scanning Probe Microscopy of Carbon Nanostructures.** Vincent Meunier, Computational Chemical Sciences Group, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Experimental techniques related to SPM imaging and spectroscopy of low-dimensional systems have significantly progressed in recent years. At the same time, new simulation methods and computational techniques have allowed the development of a theoretical basis to the interpretation and understanding of the measurements. In this contribution, we concisely review these state-of-the-art modeling methods for scanning probe microscopy. Specific applications of the formalism will be illustrated for the case of carbon nanotubes, for which numerous high-resolution images and spectra are available. Research sponsored by the Mathematical, Information and Computational Sciences Division, Office of Advanced Scientific Computing Research of the U.S. Department of Energy. Oak Ridge National Laboratory is managed by UT-Battelle, LLC, for the U.S. Department of Energy under Contract DE-AC05-00OR22725.

**2:00 PM O12.2**

**Fluorescence from Individual PbS Quantum Dots.** Jeffrey Peterson and Todd D. Krauss; Dept of Chemistry, University of Rochester, Rochester, New York.

Due to their extremely large electron, hole, and exciton Bohr radii, lead salt (PbS, PbSe and PbTe) quantum dots (QDs) can achieve levels of quantum confinement that are not accessible to more commonly studied III-V and II-VI QDs. Thus, the strong confinement regime can be attained for relatively large particles, which may mitigate deleterious surface effects and imparts to the particles novel optical and electronic properties. For example, compared to colloidal II-VI QD core particles, lead salt QDs have significantly larger fluorescence quantum yields and fluorescence lifetimes two orders of magnitude longer. In addition to being fundamentally important materials, lead salt QDs are optically active across the entire near-infrared (NIR) region, making these potentially useful materials for both telecommunication and biological-labeling applications. We will present investigations of single PbS QD fluorescence using far-field, epifluorescence microscopy. PbS QDs were synthesized with a controllable size; well-defined exciton absorbance peaks could be tuned between 765 nm and 1800 nm. Of particular note is the ability to synthesize very small QDs, which allows for the detection of

fluorescence with high sensitivity silicon detectors. Size distributions were determined from optical spectra and exhibited standard deviations of  $\pm 10\%$ . Colloidal samples were spun cast in 0.1% poly(methylmethacrylate)-toluene (g/mL) on crystalline quartz substrates. At extreme dilutions near the single dot level, we observe fluorescence intermittency, or "blinking," a hallmark of single fluorophores. As expected for single fluorophores, a narrowing of the fluorescence spectra relative to the ensemble was also observed. We found that the fluorescence maximum irreversibly blue shifts with longer integration times and higher excitation intensities, indicative of a photo-induced degradation. Photobleaching of the majority of PbS QDs occurred in 30 sec. An analysis of the blinking statistics will be discussed.

**2:15 PM O12.3**

**Local Transport Imaging in a SnO<sub>2</sub> Nanobelt Sensor in a "Real World" Environment.** Andrei Kolmakov<sup>1</sup>, Y. Lilach<sup>1</sup>, M. Moskovits<sup>1</sup>, J. Shin<sup>2,3</sup>, Arthur P. Baddorf<sup>3</sup> and S. V. Kalinin<sup>3</sup>;  
<sup>1</sup>Department of Chemistry and Biochemistry, The University of California, Santa Barbara, California; <sup>2</sup>Department of Physics and Astronomy, The University of Tennessee, Knoxville, Tennessee; <sup>3</sup>Condensed Matter Sciences Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Electronic transport in a SnO<sub>2</sub> quasi 1D nanostructure device in a field effect transistor configuration is studied with a combination of macroscopic transport measurements and Scanning Probe Microscopy (SPM). The geometric structure of the nanobelt is determined from atomic force microscopy images that provide the height of the nanowire and scanning electron microscopy images that yield the cross-section. To address local transport behavior in the nanobelt, ac and dc potential distributions in an operating sensor device were measured using scanning impedance microscopy and Scanning Surface Potential Microscopy (SSPM) correspondingly. SSPM images exhibit dc potential drops at the contacts and at the local defects, which are the primary electroactive elements in the nanowire circuit. The dc transport measurements are strongly affected by the field-induced surface charge on the nanowire and gate oxide, low mobilities of which results in relaxation times of order of hours at room temperature. Under the conditions when charging effects are minimal, the potential distribution along the nanowires under positive and negative biases is strongly asymmetric, indicative of the presence of rectifying elements in the circuit. SPM is used to measure the current-voltage characteristic of individual electroactive elements in nanowires circuit. Combined with macroscopic sensing characteristics, this provides an opportunity for a spatially-resolved understanding of sensing and memory mechanisms in these devices. Research was sponsored by the U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC and by the National Science Foundation grant DMR-0072998. Research performed as a Eugene P. Wigner Fellow (SVK). Research at USCB was supported via AFOSR DURINT grant F49620-01-1-0459

SESSION O13: Future SPM Challenges and Opportunities

**PANEL DISCUSSION**  
 Thursday, December 2, 2004  
 Commonwealth (Sheraton)  
 3:00 PM - 5:00 PM

Chair: Ward Plummer

The spectacular progress of scanning probe microscopies in the last two decades have brought about new opportunities in condensed matter physics, chemistry, biology and materials science. The examples are as diverse as imaging and quantification of low dimensional systems, correlated electron behavior and quantum phenomena, surface reactivity and reaction mechanisms, electrical, optical, electromechanical, magnetic and elastic properties. This incredible progress is in large part due to the development of advanced microscopes capable of measurements under conditions ranging from ambient to liquid to ultra high vacuum, temperatures from milliKelvin to hundreds degree, high magnetic fields. Recently developed SPMs are capable of spatial resolutions down to tens of picometers. The development of advanced SPM instrumentation is a complex task requiring strong multidisciplinary expertise both in technical aspects of microscopy and instrumentation and chemistry, physics or biology. The purpose of this panel session is to provide a forum for a discussion of the current needs for advanced SPM instrumentation development in US and the equally important aspect of transferring new developments to the scientific community. The discussion will be initiated by several leaders in the field that will present their perspective on the development of advanced SPM techniques and approaches for disseminating new instruments and techniques to the community. In the second part, the representatives

from various federal agencies will discuss their agendas and potential role of SPM development. The invited speakers include:

E. Ward Plummer (University of Tennessee and ORNL)  
Lukas Novotny (University of Rochester)  
Katryn Wahl (Naval Research Lab)  
Dawn Bonnell (University of Pennsylvania)  
Clayton Teague (National Nanotechnology Coordination Office)  
Jim Murday (Office of Naval Research, DOD)  
Tof Carim and Jim Horowitz (DOE, Basic Energy Sciences)  
G. X. Tessema (National Science Foundation)

SESSION O14: Poster Session  
Chair: Lucas M. Eng  
Thursday Evening, December 2, 2004  
8:00 PM  
Exhibition Hall D (Hynes)

#### **O14.1**

**Development of Ultra-high Vacuum Scanning Nonlinear Dielectric Microscopy and Near Atomic Scale Observation of Ferroelectric Material Surfaces.** Hiroyuki Odagawa and Yasuo Cho; Tohoku University, Sendai, Japan.

We have proposed and developed a purely electrical technique for imaging the state of ferroelectric polarization and local crystal anisotropy of dielectric materials called "scanning nonlinear dielectric microscopy" (SNDM) [1]. To date, the spatial resolution of SNDM has been improved down to 0.5 nm, exceeding piezoelectric scanning force microscopy in the resolution. [2] SNDM does not use a force detection technique but use an electrical technique; SNDM measure a slight variation of capacitance in the top of a probe tip under applying an electric field. Therefore we expect that SNDM can be applied to an inactive insulator surface, in which it is difficult to use another scanning probe microscopy because of the weak interaction between sample and probe tip. In this paper, we will describe a newly developed ultra-high vacuum type scanning nonlinear dielectric microscope and measurement results of ferroelectric materials. In this experiment, ultra-high vacuum chambers are used to remove an undesirable influence of an adsorbed layer on the sample surface, and the samples are prepared by ion beam cleaning or by cleaving in the ultra-high vacuum atmosphere. One of the experiments is a observation of a cleaving (012) surface in LiTaO<sub>3</sub> crystal. We can clearly observe a striped pattern in the period about 0.3nm corresponding to the sub-lattice period. From this image we confirm that SNDM can be applied to angstrom order measurement of a physical property in ferroelectric insulator materials, though an interpretation of this image is the next step. [1] Y. Cho, A. Kiriwara and T. Saeki: Rev. Sci. Instrum. Vol.6 (1996) 2297. [2] H. Odagawa and Y. Cho, Surface Science, Vol. 463 (2000) L621.

#### **O14.2**

**Raman and Rayleigh Analyses and Smart Imaging of Nanophases and Nanosized Materials: Alternatives Techniques to SEM, TEM and AFM.** Philippe L. Colombari, LADIR UMR7075 UPMC, CNRS, Thiais, France.

Technological advances have increased significantly the performance of Raman instruments over the past decade. On-site measurements are now relatively easy, and controlled mapping of materials and devices allows consecutive spectra to be recorded stepwise with a lateral spatial resolution close to 0.1 mm. 100x100 point maps can be recorded in a reasonable timescale with high sensitivity Raman instruments. The Raman spectral parameters in these maps can be used to generate smart images showing, for example, compositional, structural, short-range order, or particle size variation and information. This is important to the study of nanostructured/nanocrystalline materials, since most of their mechanical/electrical properties are controlled by particle size. Raman spectra and their smart images can be used to understand and even predict some properties. In addition, in some case (for example a colored phase dispersed in a translucent matrix), the elastic (Rayleigh) scattering, which is orders of magnitude higher than the inelastic Raman scattering can be used to obtain quasi-instantaneous images of a materials skin, which is important, for example in corrosion studies, for observing surface composition or roughness change. Rayleigh imaging can offer very similar information as AFM for 2nd phase carbon-containing materials. Unique properties (conductivity, diffusion, reactivity, sintering, mechanical strength) have been reported for nanostructured materials, all of which result from the interfacial characteristics. Raman scattering is a unique tool for the characterization of such materials, providing information on the chemical bond arrangement and short-range order in amorphous, nanocrystalline or nanosized phases. This paper gives an overview of the information extracted from Raman spectra in nanomaterials

chosen among coloured nanophases (pigments, carbon-containing materials), electrolytes films (ZrO<sub>2</sub>, CeO<sub>2</sub>), electrode films (Li-intercalated InVO<sub>4</sub>) and fibres. The possibility of in situ analysis of working solid-state devices (under mechanical stress or electrochemical cycling) will be discussed. Spectroscopy Europe 15/6 8-15 (2003); <http://www.spectroscopyeurope.com/>

#### **O14.3**

**Integrated Optics for Ballistic Electron Emission Luminescence Microscopy.** Ian Appelbaum<sup>1,2</sup>, Wei Yi<sup>2</sup>, K.J. Russell<sup>2</sup>, Venkatesh Narayanamurti<sup>2</sup>, M.P. Hanson<sup>3</sup> and A.C. Gossard<sup>3</sup>; <sup>1</sup>Electrical and Computer Engineering, University of Delaware, Newark, Delaware; <sup>2</sup>Gordon McKay Laboratory, Harvard University, Cambridge, Massachusetts; <sup>3</sup>Materials Department, University of California, Santa Barbara, California.

Ballistic Electron Emission Luminescence (BEEL) Microscopy employs a scanning tunneling probe to locally inject hot electrons into a light-emitting Schottky-barrier heterostructure. To image the buried luminescent layer, emitted photons must be detected by a photo-sensitive device. Unfortunately, the low injection current and poor photon extraction efficiency in the BEEL collector results in a weak signal only detectable in the far-field using single-photon detectors. To solve this problem, we have integrated a photon detector directly into the BEEL heterostructure, just below a luminescent quantum well. Results from solid-state metal-base hot electron transistors fabricated with this collector design indicate that more than 10% of the photons emitted by the quantum well excite photoelectrons in the detector region. In microscopy mode, typical tunneling currents of 1-10 nA result in a collector current of 10-100 pA; this is expected to give 1-10 pA BEEL photocurrent, well within the detection range of high-gain amplifiers.

#### **O14.4**

**Surface Exciton Energies Determined by Velocity Resolved Atomic Desorption.** Alan G. Joly<sup>2</sup>, Kenneth M. Beck<sup>2</sup>, Wayne P. Hess<sup>2</sup>, Peter V. Sushko<sup>1</sup>, Paolo E. Trevisanutto<sup>1</sup> and Alexander L. Shluger<sup>1</sup>; <sup>1</sup>Physics and Astronomy, University College London, London, United Kingdom; <sup>2</sup>William R. Wiley Environmental Molecular Sciences Laboratory, Pacific Northwest National Laboratory, Richland, Washington.

Probing the electronic structure of surface excitons is important for our understanding of the mechanisms of surface processes including photo-catalysis, local characterisation of nano-structures and application of NSOM and STM excitations. While the bulk exciton energies are generally well known, the surface excitons are very elusive species due to low detection sensitivity of conventional techniques. Recent advances in the REMPI technique have provided an extremely sensitive tool for detecting of photo-induced desorption of atoms. We have developed a new method, which uses velocities of photo-desorbed surface species to determine the energies of the surface excitons. The energies of photons are finely tuned so that to induce the hyper-thermal desorption of surface atoms, which is a signature of the surface exciton relaxation. The method has been applied to determine the surface exciton energies on alkali-halide and MgO surfaces. The energy shifts between the surface and bulk excitons range from about 3 eV for LiF to 0.2 eV in KI. They show a remarkable correlation with the inverse inter-atomic distance for the whole range of materials studied [4]. Our ab initio embedded cluster calculations demonstrate that the exciton energy shift is determined by several factors including the change in the potential and kinetic energies as well as different electronic structures of the excited states, and cannot be explained on the basis of purely electrostatic considerations. The surface exciton detection scheme developed for the desorption experiments is sensitive to as little as 10<sup>5</sup> atoms/cm<sup>3</sup> and can be extended to probe optical properties of topological defects at surfaces of other divalent materials. An important feature of this technique is that the excitation and desorption can be induced locally using NSOM or STM which provides a tool for the surface sculpturing. [1] W. P. Hess, A. G. Joly, D. P. Gerrity, K. M. Beck, P. V. Sushko, A. L. Shluger, J. Chem. Phys., 115(20), 9463, (2001) [2] A. G. Joly, K. M. Beck, M. Henyk, W. P. Hess, P. V. Sushko, A. L. Shluger, Surf. Sci., 544, L683, (2003) [3] K. M. Beck, N. F. Dupuis, P. Perozzo, W. P. Hess, P. V. Sushko, A. L. Shluger, J. Chem. Phys., 120(5), (2004) [4] W. P. Hess, A. G. Joly, K. M. Beck, P. V. Sushko, A. L. Shluger, Surf. Sci., (in press)

#### **O14.5**

**Designing Resonance Modes of AFM Cantilevers and its Application for Probing of Electric Properties and Operation with Ultrasmall Oscillation Amplitude.** Kenjiro Kimura<sup>1</sup>, Takahiro Furukawa<sup>1</sup>, Kei Kobayashi<sup>2</sup>, Hirofumi Yamada<sup>1,3</sup> and Kazumi Matsushige<sup>1,2</sup>; <sup>1</sup>Electronic Science Engineering, Kyoto Univ., Kyoto-shi, Japan; <sup>2</sup>International Innovation Center, Kyoto Univ., Kyoto-shi, Japan; <sup>3</sup>Core Research for Evolutional Science and Technology, Japan Science and Technology, Kyoto-shi.



In dynamic force microscopy (DFM), distance between the tip and the sample is regulated by maintaining its resonance frequency shift or its oscillation amplitude damping. Owing to a high Q-factor of microfabricated cantilever, weak interaction forces can be easily detected thus atomic or molecular resolution images have been obtained. Moreover, various techniques for probing local electric properties based on electric force detection, such as Kelvin-probe force microscopy (KFM) and scanning capacitance force microscopy (SCFM)[1, 2], has been derived from DFM. Sensitivity and lateral resolution for those techniques can be improved by tuning the modulation frequency of an external electric field (modulation frequency: fm) so that the probing frequency (fm for KFM and 3fm for SCFM) matches one of the mechanical resonance frequencies of the cantilever. In general, the first resonance frequency (f1) is utilized for regulating the tip-sample distance in DFM. Thus the lowest available resonance frequency is the second resonance frequency (f2) unless a two-pass (lift) mode is employed. However, since f2 is 6.3 times higher than f1 for rectangular cantilevers, it often becomes higher than the bandwidth of the deflection sensing system utilized in DFM, which is typically less than 1 MHz. Furthermore, effective spring constants at those higher resonance frequencies become quite large and thus not ideal for detecting weak interaction forces. In other words, there is a fixed relationship between the resonance frequency and the effective spring constant for all resonance modes. In order to overcome such limitations, we have newly designed a novel cantilever structure whose resonance frequency and spring constant at the second resonance mode could be adjusted independently without above-mentioned restrictions. We fabricated one or multiple resonators inside or outside of the main cantilever beam using a focused ion beam milling instrument. We tailored the resonance frequencies and the effective spring constants as optimized by the finite element method calculation and successfully utilized the novel cantilever for KFM and SCFM. Furthermore, another cantilever design for performing high-resolution DFM imaging was introduced. We fabricated a resonator whose vibration amplitude is much larger than that of a tip on the main cantilever beam. The design helps DFM operation at a very small amplitude without sacrificing signal-to-noise ratio of the deflection signal detected on the resonator.

#### O14.6

**High-Resolution Photochemical Reaction using Triplet-Sensitizer Probes.** Akira Otomo, Hideki Miki and Shinro Mashiko; Kansai Advanced Research Center, National Institute of Information and Communications Technology, Kobe, Hyogo, Japan.

We propose molecular scale photochemical reaction control using triplet-triplet energy transfer from a donor molecule attached on a probe to an acceptor on the insulator surface. Nanoscale photochemical reaction is expected to play important roll in both building complicated molecular device structure and nanoscale lithography for semiconductor devices. Electric current induced chemical reaction has been widely investigated with SPM probes, however, it can be used only with conductive surfaces. For insulating surfaces, photochemical approach can be used with NSOM probes. However, resolution of NSOM is not small enough to apply for molecular scale reaction control. Since triplet-triplet energy transfer occurs a few nanometers range, resolution is determined by size of molecules attached on the SPM probe. It is challenging to attach only one molecule on the probe. However, because T-T interaction is effective only in the range of a few nanometers for the donor-acceptor distance, low surface density of the donor molecules on the probe becomes equivalent as a single molecule for T-T energy transfer. We investigated T-T energy transfer between sensitizer molecules (e.g. Michler's ketone) attached on the AFM probe and acceptor molecules (e.g. cinnamic acid) on the insulator substrate. We also discuss about fabrication of molecule-attached probes using dendrons.

#### O14.7

**Ultra-Violet Near-Field Scanning Optical Microscopy of 1-Dimensional ZnO Nanostructures.** Dake Wang<sup>1</sup>, Heewon Seo<sup>1</sup>, Yonhua Tzeng<sup>2</sup>, Chin-Che Tin<sup>1</sup>, Michael Bozack<sup>1</sup>, John Williams<sup>1</sup> and Minseo Park<sup>1</sup>; <sup>1</sup>Department of Physics, Auburn University, Auburn, Alabama; <sup>2</sup>Department of Electrical and Computer Engineering, Auburn University, Auburn, Alabama.

Near-field scanning optical microscopy (NSOM) is an emerging technique for nanoscale optical characterization. Our NSOM system is capable of collecting UV fluorescence signal from the sample. The 325 nm line of HeCd laser was used as an excitation. Zinc oxide (ZnO) is an interesting material for short-wavelength optoelectronics due to its wide band gap. The nanostructures of ZnO are also intriguing since a variety of morphology can be obtained by employing a different processing parameter. In our laboratory, different shapes of the 1-dimensional ZnO nanostructures were successfully synthesized using

thermal chemical vapor deposition. The photocurrent from the aligned nanowires was measured using a conductive scanning probe tip and lock-in amplifier. The morphology of the sample was studied using scanning electron microscopy and atomic force microscopy. Both apertured and apertureless NSOM were employed for analysis. Near-field fluorescence image of the single ZnO nanowire will be presented. Spectroscopic information from the nanowires will also be presented.

#### O14.8

**Study of Vertically Aligned Carbon Nanotubes by Atomic Force Microscopy and Transmission Electron Microscopy.**

Oxana Vasilievna Kharissova<sup>1</sup>, Ubaldo Ortiz<sup>2</sup>, Eduardo Perez Tijerina<sup>1</sup> and Jesus Rangel Cardenas<sup>1</sup>; <sup>1</sup>FCFM, UANL, Monterrey, Mexico; <sup>2</sup>FIME, UANL, Monterrey, Mexico.

Since their discovery, carbon nanotubes, both single-walled and multiwalled, have been a focus in materials research. Fundamental research and application development hinge on high-quality nanotube materials and controlled routes to their organization and assembly. The development of a highly efficient one-step technique was carried out to obtain long and aligned carbon nanotubes with or without Fe filling. The synthesis, characterization, and modification of individual molecules, one dimensional (1D) crystal have been investigated in several laboratories. The aligned carbon nanotubes (CNTs) were synthesized by microwave (MW) irradiation heating from a ferrocene. In this research, Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM) are used for study the growth of aligned carbon nanotubes by microwave heating. As a contribution of this method, the aligned multi-layer carbon nanotubes were obtained. CNTs have a metal particle at the tip of each tube. This carbon nanostructure promises to become important in fuel cells and in nanoscale engineering of other systems in which electrical, mechanical, and chemical interactions are integrated to produce macroscale effects. The morphology of the carbon nanotubes was studied by Scanning Electron Microscopy, Atomic Force Microscopy, and Transmission Electron Microscopy. Measurements of distinct angles, distances, and spaces between waves were made.

#### O14.9

**Microscopy Study of Polydispersed Nanoparticles.**

Jacquelynn Keefe McGuinness<sup>1</sup>, Christine Caragianis Broadbridge<sup>1</sup>, E. Anderson<sup>1</sup>, J. Lovering<sup>1</sup>, D. Day<sup>1</sup>, A. Lehman<sup>2</sup>, K. H. Wee<sup>2</sup>, S. X. Luo<sup>3</sup>, S. Calvin<sup>3</sup>, S. A. Morrison<sup>4</sup> and L. K. Kurihara<sup>4</sup>; <sup>1</sup>Department of Physics, Southern Connecticut State University, New Haven, Connecticut; <sup>2</sup>Facility for Electron Microscopy, Trinity College, Hartford, Connecticut; <sup>3</sup>Department of Physics, Sarah Lawrence College, Bronxville, New York; <sup>4</sup>Materials Physics, Naval Research Laboratory, Washington, District of Columbia.

Samples of platinum nanoparticles exhibiting moderate polydispersion were studied by transmission electron microscopy (TEM) and atomic force microscopy (AFM), coupled with x-ray diffraction (XRD) and extended x-ray absorption fine structure (EXAFS). While TEM and AFM yielded a size distribution for each sample, XRD (using Scherrer analysis), and EXAFS (using a homogeneous spherical model) were used to determine a mean particle size. Since XRD is weighted by the square of the volume and EXAFS by less than the volume, XRD is expected to generally yield a larger mean value than EXAFS. TEM and AFM are expected to yield the highest precision. The focus of this study is TEM and AFM (non-contact and contact) imaging of nanoparticle dispersions and the potential impact of specimen preparation on the size distributions measured by these imaging techniques. The TEM and AFM data collected revealed distributions that were compared with the results of XRD and EXAFS experiments. In this way, the relative merits of XRD and EXAFS for providing estimates of particle size were evaluated while the potential impact of sample preparation on TEM and AFM data collection was determined.

#### O14.10

**Photoresponse of a Single CdS Nanoribbon Device.** An Jin<sup>1</sup>, Chen Jian<sup>1</sup>, Xue Kun<sup>1</sup>, Li Quan<sup>2</sup> and Xu Jianbin<sup>1</sup>; <sup>1</sup>Department of Electronic Engineering and Material Science and Technology Research Center, The Chinese University of Hong Kong, Shatin, NT, Hong Kong SAR, China; <sup>2</sup>Department of Physics and Material Science and Technology Research Center, The Chinese University of Hong Kong, Shatin, NT, Hong Kong SAR, China.

We report on a study of individual CdS nanoribbon field effect transistors (FET) fabricated on SiO<sub>2</sub>/p-Si substrates. The CdS nanoribbon devices has exhibited n-channel normal-on FET characteristics. The field effect mobility is around 50 cm<sup>2</sup>/V.s, the threshold voltage -27 V, and the on/off current ratio of 10<sup>4</sup>, respectively. The photoresponse properties of CdS nanoribbons have been studied. The magnitude of photocurrent is found to increase linearly with the low light intensity, and saturate with the high light

intensity. The on-off ratio is as high as  $10^5$ . Also it is observed that the photocurrent depends on the light polarization, which is attributed to the crystal orientation of CdS, not to the quantum confinement effect widely found in carbon nanotubes and some nanowires. From the photocurrent of CdS versus light wavelength, it is shown that the CdS nanoribbons have a significant photoresponse below the wavelength of 520nm, with a fast photoresponse and decent stability. Scanning probe microscopy has been employed to study the contact properties between n-CdS nanoribbons with different metals. The surface potential (SP) difference at CdS/Au and CdS/Al interface have been measured with the aid of scanning Kelvin probe microscopy. It is found that the Au/CdS contact forms a larger barrier height than that of the CdS /Al, which is consistent with I-V characteristics. Under illumination, it is found that the surface potential signal of CdS increases with the light intensity, it can be ascribed to the surface photovoltage effect.

#### O14.11

**Structure, Growth Behavior and Electric Transport Property of ZnSe Nanowires.** Ning Wang, Y. Cai, S.K. Chan, Y.F. Chan and I.K. Sou; Physics Department, The Hong Kong University of Science & Technology, Hong Kong, Hong Kong.

Single crystalline ZnSe nanowires with uniform diameters (about 10nm) have been grown based on Au-catalyzed vapor-liquid-solid reaction by molecule beam epitaxy [1]. As determined by electron diffraction and high-resolution transmission electron microscopy (HRTEM), these nanowires grew epitaxially on GaP or GaAs substrates. We have observed that the diameters of the pre-formed Au-catalysts determined the nanowire growth direction. The growth direction of a ZnSe nanowire with a diameter greater than 30nm was generally along the  $\langle 111 \rangle$  direction. A 20nm ZnSe nanowire grew mainly along the  $\langle 112 \rangle$  direction. Most thin ZnSe nanowires with diameters smaller than 10nm grew along  $\langle 110 \rangle$  direction. In the earlier growth stage, the nano catalysts in the form of Au-Ga alloy nucleated on the substrate surface. Interface and surface structures played important roles for the formation and growth of the nanowires. We found that ultra thin ZnSe nanowires preferred the  $\langle 110 \rangle$  growth. The electric transport measurement of individual ZnSe nanowires has been carried out in-situ in a STM/HRTEM. ZnSe nanowires were determined to be P-type semiconductor. Au-catalyst on the tip of each ZnSe nanowire formed a Schottky barrier which showed rectifying behaviors. However, the conductivity of the nanowire was influenced by its surface states. A large electric current passing through ZnSe single nanowire resulted in material transport along the nanowire. [1] Y. F. Chan, X. F. Duan, S. K. Chan, I. K. Sou, X. X. Zhang, and N. Wang, "ZnSe nanowires epitaxially grown on GaP(111) substrates by molecular-beam epitaxy", Appl. Phys. Lett. 83 (2003)2665.

#### O14.12

Abstract Withdrawn

#### O14.13 TRANSFERRED TO O2.5

#### O14.14

**Atomic Force Microscopy Imaging of Viruses.** Azam Ghafoor, Demir Akin, Amit Gupta and Rashid Bashir; Biomedical Engineering, Purdue University, West Lafayette, Indiana.

Imaging of virus particles are customarily done by scanning or transmission electron microscopy; however, these methods require the samples to be dried, fixed, coated, and laboriously prepared for imaging. These methods are incompatible with processing and analysis of virus particles in real time. Furthermore, during the preparation of the samples, the sample integrity may be compromised and their use in imaging of dynamic changes can be very limited. Atomic force microscopy not only allows for investigation of real-time imaging of virus particles but also give information on the topological and mechano-elastic features of the particles in three-dimensions which cannot be obtained via other imaging techniques. Here we describe the use of atomic force microscopy for imaging and characterization of the vaccinia virus particles in their native and non-native states. Vaccinia virus, a close relative to the smallpox virus, is one of the largest and complex structured viruses. Its lack of a symmetrical icosahedral or helical capsid has defied traditional biochemical techniques in producing high resolution images of its structural components. Using atomic force microscopy, the inherent complexities of the virus can be examined and detailed mechanical and topological properties of the virus can be determined. We will present results on the imaging of the virus particles and its capsids and also show our results of imaging of whole virus particles as compared to those that are lysed, with the long term goal of extraction of genetic material from within the viruses within micro-fluidic devices.

#### O14.15

**Evanescence Wave Microspectroscopy for Proteomics.** Mesut Eraslan<sup>2</sup>, Bennett Goldberg<sup>1,2</sup>, M. K. Hong<sup>1</sup>, Xihua Wang<sup>1</sup> and

Shyamsunder Erramilli<sup>1</sup>; <sup>1</sup>Physics Department & Center for Photonics, Boston University, Boston, Massachusetts; <sup>2</sup>Department of Electrical & Computer Engineering, Boston University, Boston, Massachusetts.

Infrared spectroscopy is a sensitive method for detection of proteins. Most biomolecules have characteristic normal modes of vibration that are infrared-active. The presence of protein molecules in solution can be inferred by measuring this intrinsic infrared absorption, with the amide I absorption band near  $1660 \text{ cm}^{-1}$  having the largest cross-section. Because IR absorption does not involve the use of perturbative stains or labels, its application to the newly emerging field of proteomics is promising. Several factors limit the use of IR absorption from the amide I band, such as the high absorption of infrared radiation due to liquid water, and the large wavelengths associated with the amide I band that reduce the spatial resolution for surface microscopy applications. Both of these limitations can be overcome using evanescent wave coupling, by a suitably designed numerical aperture increasing lens (NAIIL). Here we describe an investigation of hemispherical lenses made of the wide-bandgap semiconductor GaP for IR evanescent wave absorption microscopy. A peptide of selected sequence, or an antibody can be attached covalently to the flat surface of the lens using standard protocols. IR radiation from a femtosecond tunable OPA laser source is incident on the curved face of the hemisphere. An evanescent wave at the flat surface couples into the peptide or antibody and is used to measure the absorbance of the solution above the surface. When a protein binds to the surface, a change in absorbance is detected due to the presence of increased amide I absorption. Past studies have attempted to use Germanium for such studies, because of its high refractive index  $n \approx 4$ . Attaching peptides to the surface of Ge is not easy. Further, for proteomics applications, detection from an array of closely spaced spots is desired, along with a co-registered optical image for convenience. It is difficult to obtain a co-registered optical image of the peptide spots from Ge which is not transparent in the visible region. In order to extend this method to an array of peptide spots suitable for proteomics, while simultaneously obtaining a co-registered optical image, hemispherical lenses of GaP are therefore promising. The refractive index of GaP ( $n \approx 3$ ) is higher than that for materials like ZnSe. Characterization of the topography of the polished GaP hemispheres shows that the flatness is within the  $\lambda/4$  tolerance in the mid-infrared region. The feasibility of detecting changes in the amide I region for characterizing proteome arrays will be discussed. Acknowledgements. We gratefully acknowledge support from the National Science Foundation Award DBI-0242697 and the Department of Defense.

#### O14.16

**Transport properties of DNA with respect to the linker molecules by Scanning Probe Microscopy.** Jeong min Son<sup>1</sup>, Nam ju Lee<sup>1</sup>, Yong Sang Kim<sup>3</sup>, Chi Jung Kang<sup>1</sup> and D. Jeon<sup>2</sup>; <sup>1</sup>Physics, Myongji University, Yongin, Kyunggido, South Korea; <sup>2</sup>Physics education, Seoul National University, Seoul, South Korea; <sup>3</sup>Electric Engineering, Myongji University, Yongin, South Korea.

To find out the effect of junction resistance between biomolecule such as DNA and inorganic substrate for studying the transport property, we used local probe microscopy with different linker molecules. Gold dots deposited on the silicon substrate and lambda DNA that has thiol group and biotin at either end were prepared for this experiment. Electrical property according to the linker molecule and substrate combination is characterized. We also present the local electrical properties of DNA imaged and analyzed by electrostatic force microscopy

#### O14.17

**Photo-electron emission microscopy of polarity-patterned materials.** Brian J. Rodriguez<sup>1</sup>, Woochul Yang<sup>1</sup>, Alexei Gruverman<sup>2</sup> and Robert J. Nemanich<sup>1</sup>; <sup>1</sup>Physics, North Carolina State University, Raleigh, North Carolina; <sup>2</sup>Materials Science and Engineering, North Carolina State University, Raleigh, North Carolina.

We report variable photon energy photoelectron emission microscopy (PEEM) observation of polarity contrast and measurement of the photo-threshold from polarity-patterned epitaxial GaN films and ferroelectric LiNbO<sub>3</sub> (LNO) single crystals and PbZrTiO<sub>3</sub> (PZT) thin films. Additionally, scanning probe microscopy techniques are employed to determine domain orientation, surface charge and surface potential. The photo-electrons were excited with spontaneous emission from the tunable UV-free electron laser at Duke University. For a cleaned GaN film with laterally patterned Ga- and N-face polarities, we found a higher photoelectric yield and a lower photo-threshold for the N-face regions ( $\approx 4.9 \text{ eV}$ ) compared with the Ga-face regions ( $> 6.3 \text{ eV}$ ). In LNO and PZT, bright emission and lower photo-thresholds were observed for the negatively poled domains. For LNO, the measured photo-threshold was  $\approx 4.6 \text{ eV}$  at the negative domain and

6.2 eV at the positive domain, while for PZT, the threshold of the negative domain was less than 4.3 eV. The PEEM polarity contrast is discussed in terms of the internal screening from free carriers and defects and the external screening due to absorbed ions.

#### **O14.18**

**Abstract Withdrawn**

SESSION O15: Dynamic Phenomena in Soft Condensed Matter

Chair: Sergei Kalinin

Friday Morning, December 3, 2004  
Commonwealth (Sheraton)

#### **8:30 AM \*O15.1**

**Charge Transport in Alkanethiols and OPEs Probed by STM.**  
James Batteas, Carlos Gonzalez and Yamil Simon-Manso; NIST, Gaithersburg, Maryland.

A significant challenge exists in understanding and predicting electron transport in metal-molecule-metal junctions important in molecular based electronic and optical systems. As the electrical behavior of molecules depends on their chemistry and organization at metal contacts, we have carried out a series of STM experiments on alkanethiols with varying molecular orientations, as well as molecular wires of unsubstituted and fluorinated oligo-phenylene-ethynylene (OPE) compounds. The adsorption of alkanethiols on Au results in domains of several architectures, ranging from molecules in closed packed structures covalently linked through the formation of a thiolate-Au surface bond with the molecular axis near perpendicular to the surface, to lower density phases with the molecular axis parallel to the surface. As alkanethiols are frequently employed as test cases for conductance in metal-molecule-metal junctions, one may envision that a distribution of orientations may exist in many such studies. Here we have employed STM to investigate the transport properties of several phases of dodecanethiol on Au. For domains with the molecules chemisorbed to the surface with the molecular axis orientated relatively perpendicular to the surface, significant asymmetry is observed in the I-V spectra between the positive and negative bias regions of the I-V curve which can be associated with the differences in the metal-molecule-metal contact configuration. For physisorbed molecular domains with the molecules laying flat on the surface, a near symmetric tunnel junctions are formed resulting in nearly symmetric I-V curves. STM investigations of unsubstituted and fluorinated oligo-phenylene-ethynylene (OPE) compounds find that the addition of the F group to the molecule results in an increased rectifying behavior as compared to the unsubstituted system. In conjunction with the STM measurements of the OPEs, conductance of the molecule-metal junctions based on Density Functional Theory calculations of modified quasi-molecular Green functions in a capacitor-like electric field have also been employed to compare the effects of orientation on the transport properties, with excellent agreement between theory and experiment.

#### **9:00 AM O15.2**

**Scanning Probe Recognition Microscopy Investigation of Elastic Properties of Tissue Scaffolding.** Q. Chen<sup>1</sup>, Y. Fan<sup>1</sup>, V. M. Ayres<sup>1</sup>, L. Udpa<sup>1</sup>, M.S. Schindler<sup>1</sup> and A.F. Rice<sup>2</sup>; <sup>1</sup>Michigan State University, East Lansing, Michigan; <sup>2</sup>Veeco Metrology Group, Santa Barbara, California.

Scanning probe recognition microscopy is a new scanning probe capability under development within our group to reliably return to and directly interact with a specific nanoscale feature of interest, without the use of a zoom box with its thermal drift and local origin difficulties. It is a recognition-driven and learning approach, made possible through combining SPM piezoelectric implementation with on-line image processing and dynamically adaptive learning algorithms. Segmentation plus a recognized pattern is implemented within a scan plan and used to guide the tip in a recognition-driven return to a specific site. The specific application focus of our group is on the development of Scanning probe recognition microscopy for nanobiological investigations and we will present our results within this context. In previous work, we have successfully recognized and classified tubular versus biological objects from experimental AFM images using a method based on normalized central moments. We have also extended this work to include recognition schemes appropriate for more subtle differences between biological objects of similar globular shape by adding the Continuous Wavelet Transform (CWT) with a differential Gaussian mother wavelet [2]. Normalized central moments are translation, rotation and scale invariant, and the 2-D continuous wavelet transform allows multi-scale analysis of images. Thus, these two methods together can be applied to analyze biological objects of any scale. In the present work, scanning probe recognition microscopy is used in a direct investigation of the surface

and elastic properties along individual tubules within a tissue scaffolding matrix. Elastic properties are indicated as important influences on actin polymerization and cell pseudopodia extension/contraction and adhesion. 1. Qian Chen, Virginia Ayres, and Lalita Udpa, Biological Investigation Using Scanning Probe Recognition Microscopy, Proceedings of the 3rd IEEE Conference on Nanotechnology (IEEE-NANO 2003), Vol. 2, pp. 863-865 (2003) 2. B. Goolsby, Q. Chen, L. Udpa, Y. Fan, R. Samona, B. Bhooravan, F. M. Salam, D. H. Wang, and V. M. Ayres, Scanning Probe Microscopy with Landmark Referenced Control For Direct Biological Investigations, J. Nanosci. Nanotech., Vol. 3, No. 4, PP.347-350 (2003).

#### **9:15 AM O15.3**

**Electronic Properties of Modified Surfaces Using Contact and Non-Contact Scanning Probe Microscopy Techniques and SECM.** Aaron K. Neufeld<sup>1</sup>, Anthony O'Mullane<sup>2</sup> and Alan M. Bond<sup>2</sup>; <sup>1</sup>Manufacturing and Infrastructure Technology, CSIRO Australia, Highett, Victoria, Australia; <sup>2</sup>School of Chemistry, Monash University, Clayton, Victoria, Australia.

During the course of electrochemical experiments with tetracyanoquinodimethane (TCNQ) modified electrodes in contact with aqueous copper containing electrolytes, the incorporation and expulsion of copper ions in and out of the compound are coincident with nucleation and growth processes and significant crystal fragmentation. This results in the formation of particles with dimensions of the order of 10's of nanometres. During reduction of TCNQ and intercalation of copper ions, different phases of the semiconducting compound CuTCNQ are formed.[1] The preparation of both conducting and insulating substrates with electroactive TCNQ and CuTCNQ particles of variable size have been made by dip and spin coating procedures. New results from the study of the nucleation and growth process suggest that the phase and subsequently quite different electronic properties of CuTCNQ is dependent on the size of particles that decorate the electrode surface. Combining atomic force microscope (AFM) based methods that interrogate the morphological and electronic properties of nanometre sized particles in combination with use of a scanning electrochemical microscope (SECM) is a new approach that has proved highly valuable in understanding the particles' highly complex behaviour. [1] Neufeld, A. K.; Madsen, I.; Bond, A. M.; Hogan, C. F. Chemistry of Materials 2003, 15, 3573-3585.

#### **9:30 AM O15.4**

**AFM Method for Nanostructures Thermoelectric Properties Characterization.** Claudiu Hapenciuc and Theodorian Borca-Tasciuc; Mechanical Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, Troy, New York.

Quantum and size effects in nanostructures may lead to significant enhancements of their thermoelectric energy conversion efficiency. Hence, techniques able to perform fast and reliable local measurements of electrical conductivity, Seebeck coefficient, and thermal conductivity are of high interest. However, experimental characterization of thermoelectric transport properties in nanoscale samples is a challenging task, due to difficulties such as contact resistance and the measurement of the local temperature and heat flow to the sample. This work reports an AFM based method for measurement of thermoelectric properties of nanostructures such as thin-films of nanoparticles and nanowires. In this method a resistively heated AFM tip brought in contact with the sample gives rise to a temperature drop and a Seebeck voltage. The temperature drop and the heat transfer rate to the sample is estimated from the measured temperature rise of the tip using a heat transfer model which also takes into account the electrical and thermal resistance at the tip-sample interface. Thermal conductivity is then determined from the measured thermal conductance. The Seebeck coefficient value is calculated using the estimated temperature drop and the Seebeck voltage directly measured across the sample. Electrical conductivity is determined from electrical resistance measurements. The method was tested on bulk and nanoparticle Bi<sub>2</sub>Te<sub>3</sub> films deposited on glass substrates

#### **9:45 AM O15.5**

**Effects of Surface Functionality and Humidity on the Adhesion Force and Chemical Contrast Measured with AFM.** Tinh Nguyen, Xiaohong Gu, Lijiang Chen, Michael Fasolka, Kim Briggman, Jeeseong Hwang and Jon Martin; NIST, Gaithersburg, Maryland.

Mapping chemical heterogeneity of surfaces at the nanoscale spatial resolution is the subject of great interest in materials sciences and technologies. We have demonstrated previously that by using a combination of hydrophilic AFM probes and manipulating the relative humidity (RH) of the tip-sample environment in an AFM, hydrophilic/hydrophobic regions in model self-assembled monolayer

(SAM) and polymer samples can be readily imaged. This study investigated the effects of environmental relative humidity and surface functionalities on the tip-sample adhesion force and chemical contrast of gradient SAMs. Samples of chemically-homogeneous SAMs having CH<sub>3</sub>, OH, NH<sub>2</sub> or COOH surface terminated groups prepared by thiol chemistry and hydrophilic/hydrophobic gradient SAMs fabricated by silanization followed by UV/Ozone treatment were employed. CH<sub>3</sub>, COOH, and OH terminated AFM tips were used as the probes. The chemical specificity of both the samples and AFM tips were characterized by a variety of spectroscopic techniques. A humidity generator connected to a specially-designed humidity chamber fitted to an AFM is employed to provide highly controlled tip-sample RH between 0 and 95 %. For all tip-sample combinations, the AFM image contrast between the hydrophilic and hydrophobic regions is poor (non-distinguishable) when the tip-sample RH is below 50 %. With the hydrophilic tips, the chemical image contrast increases markedly with RH > 50 %. At high RH levels, even chemical domains that have small difference in the polar surface energy component could still be detected. Further, except for the hydrophobic tip where RH has little effect, other tip-sample adhesion forces show a maximum between 50 and 70 % RH. Mechanism for the enhanced chemical contrast and tip-sample adhesion force in elevated humidities will be discussed.

#### 10:30 AM O15.6

**Kinetic Studies of Molecular Motion via Nanoshear: Issues of Surface, Bulk and Perturbation.** Greg Haugstad, Ronald Schmidt, Craig Dystra and Wayne Gladfelter; University of Minnesota, Minneapolis, Minnesota.

The temperature and rate dependence of shear response on polymers, under constant-velocity sliding or small-amplitude harmonic drive, has been examined by several groups within the past several years using AFM methods. The "local" aspect of these measurements has regarded mainly the vertical rather than lateral dimension, interrogating near-surface response and contrasting with bulk response from conventional, macroscale methods. Interpretations often have assumed that the probe is sensing intrinsic "surface" dynamics; results then have been compared with conventional bulk characterization to conclude that the surface differs, or does not differ, from the bulk. In view of conflicting findings as briefly reviewed here, it is clear that *determining* the extent to which surface or bulk response is probed must be a focus of any AFM-based investigation. Moreover, the degree to which this response is *altered* by the tip interaction must be examined. We present several findings from the authors and other groups that have examined this "perturbation" question, wherein the effects of load as well as tribological heating and memory were systematically characterized. Our findings demonstrate that both sliding friction and small-amplitude harmonic shear modulation on polystyrene are inherently perturbative, mobilizing conformers in the former and penetrating to considerable depth in the latter. Based on these and other observations we suggest that AFM studies should not claim to measure intrinsic surface mobility on polymers without careful analyses of perturbation and depth penetration. This should include the presentation of *images* of the affected region following measurement procedures. We conclude that whereas AFM is a uniquely powerful probe to study the fundamentals of tribology, it is a problematic tool to study intrinsic surface dynamics.

#### 10:45 AM O15.7

**Micro-TA - An Analytical Tool in Materials Science.** Hartmut Rudolf Fischer, Innovative Materials, TNO TPD, Eindhoven, Netherlands.

The new analytical technique micro-thermal analysis (m-TA) will be introduced and evaluated. It combines the visualization power of atomic force microscopy (AFM) and its ability to image topography, phase shifts, friction, stiffness, and adhesion with the characterization capabilities of thermal analysis (thermal conductivity, micro-differential thermal analysis and micro-thermo mechanical analysis) resulting in a characterisation of surfaces with respect to their thermal and thermo-mechanical properties. The scanning mode may be used for the inspection of surfaces with respect to their thermal properties using a scanner with an active thermal sensor (heater, height and thermal sensor) as probe (tip), simultaneously acquiring topography, thermal conductivity and thermal diffusivity images. A second mode gives the option to perform a local thermo-mechanical analysis of discrete areas of a few square microns (L-TMA and L-CA), and detects at the same time during heating in contact mode changes of the sensor position and the heat flow to the sample in a very short time. Any thermodynamic phase transition, connected with a change in mechanical properties (softening, expansion, melting) and thermal properties (heat of fusion, change of heat capacity) will thus be detected by both methods. Therefore, such experiments surpass mere topographical mapping (commonly used in AFM imaging) because heat flow differences can be correlated to differences in chemical or physical structure. Applications for the analysis of many different types of materials including hard inorganic

coatings and bulk materials, microelectronic circuits, organic coatings and bulk materials as well as of soft materials are presented and critically discussed. An application of the technique as a tool for high-throughput analysis seems possible. Moreover, the use of the instrument for controlled surface treatment and patterning is demonstrated with different samples. This may be used for data storage as well as for the creation of super smooth polymeric surfaces. Also attempts to determine thermal conductivities on surfaces on a quantitative matter will be presented and discussed.