

# Monday Morning, November 13, 2006

## Applied Surface Science

Room 2005 - Session AS+BI+NS+NM-MoM

### Organic Surface Modification and Nanoscale Chemical Patterning

Moderator: R. Haasch, University of Illinois, Urbana

#### 8:00am AS+BI+NS+NM-MoM1 Biomolecular Lithography on GaAs Surfaces, A. Ivanisevic, Purdue University INVITED

Atomic force microscopy (AFM) was used to fabricate well-defined peptide templates onto GaAs surfaces via Dip-Pen Nanolithography (DPN). DPN is a powerful technique to write specific organic and/or inorganic molecules onto a surface with an AFM tip. In this work, DPN was used to construct arrays of peptides with nanometer features. TAT peptides (e.g. CGISYGRKKRRQRRR) which exhibit rapid uptake in cells, were patterned onto the surface in either contact or tapping mode. Several techniques were used for the characterization of the modified surfaces: X-ray photoelectron spectroscopy (XPS), Fourier Transforms Infrared (FT-IR) spectroscopy and contact angle. Transmission FT-IR provided structural information such as peptide conformation. The complementary analysis confirmed the binding of the peptide onto the substrates and allowed to quantify the density of immobilized peptides on a given surface. Furthermore, the nanoscopic features were successfully used in recognition experiments where an RNA sequence with a loop structure, known for its specific interaction with the peptide, was tested. The results in this report indicate that one can use nanolithographic strategies to pattern GaAs surfaces, and therefore provide a proof-of-concept experiment that can be transferred in complex microfabricated semiconductor architectures.

#### 8:40am AS+BI+NS+NM-MoM3 The Effect of Ring Substitution Position on the Structural Conformation of Mercaptobenzoic Acid Self-Assembled Monolayers on Au(111), J.R.I. Lee, Lawrence Livermore National Laboratory; T.M. Willey, J. Nilsson, L.J. Terminello, J.J. De Yoreo, T. van Buuren, Lawrence Livermore National Laboratory

Mercaptobenzoic acid (MBA) is a viable alternative for preparing SAMs with carboxyl functionality. These molecules, as opposed to carboxyl-terminated alkythiols, offer an extended pi-bonded system formed by the aryl and carboxyl groups facilitating charge transfer, and these extremely thin SAMs (< 8 Angstroms) have been shown via STM to contain a high degree of structural order. Furthermore, three different isomers of mercaptobenzoic acid may lead to differing surface properties. In this work, the orientation and bonding of self-assembled monolayers of the three positional isomers of mercaptobenzoic acid adsorbed on gold are investigated using near edge X-ray absorption fine structure (NEXAFS) spectroscopy and photoemission spectroscopy (PES). The isomer of MBA and solvent chosen in SAM preparation has considerable bearing upon film morphology. Monomers of 3- and 4-MBA assume an upright orientation on the Au substrates in monolayers prepared using an acetic acid in ethanol solvent. The aryl ring and carboxyl group of these molecules are tilted from the surface normal by a colatitudinal angle of ~ 30 degrees. Preparation of 4-MBA SAMs using pure ethanol solvent, a more traditional means of synthesis, had no appreciable effect upon the monomer orientation, but S(2p) PES measurements illustrate that it results in extensive bilayer formation via carboxyl group hydrogen-bonding between 4-MBA monomers. In 2-MBA monolayers prepared using acetic acid/ethanol solvent, the monomers adopt a more prostrate orientation on the Au substrates, in which the aryl ring and carboxyl group of the molecules are tilted ~ 50 degrees from the surface normal. This configuration is consistent with an interaction between both the mercaptan sulfur and carboxyl group of 2-MBA with the underlying substrate. This work was supported by the Division of Chemical Sciences, Office of Basic Energy Science, and performed under the auspices of the U.S. DOE by LLNL under contract No. W-7405-ENG-48.

#### 9:00am AS+BI+NS+NM-MoM4 XPS Analysis of Arenes and Proteins on Gold Substrates Attached by Electrochemical Reduction of Aryldiazonium Salts, J.L. Fenton, University of New Mexico; S.M. Dirk, D. Wheeler, Sandia National Labs; J.E. Fulghum, University of New Mexico

The properties of most materials can be altered by the attachment of molecules to their surface. Recent work has shown that diazonium salts can be used to attach aryl compounds to metal and semiconductor surfaces including silver, iron, platinum, silicon, and glassy carbon. Diazonium salts can be electrochemically reduced in acetonitrile leading to the attachment of aryl groups to the surface of a substrate. The attachment of aryl compounds using diazonium salts developed in our work, allows one to

attach aryl compounds with different organic moieties onto metal substrates including gold. Aryl organic layers can be made of different thicknesses, and stacked structures can be created by alternating layers of molecules. This work is then expanded upon to attach proteins to metal surfaces. This approach provides a methodology to generate arrayed proteins on electrode arrays without the use of microfluidic methods such as ink jetting etc. The development of protein arrays can often provide high-throughput, quantitative measurement of receptor-ligand complexation giving an understanding of protein function, modification, and regulation. X-ray photoelectron spectroscopy (XPS) is used to deduce the different types of chemistries in single molecule samples, stacked structures, and surface tethered proteins. Angle resolved XPS is used to determine the relative location of each molecule in the stacked structures and possible orientation of proteins as well as to deduce the existence of azo linkages.

#### 9:20am AS+BI+NS+NM-MoM5 Challenges in the Modification and Characterization of Two- and Three-Dimensional Biointerfaces, M. Textor, ETH Zurich, Switzerland INVITED

Surface modifications based on biochemical or biological principles are important tools for the fabrication of biosensor chips, biomedical devices such as implants, and of drug delivery carriers. Moreover, well-designed model biointerfaces have substantially contributed in the last decade to a better insight into fundamental aspects of cell-surface interaction. An overview will be given on tools enabling the surface engineer to tailor the interface of biomaterials, with special emphasis on the approach of eliminating non-specific adsorption and adding to such a silent surface biological functionalities. Preservation of active conformation and optimum presentation (orientation, density) of surface-immobilized moieties are particular challenges in this field. Different approaches to micro- and nanopatterning of surfaces, their specific advantages/disadvantages and applications in biorelated fields are discussed. Special emphasis is placed on methods that combine top-down (e.g., lithography) and bottom-up (self-assembly) approaches. Substantial new insight into the factors that govern cell-surface interactions and cell differentiation has recently been gained by using two-dimensional (2D) patterns. There is, however, an increasing interest to explore (stem) cell development in 3D microwells with well-controlled geometry (shape/size), surface chemistry and mechanical properties (substrate stiffness). The challenges of independently tailoring chemistry and structure/topography are addressed; self-assembly techniques are in this respect particularly useful. Finally, characterization/imaging techniques that allow the monitoring of biointerface reactions in situ, in real time and quantitatively are important tools; selected methods will be presented based on highly surface- and detection-sensitive evanescent-field-based sensing.

#### 10:20am AS+BI+NS+NM-MoM8 Nanoscale Chemical Patterning and Architectures, P.F. Nealey, University of Wisconsin INVITED

Diblock copolymers are self-assembling materials consisting of two polymer chains connected at one end that tend to form ordered nanostructures, including spheres, cylinders, and lamellae, whose shape and dimensions depend on the molecular weight and composition of the polymer. Block copolymer lithography refers to the use of these ordered structures in the form of thin films as templates for patterning through selective etching or deposition. Already block copolymer lithography has been used to pattern dense periodic arrays for the applications such as quantum dots, nanowires, magnetic storage media, increased capacitance gate devices and FLASH memory. One of the current goals in block copolymer nanolithography is to reproduce many of the characteristics of the lithographic process used ubiquitously in nanomanufacturing, including pattern perfection over macroscopic areas, the ability to pattern arbitrary and non-regular geometries, dimensional control of features within exacting tolerances and margins, and registration and overlay. Two strategies will be discussed to integrate self-assembling materials into existing manufacturing practices so as to achieve molecular-level process control and the ability to produce useful architectures: directed assembly of block copolymers on topographically patterned substrates, and directed assembly of block copolymers on chemically nanopatterned substrates.

#### 11:00am AS+BI+NS+NM-MoM10 Tailoring of Functional Nano-Patterned Surfaces for Biosensing Applications by Combination of Plasma Processes and Electron-Beam Lithography, F. Brétagnol, L. Ceriotti, A. Valsesia, T. Sasaki, D. Gilliland, G. Ceccone, P. Colpo, F. Rossi, IRC-IHCP-BMS, Italy

Fabrication of micro and nano-patterned surfaces with a well defined geometry and a controlled chemistry is a fundamental step for the development of bioengineered materials. Micro-arranged surfaces

containing functionalities such as cell or protein adhesive in a non non-adhesive matrix provide a very useful tool in a large field of applications (tissue engineering, cell behavior investigations, artificial growth of neurons networks?). Numerous methods have been successfully developed for the production of such surfaces including conventional photolithography, photochemistry, micro-contact printing, micro fluidic patterning. Nevertheless, the development of surfaces chemically patterned at nano scale is still a challenging issue for the implementation of new generation of miniaturized biochips and for the study of cell surface interactions. In this study, we present an innovative method for the fabrication of chemically nano-patterned surfaces. Maskless lithography based on electron-beam technique was successfully combined with plasma-based processes to create COOH terminated spots over a PEO-like matrix. Spots from micron to submicron size have been created. Quality control of the patterned surface was studied by Atomic Force Microscopy, XPS and ToF-SIMS analysis. Experiments with fluorescent proteins on the patterned surfaces exhibit a preferential adhesion on the active region showing the ability of this technique for the design of biosensing platforms.

**11:20am AS+BI+NS+NM-MoM11 Optically-Stimulated Surface Diffusion Exploited for Directed Self-Assembly on Amorphous Semiconductors, E.G. Seebauer, Y. Kondratenko, University of Illinois at Urbana-Champaign**

Nanoscale device fabrication technologies require toolsets for miniaturization and organization of materials at nanometer dimensions. Current toolsets have developed from two diametrically opposite strategies: top-down and bottom-up. This laboratory is taking a different approach based on a new physical mechanism for photostimulated diffusion discovered here. This new strategy combines attractive features of top-down and bottom-up approaches by exploiting the self-organization capabilities latent in amorphous materials, but in a way that can be controlled by optical or electron beam exposure tools. We have developed a new surface self-assembly method at the 10-200 nm length scale using amorphous semiconducting materials. Patterned optical or electron beam exposure yields a spatially varying surface mass flux that, when performed at an annealing temperature just at the cusp of crystallization, provides the extra nudge to crystallize subcritical nuclei in regions dictated by the light flux. The full-fledged crystallites then grow by surface diffusion and Ostwald ripening until the desired fraction of the film has accreted onto the original nuclei. We have demonstrated this technique with titanium dioxide as the substrate material. This scheme should apply to a wide variety of semiconducting materials on nearly arbitrary substrates to form nanoarrays, nanowalls, and possibly three-dimensional structures. Possible applications include chalcogenide semiconductors for data storage media; nanoparticles arrays for direct use in sensors and solar cells; and semiconductor arrays for indirect use as seed layers for the subsequent deposition of sintered particle films in fabricating advanced ceramics and devices such as rechargeable batteries, solar cells, gas sensors, and photonic band gap materials in solar windowpanes.

**11:40am AS+BI+NS+NM-MoM12 Nanowires and Nanodevices via Assembly of Clusters, S.A. Brown, Nano Cluster Devices Ltd, New Zealand**

We report the achievement of contacted electronic devices, self-assembled from atomic nanoclusters. Features of this technology, which overcomes the difficulties in the assembly of building blocks inherent to many bottom-up approaches to nanotechnology, include: Interchangeable cluster sources so as to have available a wide variety of cluster materials in a useful size range, allowing exploitation of novel structures and properties. Self-assembly methods which avoid time consuming positioning of building blocks. Use of lithographic processes which are compatible with both cluster deposition technology and standard microelectronics fabrication protocols. Understanding of basic physical assembly processes in order to predict and control device formation, including availability of suitable computer simulations. In this paper we will review the assembly methods developed, which include directed assembly in silicon V-grooves and on polymer-patterned surfaces, as well as stenciling techniques. We will then focus on the application of these techniques to one class of devices which have been realised i.e. cluster-assembled hydrogen sensors. These sensors are realised by the deposition of Pd clusters on a substrate such that one or several conduction paths are formed between a pair of contacts. The sensing principle relies on the expansion of Pd clusters and the resulting change of conductance as a result of their absorption of hydrogen and we have demonstrated very high sensitivities.

## Nanometer-scale Science and Technology

### Room 2016 - Session NS-MoM

#### Nanoscale Imaging Techniques

Moderator: M.C. Hersam, Northwestern University

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

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

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

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

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

MgO remains one of the most technologically essential surfaces due to its importance in catalysis. It has also been the subject of intense recent research as a substrate in nanocatalysis. Oxygen vacancies (F-centers) on the surface have been proposed to play an important role as nucleating centres for catalytic nanoparticles. Dynamic force microscopy (DFM) has

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the potential to image both the surface and any defects or adsorbates in atomic resolution, providing unprecedented details into surface processes. In this work we compare low temperature atomic resolution DFM imaging of the MgO (001) surface in UHV with first principles simulations of the tip-surface interaction. The comparison between theory and experiment is made possible by experimentally obtained site-specific force spectroscopy, which can be directly compared to computational predictions of the short range interaction with different tip models, therefore leading to unambiguous characterization of surface species and point defects. Tips are nanofabricated from silicon, but during DFM imaging, material is exchanged with the surface as well as with the ambient. Hence, we construct a set of probable tip models including adsorbates such as hydroxyl groups, hydrogen, magnesium and oxygen, as well as clean silicon tips. After testing imaging of a perfect area of the MgO(001) surface with two dozen most probable tip models, we establish a best fit to experiment and further simulate imaging of the characteristic defects seen on the surface in experiments.

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

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

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

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

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

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

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

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

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

High resolution imaging of ferroelectric materials is demonstrated using piezoresponse force microscopy (PFM) in an aqueous environment. In the last decade, PFM has been established as a powerful tool for nanoscale imaging, spectroscopy, domain patterning and lithography of ferroelectric thin films, as well as the characterization of capacitors used for ferroelectric memories and data storage. Recent work has demonstrated the applicability of PFM to biological systems where it is possible to image structural properties and molecular orientation with a sub-10 nm resolution. The primary factors limiting the resolution and sensitivity of PFM are electrostatic contributions to the signal and capillary forces. Here, we performed PFM in an aqueous environment to simultaneously minimize both the electrostatic and capillary interactions. A resolution on the order

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of 1-3 nm, approaching the theoretical domain wall width, as compared to a resolution of ~30 nm in ambient, is reported. The dynamic behavior of the cantilever was analyzed using conventional amplitude-frequency and 2D amplitude-frequency-bias spectroscopy. It is shown that the cantilever dynamics in liquid are significantly different from ambient conditions due to the higher viscosity and added mass effects. Imaging at frequencies corresponding to high-order cantilever resonances is shown to minimize these effects thus allowing sensitivities comparable to ambient conditions. The absence of both long-range electrostatic forces and capillary interactions results in the localization of the ac field to the tip-surface junction and allows the tip-surface contact area to be controlled. This unusual mechanism enables spatial resolutions approaching the intrinsic domain wall width. PFM in liquids will provide novel opportunities for high-resolution studies of ferroelectric materials, imaging of soft polymer materials, and the study of biological systems in physiological environments on, ultimately, the molecular level.

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

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

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

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

## Nanometer-scale Science and Technology Room 2016 - Session NS-MoA

### Nanoscale Structures and Characterization I

Moderator: J. Wendelken, Oak Ridge National Laboratory

**2:00pm NS-MoA1 Induced Water Condensation and Bridge Formation by Electric Fields in Atomic Force Microscopy, G.M. Sacha, A. Verdaguer, M. Salmeron,** Lawrence Berkeley National Laboratory

The formation of nanometer-sized water bridges between an Atomic Force Microscope (AFM) tip and a sample is a phenomenon that has produced a great interest in the last years. New applications in imaging and nanofabrication have driven efforts to understand nanometer-size capillarity. Water bridges play also an important role in contacts between objects, where it affects friction and energy dissipation. The interest in the topic has spurred theoretical efforts to model different aspects of capillarity meniscus formation. Most models are based on macroscopic approximations, molecular level grand canonical Monte Carlo Simulations and Density Functional Theory, all of them involving substantial computational effort. We have developed a simple analytical expression that can be used to determine in a quantitative way the presence and shape of the water film that forms between tip and sample in humid environments. This film grows under the influence of the electric field forming a meniscus that becomes unstable when a critical field is reached, at which point it suddenly forms a bridge between tip and surface. In AFM the capillary force bends the lever, which for small spring constants can bring the tip in contact with the surface. This is observed experimentally as a sudden jump-to-contact. Our approximation allows us to determine the distance and the voltage at which this capillary jump takes place. Apart from providing a simple way to calculate the critical field and distance it improves our understanding of the mechanisms of water induced jump-to-contact. Understanding and predicting water bridge formation is important for many applications and has implications in other physical processes including friction and adhesion in humid environments. @FootnoteText@ @footnote 1@ Gomez-MoAivas, S.; SÄienz, J.J.; Calleja, M. and Garcia, R. Phys. Rev. Lett. 2003, 91, 56101. @footnote 2@ Paramonov P. V. and Lyuksyutov S. F. J. Chem. Phys. 2005, 123, 084705.

**2:20pm NS-MoA2 Carbon Nanosheets, a Novel, Free-Standing, Two-Dimensional Carbon Nanostructure, B.C. Holloway, J.J. Wang, R.A. Outlaw, X. Zhao, M. Zhu, P. Miraldo, S. Wang, K. Hou,** College of William & Mary

This presentation summarizes our work on the synthesis and characterization of a new, free-standing, 2-D carbon nanostructure, carbon nanosheets, on a variety of substrates including metals, semiconductors, and insulators by RF plasma-enhanced chemical vapor deposition. SEM, TEM, SAED, Raman, XPS, AES, FTIR, and XRD all indicate that carbon nanosheets deposit as smooth graphite sheets which are up to 8 microns in height but are 1 nm or less in thickness. Such nanosheets consist of, at most, a few graphene layers. The nanosheets stand off the growth substrate in a manner similar to aligned nanotubes grown by chemical vapor deposition. However, in contrast to CVD nanotubes, nanosheets do not require a catalyst for growth and can be patterned after deposition using standard photolithography techniques. Results of thermodynamic modeling and parameter variations show that hydrogen etching promotes the formation of the atomically thin structures while the anisotropic dipole created in the graphene planes by the plasma sheath promotes the vertical orientation. Several applications which use the high surface area, vertical orientation, substrate flexibility and atomically sharp edges will also be outlined.

**3:00pm NS-MoA4 Morphology Control of Ferromagnetic Core-Shell Nanoparticles Electrodeposited on H-terminated Si(100), L.Y. Zhao, N. Panjwani, T. Chan, N. Heinig, K.T. Leung,** University of Waterloo, Canada

Our recent work on metal nanoparticles (Cu, Ni, Co) electrochemically deposited on an ultrathin polypyrrole film grown on a gold-coated silicon electrode shows that the morphology (size, shape, density and distribution) of these nanostructured materials can be easily controlled by varying the wet deposition conditions (pH, electrolyte concentration, deposition potential, charge, and current density), and the thickness and morphology of the polypyrrole film. Using similar electrochemical techniques and by manipulating the solution chemistry, we have recently obtained on a H-terminated Si(100) surface mono-sized, uniformly distributed Fe core-shell nanoparticles with several different morphologies: quantum dots of 4-10 nm in diameter, 30x120 nm "nano-rice", and 40-60 nm nanocubes. These

nanoparticles are found to consist primarily of a polycrystalline Fe metallic core and a mixed Fe oxides shell (of several nm thick) and all exhibit interesting magnetic properties. In the present work, we illustrate that morphological changes can be induced by controlling the substrate conditions, solution parameters and an external magnetic field.

**3:20pm NS-MoA5 Boron-based Nanostructures for High Temperature Energy Conversion, T. Xu,** The University of North Carolina at Charlotte

Thermoelectric (TE) materials with high figure-of-merit (ZT) are of fundamental and practical interest for energy conversion. Low-dimensional nanoscale materials provide new possibilities to improve ZT based on quantum effects. The use of quantum dots, wires and wells as TE materials is an active area of study. However, most investigations focus on TE nanomaterials (e.g., Bi@sub 2@Te@sub 3@, Bi) for refrigerator (cooling) applications. We propose to study a new class of boron-based TE nanomaterials that will operate at high temperature, and be used for power generation. In this presentation, recent experimental results on synthesis of n-type boron-based TE one-dimensional nanostructures (i.e., CaB@sub 6@, SrB@sub 6@ and BaB@sub 6@ nanowires) will be reported. The nanostructures were synthesized by pyrolysis of diborane (B@sub 2@H@sub 6@) over certain metal oxide powders (e.g., calcium oxide (CaO) for synthesis of CaB@sub 6@) at elevated temperature and low pressure. The experiments were performed in a home-built low pressure chemical vapor deposition (LPCVD) system. Gold (Au), Nickel (Ni), Platinum (Pt) and Palladium (Pd) are effective catalytic materials for growth of aforementioned hexaboride TE nanostructures. The as-synthesized nanostructures were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), and Raman spectroscopy. Possible mechanisms for the growth of these novel boron-based TE nanostructures will be presented. Combined with other properties of boron-based materials (e.g., low-density, superior mechanical properties, excellent thermal and chemical stability), these new TE nanostructures may find applications in the automotive industry and in high temperature micro- and nanoelectromechanical systems (MEMS and NEMS), electronics, and others.

**3:40pm NS-MoA6 Plasmon Excitations in Metallic Nanoparticles Arranged in Crystalline Structures, H.J. Steffen,** Steffen and Associates, Inc., Canada

The study of the character of collective electron density oscillations provides a fundamental understanding of the optoelectronic and photonic behavior of nanostructures. Consequently, electron-energy loss spectroscopy represents an important method for the structure analysis of materials with metallic nanoparticles since it contains valuable information about size, geometry and chemical composition of the particles and in addition, their spatial configuration, distance and crystalline order. Therefore, analytical model calculations and a parameter study were accomplished based on the hydrodynamic and density functional theory. Eigenfrequencies of plasmons were calculated and theoretical energy loss spectra were obtained by modelling the associated energy loss functions. Electron-energy losses appear at energies below 5 eV, far below the well-known dipole resonance energy of metallic spheres. The energy positions are dependent on the electron density, geometry, particle size, distance and crystalline structure of the particle arrangement.

**4:00pm NS-MoA7 Growth of Tungsten Nanoripples Induced by Linearly Polarized Femtosecond Laser, H. Zhang, M. Tang, J. McCoy, T.H. Her,** The University of North Carolina at Charlotte

Single-laser-beam induced nanoripples of tungsten is demonstrated in a laser-induced chemical vapor deposition (LCVD) system using tungsten hexacarbonyl as precursor and a linearly-polarized 400-nm femtosecond laser beam at 80MHz. The ripples have the shape of willow leaf with a long strip and two pointed ends, whose width is less than 100 nm and height on the order of 15 nm. The periodicity of the ripples is around 130 nm, which is about one third of the wavelength of the laser beam. The ripple orientation was found always parallel to the laser polarization, and hence can be controlled by changing the laser polarization direction. Different grating patterns were formed by scanning the laser beam along the substrate surface. Transverse pattern was formed when the scanning direction is perpendicular to the laser polarization, and longitudinal pattern was obtained when the scanning direction was parallel to the laser polarization. Effects of the laser power, exposure time, and scanning speed on the ripple formation were investigated. Different from the conventional laser-induced periodic surface structures (LIPSS)@footnote 1,2@ which can only occur by irradiating substrates near melting threshold, the tungsten nanoripples and gratings we report here can be heterogeneously deposited on a variety of substrates including insulators (glass, quartz, and sapphire

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etc.), wide band gap semiconductor (gallium nitride), and metals (gold, copper, and palladium etc.). Considering the simplicity of this process and material flexibility of CVD, our finding may provide a novel cost-effective patterning method to produce periodic subwavelength nanostructures of a wide range of materials. @FootnoteText@ @footnote 1@ Dieter Bäuerle, Laser processing and chemistry, 3rd. ed. (Springer, Berlin, 2000).@footnote 2@ J. E. Sipe, J. F. Young, J. S. Preston, and H. M. van Driel, Phys. Rev. B 27, 1141-1154 (1982).

4:20pm **NS-MoA8 Nanoscale Stepping Stones for Enhanced Charge Transport through Organic Materials**, *D.B. Robinson*, Sandia National Laboratories; *A.A. Talin*, Sandia National Laboratories, 94550; *R.J. Anderson*, Sandia National Laboratories

The charge transport properties of organic materials, and the mechanisms of this transport, depend strongly on the length scale being studied. At very short length scales, charge transport is sensitive to geometry, and experimental methods tend to be exotic and expensive, with low sample throughput. Among the most tractable device geometries that have been demonstrated is one consisting of an array or superlattice of particles spanning electrode pairs. We have developed a test platform for organic electronics based on this geometry that is especially simple, relying only on parallel, low-cost techniques including solution-based self assembly, and that is compatible with a range of characterization techniques such as cyclic voltammetry, surface-enhanced Raman spectroscopy, UV-visible reflectance spectroscopy, Auger electron spectroscopy, and electron microscopy. It is also compatible with architectures for sensing applications.@footnote 1@ For example, the in-plane conductivity through a thin film of a conducting polymer, poly (3-hexylthiophene), is enhanced by the presence of a regular array of metallic nanoparticles spaced on a length scale comparable to the persistence length of the polymer, perhaps because the particles provide resting points for charge carriers from which they can more easily proceed than they could from usual bulk trapping sites. Electrical properties are observed as a function of polymer molecular weight, particle type, and deposition method. @FootnoteText@ @footnote 1@ Wohltjen, H.; Snow, A.W. Anal. Chem. 70 947 (1998).

4:40pm **NS-MoA9 Partial Crystallization of HfO<sub>2</sub> and Its Application to Nanoscale Flash Memories**, *G. Zhang*, National University of Singapore; *W.J. Yoo*, Sungkyunkwan University, Korea, Singapore

The demands for non-volatile memories (NVM) in recent years have increased rapidly due to the growth of mobile device industries. Silicon-Oxide-Nitride-Oxide-Silicon (SONOS) type flash memory layer is extensively studied because of its potential as a high density memory. The high-k dielectric material HfO<sub>2</sub> is considered as a better trapping layer material than Si<sub>3</sub>N<sub>4</sub> in SONOS because high k can provide better vertical scalability and data retention property. Amorphous HfO<sub>2</sub> crystallizes during high-temperature (> 650°C) treatments, which is a drawback of HfO<sub>2</sub> since the crystallization can induce high leakage current. According to the crystallization result obtained by X-Ray Diffraction (XRD), high intensity peaks of (-1 1 1) and (1 1 1) are observed after the thermal treatment for 5 s in the temperature range of 800°C - 1000°C. However, conducting-AFM topography image clearly shows that the crystallization across the HfO<sub>2</sub> film is non-uniform and the grains in the film are formed via partial crystallization. A two dimensional current image of C-AFM confirmed the non-uniform distribution of the grains, where leakage current is significantly higher than amorphous HfO<sub>2</sub>. The z results (contrast in image) are 2nm for topography and 6 pA for current image. We consider that these grains act similarly to nanocrystal dots, which effectively suppress lateral charge migration under the programmed state, via charge localization inside the grains or at the grain boundaries. Excellent 2-bit/cell and 4-level/bit memory properties are achieved by Channel Hot Electron Injection (CHEI) program and outstanding charge retention property is achieved in both room temperature and at 85°C. The novel way of mimicking nanocrystal structure with partially crystallized continuous HfO<sub>2</sub> trapping layer easily enables multi-bit/cell operation and greatly extend the storage capacity of SONOS type flash memory.

## Nanometer-scale Science and Technology Room 2016 - Session NS1-TuM

### Nanoscale Structures and Characterization II

**Moderator:** S.V. Kalinin, Oak Ridge National Laboratory

8:00am **NS1-TuM1 Electronic Transport in Nanometer-Scale Silicon Membranes**, *P. Zhang, E. Tevaarwerk, B. Park, D.E. Savage*, University of Wisconsin-Madison; *G. Celler, Soitec USA; I. Knezevic, P. Evans, M.A. Eriksson, M.G. Lagally*, University of Wisconsin-Madison

Use of silicon-on-insulator (SOI), a thin single-crystal silicon layer on silicon dioxide, is already pervasive in microelectronics. Additionally, SOI provides a potential new paradigm for surface studies, as these thin membranes become almost "all surface". For example, novel electronic properties emerge with decreasing membrane thickness, as surfaces and interfaces dominate bulk properties. We have investigated the conductivity of very thin (10nm) Si membranes bounded by one or two Si-SiO<sub>2</sub> interfaces. Below a certain thickness, free carriers in the membrane will be completely trapped by the oxide/Si interface states. For a typical doping level of 10<sup>15</sup> cm<sup>-3</sup>, the depletion thickness is of the order of 100 nm; in other words, a thin Si membrane bounded by two oxide layers will act like intrinsic Si. We demonstrate both experimentally and theoretically that the conductivity is vanishingly small for this case. Therefore scanning tunneling microscopy (STM) from thin membranes should be impossible. On the contrary, we successfully image 10 nm thick Si, when the top native oxide is removed and a clean reconstructed Si (001) surface exposed. We show that electronic conduction in a thin Si membrane is determined not by its "bulk" dopants but by the thermal excitation at 300K of Si valence band charges to the surface band. Bulk dopant concentration is virtually irrelevant for electronic properties of Si nanomembranes. Conductivity in the membrane can be tailored by modifying the surface chemistry. We predict that either electrons or holes can be thermally generated, depending on the exact positions of the HOMO and LUMO bands, relative to the Si nanomembrane conduction and valence bands edges, of molecules adsorbed on the surface of Si. The addition of such layers may provide a practical approach to manufacture nanoscale sensors with high sensitivity and reliability based on electronic readout. We show that electronic conduction in a thin Si membrane is determined not by its "bulk" dopants but by the thermal excitation at 300K of Si valence band charges to the surface band. Bulk dopant concentration is virtually irrelevant for electronic properties of Si nanomembranes. Conductivity in the membrane can be tailored by modifying the surface chemistry. We predict that either electrons or holes can be thermally generated, depending on the exact positions of the HOMO and LUMO bands, relative to the Si nanomembrane conduction and valence bands edges, of molecules adsorbed on the surface of Si. The addition of such layers may provide a practical approach to manufacture nanoscale sensors with high sensitivity and reliability based on electronic readout. (Footnote 1) Pengpeng Zhang, et al. Nature 439, 703-706 (2006).

8:20am **NS1-TuM2 Nanoelectronic Device Characterization: Correlating Internal Nanoscale Chemical and Physical Structure with Electrical Behaviour**, *W.F. Stickle*, Hewlett Packard Company; *D.R. Stewart, J.J. Blackstock, C.L. Donley, R.S. Williams*, Hewlett Packard Labs

In emerging nanoscale electronic devices, the critical active layers within the device stacks have been reduced to only a few nanometers thick. Detailed understanding of the nanoscale physical and chemical structures of these layers and interfaces is essential for understanding and engineering the electronic behavior of such devices. Unfortunately, these critical layers are almost always buried underneath complex materials stacks, deposited in steps that often induce physical and chemical modifications. Reliable data on the nanoscale chemical and physical structure of the devices can only be obtained from challenging in-situ investigations. We present new techniques for accessing the internal chemical and physical structure of nanoscale layers and interfaces buried within complex device stacks. We present in-situ UHV spectroscopy and scanning-probe-microscopy data acquired using these techniques from critical nanoscale layers within experimental nanoelectronic devices being developed at HP Labs. These data are compared with data acquired using conventional techniques (such as depth-profiling and pre-fabrication characterization) to demonstrate additional information these new techniques can provide. Finally, we illustrate the importance of such detailed characterization by correlating understanding of the nanoscale physical and chemical structure of our experimental nanoelectronic devices to their electrical properties. Using information from these new techniques allows us to postulate a new model for the electrical switching behaviour of our devices, based on the electrochemical behaviour of metal-oxide species within the critical nanoscale layers of our devices.

8:40am **NS1-TuM3 Manipulating Liquids on the Tunable Nanostructured Surfaces**, *T.N. Krupenkin, A. Taylor, P.R. Kolodner, M.S. Hodes, J. Aizenberg*, Bell Labs, Lucent Technologies **INVITED**

An ability to manipulate microscopic volumes of liquids with the high precision becomes increasingly important with the recent progress in

micro- and nanofluidics and its rapid penetration in various industrial applications. Dynamic control over the interaction of liquids with the solid substrate constitutes a very important aspect of this problem. Nanostructured solid surfaces offer a promising way to achieve this goal. In this talk we discuss recently demonstrated dynamically tunable nanostructured surfaces. The behavior of these surfaces can be reversibly switched between superhydrophobic and hydrophilic states by the application of electrical voltage and current. The proposed approach potentially allows novel methods of manipulating microscopically small volumes of liquids. This includes almost frictionless liquid transport, the ability to precisely control droplet shape and position, as well as dynamic control over the penetration on liquids through the nanostructured layer. The obtained results potentially open new and exciting opportunities in chemical microreactors, bio/chemical lab-on-a-chip devices, optofluidics, thermal management of microelectronics, and many other areas.

9:20am **NS1-TuM5 Ideal Control of Carbon Nanotube Field Effect Transistor Characteristics depending on Precise Work Function Difference**, *K. Matsumoto*, Osaka University, Japan

We have succeeded in controlling the ideal characteristics of Carbon Nanotube Field Effect Transistor (CNT FET) which shows 1) the extremely high time stability of FET characteristics, 2) the completely no hysteresis characteristics, and 3) the precise control of the transfer characteristics of p-type and n-type only by changing the electrode metals with the different work functions. It is so far considered that the large time instability and hysteresis characteristics in CNT FET were the attribution of the water and oxygen those adhered the surface of the nanotube. Even if these are completely removed, however, CNT FET still shows the large time instability and hysteresis characteristics. We found out not only the water and oxygen but also the residue of the photo-resist which adhered during the fabrication process of CNT FET is the important origin of those problems. We have established the new fabrication process which could completely remove the residue of the photo resist, and the surface of the nanotube is protected by SiN film after heating the CNT at 700C to prevent the adhesion of the water and oxygen. The electrode metal of Ti with the work function of 4.33eV slightly smaller than that of nanotube of 4.8eV, was used. Though the drain current of CNT FET fabricated by the conventional process shows the large time instability of 20~30%, the drain current of CNT FET by new process shows the fluctuation of ~ 0.1% and improvement of the time fluctuation is more than 100times. The transfer characteristics of n-type CNTFET can be easily obtained only by selecting the electrode metal of Ti with the work functions smaller than the nanotube, when the nanotube is completely pure condition. Because of the pure condition of nanotube, the hysteresis Voltage of the present CNT FET shows almost 0V, which is far smaller value compare to the conventional CNTFET. Thus, we have succeeded in realizing the ideal control of the CNT FET characteristics.

9:40am **NS1-TuM6 Vertically Aligned Carbon Nanofibers for Energy Storage Applications**, *K.-Y. Tse, V. Dementiev, L.Z. Zhang, S.E. Baker, P. Warf, R. West, R.J. Hamers*, University of Wisconsin-Madison

Vertically-aligned carbon nanofibers (VACNFs) are interesting materials for applications such as energy storage because the stacking of graphene sheets within the individual nanofibers exposes a comparatively large number of edge planes, which are more chemically and electrochemically reactive than the basal planes. Thus, VACNFs have very different chemical and electrochemical properties than carbon nanotubes and other forms of nanostructured carbon. We have investigated the electrical properties of VACNF forests in a variety of electrolytes for applications as electrochemical double layer capacitors (also known as supercapacitors) and lithium ion batteries. By combining VACNFs with organosilicon electrolytes, it is possible to fabricate supercapacitors that exhibit high capacitance, good stability, and excellent frequency response. The vertical orientation of the nanofibers provides excellent ionic accessibility and low resistance, which translate to good high-frequency response when used in energy storage devices. We will also discuss effects of chemical modification with molecular monolayer, nanofiber aggregation, and further experiments to improve the performance of VACNFs electrodes.

10:40am **NS1-TuM9 Local Probing of Polarization Switching in Low-Dimensional Ferroelectrics**, *S. Jesse, B.J. Rodriguez, S.V. Kalinin*, Oak Ridge National Laboratory; *E.A. Eliseev, A.N. Morozovska*, National Academy of Science of Ukraine

Quantitative measurements of local switching characteristics and the relationship between switching behavior and defects on the nanoscale are crucial for the application of ferroelectric materials as non-volatile

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memories and ultra high density data storage. Switching Spectroscopy Piezoresponse Force Microscopy (SS-PFM) is developed as a quantitative tool for real-space mapping of switching properties of ferroelectrics, including imprint, coercive bias, remanent and saturation responses, and work of switching. In SS-PFM, multiple electromechanical hysteresis curves are collected at each point in an image to extract maps of parameters describing the switching behavior. The nucleation and growth of a single domain occurs under a sharp tip and hysteresis follows the development of a domain at a given location. The theory for hysteresis loop formation in the thermodynamic and kinetic limits is developed to relate SS-PFM data with local material properties and polarization dynamics. Several examples of switching behavior in low dimensional ferroelectrics are presented including (a) pinning at grain boundaries in polycrystalline PZT, (b) non-uniform work of switching in ferroelectric nanodots and (c) switching in the vicinity of topographic defects. In nanodots, the non-uniform work of switching was imaged with  $\sim 10$  nm resolution within 50 nm ferroelectric nanoparticles. "Abnormal" hysteresis loops were observed in the vicinity of topographic defects in ferroelectric thin films and PZT ceramics and attributed to the interaction of the nascent domain with the strain field of the defect. Future prospects for SS-PFM as a tool for probing ferroelectricity in low-dimensional systems and ferroelectric-based device characterization are discussed.

**11:00am NS1-TuM10 Quantitative Measurement of Tip-Sample Forces in Amplitude Modulation Atomic Force Microscopy, H. Holscher, University of Munster, Germany**

The amplitude modulation atomic force microscopy (AM-AFM) - also known as "tapping mode" - is the workhorse under the scanning probe microscopy techniques applied in ambient conditions and liquids. During the approach towards the sample surface the oscillation of a cantilever with integrated tip is detected by a change of the oscillation amplitude and phase. Unfortunately, the analysis of these signals is greatly complicated by the nonlinearity of the tip-sample force resulting in instabilities and hysteresis. Nonetheless, it has been shown how the energy dissipation between tip and sample is linked to the phase. In our talk we present an approach enabling also the quantitative reconstruction of tip-sample forces in amplitude modulation AFM. The proposed algorithm is based on the systematic recording of amplitude and phase vs. distance curves. The reliability of the method is demonstrated by numerical verifications. Experimental applications demonstrate its practicability.

**11:20am NS1-TuM11 Novel Standards for Dimensional and Analytical Nanometrology, T. Dziomba, L. Koenders, P. Hinze, T. Weimann, Physikalisch-Technische Bundesanstalt, Germany; M. Ritter, A. Kranzmann, M. Senoner, Bundesanstalt fuer Materialforschung und -pruefung (BAM), Germany**

Many measurement methods advance further into the sub-micrometer and increasingly into the nanometer range. Therefore, National Metrology Institutes (NMI) such as PTB and BAM face the challenge to develop suitable standards for characterization & traceable calibration of both spacial-analytical and purely dimensional high-resolution measurement techniques. For dimensional calibration e. g. of Scanning Probe Microscopes (SPM), we present a novel 3D standard with sub-microscopic features used as nanometric landmarks. In a certified calibration, all three coordinates of each of these nanomarkers are determined and given to the user as reference data together with the standard. The user measures the 3D standard with his instrument and compares his results with the reference data by simply applying a special software package delivered with the standard. Unlike step height and lateral standards, this new method allows a complete 3D calibration with just one type of standards. It is thereby fast and accurate. This novel 3D standard has proven to be well-suited also for other high-resolution microscopy techniques such as stereogrammetric SEM and Confocal Laser Scanning Microscopy (CLSM). Standards for the calibration of spacial-analytical instruments such as Secondary Ion Mass Spectrometers (SIMS) and Auger Electron Spectroscopy (AES) should show a clear material contrast while being topography-free. Two different concepts have been realized at PTB and BAM with success: Firstly, a system of alternating layers of well-defined thickness in the range from a few nm to several hundred nm has been deposited on a wafer, which is later cleaved so that a 1D pattern becomes accessible. Secondly, 2D analytical standards have been fabricated by placing patterns of well-defined geometry and size consisting of metal A (e. g. Au) in an environment of metal B (e. g. Ag) in a process involving several lithography steps in order to ensure that the sample is largely topography-free.

**11:40am NS1-TuM12 Electronic Structure Studies of CdSe Nanocrystals Using Synchrotron Radiation, R.W. Meulenberg, J.R.I. Lee, L.J. Terminello, T. van Buuren, Lawrence Livermore National Laboratory**

We have examined band edge shifts in colloidal CdSe nanocrystals using synchrotron radiation. In particular, Cd s states located at the conduction band minimum (CBM) show drastic size dependencies while the Cd d levels located a few electron volts up from the CBM show more localized behavior. This was not expected based on effective mass theories, although more sophisticated calculations are shown to agree better with experiment. Changing the surface ligand has very little effect on the shift of the band edges, but does affect the local bonding significantly. In addition, we find that the electronic structure can further be tuned by the incorporation of dopant atoms. Our experimental findings will be compared with recent theory to help resolve the nature of quantum confinement in CdSe nanocrystals. This work was supported by the Division of Materials Sciences, Office of Basic Energy Science, and performed under the auspices of the U.S. DOE by LLNL under contract No. W-7405-ENG-48.

**12:00pm NS1-TuM13 Reliable XPS Measurement of Sub nm SiO<sub>2</sub> Thickness by Determination of the Surface Normal, K.J. Kim, Korea Research Institute of Standards and Science, Republic of Korea; D.W. Moon, Korea Research Institute of Standards and Science, Korea; J.S. Jang, H.J. Kang, Chungbuk National University, Korea**

Recent international technology roadmap for semiconductor showed that the reliable measurement of ultra-thin gate oxide thickness below 1 nm is required for the next generation of semiconductor devices. X-ray photoelectron spectroscopy (XPS) is an ideal candidate. Recently, SiO<sub>2</sub> film thickness measured by various techniques was compared in the first pilot study in the surface analysis working group of CCQM. However, further studies are required to improve the traceability of the XPS measurement of SiO<sub>2</sub> thickness in the sub nm range. The diffraction effect of photoelectrons due to the crystallinity of the substrates has been regarded as one of the major issues in angle-resolved (AR) XPS analysis. The diffraction effect could be completely removed by using amorphous SiO<sub>2</sub> overlayers grown on an amorphous Si layer by ion beam sputter deposition. The film thickness could be measured without surface contamination by in-situ AR-XPS analysis. The thickness of the films was estimated by the growth rate determined from the high resolution-TEM. Without the diffraction effect and the surface contamination, the calibration curves of XPS measurement passed the origin. Additionally, the polar angle control is one of the critical factors for the accurate thickness measurement of SiO<sub>2</sub> films by AR-XPS. For the accurate control of the polar angle, the surface normal must be calibrated precisely. The accurate surface normal can be simply determined by minimizing the standard deviation of the thickness values estimated at different polar angles around the surface normal. With the new procedure, the surface normal could be determined accurately within 0.05°. This study demonstrates that AR-XPS can provide reliable measurement of SiO<sub>2</sub> thickness down to sub nm range by accurate calibration of the surface normal. @FootnoteText@ @footnote 1@K. J. Kim, K. T. Park and J. W. Lee, Thin Solid Films 500, 356-359 (2006).

## Nanometer-scale Science and Technology

### Room 2020 - Session NS2-TuM

#### Nanoscale Manipulation and Assembly

Moderator: J. Randall, Zyvex Corporation

**8:00am NS2-TuM1 Deterministic Growth of Individual Ge Nanowires within Lithographically Patterned Arrays, C.E.D. Chidsey, Stanford University**

INVITED

Germanium nanowires and microwires are grown epitaxially from lithographically defined regions on silicon substrates in preferred crystallographic directions from electrodeposited gold nanoparticles and gold alloy nanoparticles. Nucleation, growth, surface termination and gold removal are discussed.

**8:40am NS2-TuM3 Investigating the Motion of Molecular Machines on Surfaces: Driving Nanocars, K.F. Kelly, A.J. Osgood, J. Zhang, Y. Shirai, T. Sasaki, J.-F. Morin, J.M. Tour, Rice University**

We have investigated by STM the controlled molecular motion of individual molecules on surfaces through the rational design of fullerene- and carborane-based nanostructures. The NanoCar molecule (four fullerene wheels connected by rotating alkyne axles to a central chassis) and the related molecular structures are designed and fabricated specifically for



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nanoscale manipulation. Our work has consisted of characterizing by variable-temperature microscopy and manipulating these molecules using the STM tip to probe the effects of surface-molecule interactions on the direction and speed of motion. This family of molecules is all built from a similar set of "molecular tinker toys" that includes interchangeable components for "chassis," "axles," and "wheels." The results of these studies underscore the ability to control directional motion in molecular-sized nanostructures through precise molecular synthesis with possible applications to surface catalysis and nanoscale tribology.

## 9:00am NS2-TuM4 Toward Stable Molecular Devices: Desorption of Cyclopentene from p-Si(100) with UHV-STM and Density Functional Theory, N.L. Yoder, N.P. Guisinger, M.C. Hersam, Northwestern University

In recent years, substantial progress has been made in the emerging field of molecular electronics. Of particular interest is the integration of molecular electronic devices with conventional silicon microelectronic technology. The presence of the energy band gap in silicon allows for resonant tunneling through individual molecules, leading to interesting effects such as room temperature negative differential resistance.<sup>1</sup> Additionally, studies of organic molecule-silicon junctions can yield important insights into the feasibility of future hybrid molecule-silicon devices. In particular, the reliability of these molecular junctions is of critical importance to potential devices, and consequently warrants further investigation. Single cyclopentene molecules on silicon provide a useful test case, since the binding geometry has been studied both experimentally and theoretically. In this study, a combination of experimental and theoretical tools were employed to investigate the stability of cyclopentene molecules on a degenerately doped p-type Si(100) surface. Experiments were performed using a cryogenic ultra-high vacuum scanning tunneling microscope. At 80 K, cyclopentene desorbs from the surface at both positive and negative sample bias polarities over a range of tunneling currents. The desorption rate is roughly linear with tunneling current, indicating a single-electron/hole process. The desorption yield is a strong function of bias, and has turn-on voltage of -2.5 V at negative bias and 3.5 V at positive bias. The magnitude of the yield ranges from  $2 \times 10^{-12}$  to  $1 \times 10^{-8}$  events/electron at both polarities, which is 500-1000x smaller than the reported yields of Benzene on Si(100) and Chlorobenzene on Si(111). Density functional theory and reaction path calculations were performed in order to determine the details of the desorption process. <sup>1</sup>FootnoteText@ <sup>1</sup>footnote 1@N.P. Guisinger et al, Nano Letters 4, 55 (2004) and Proc. Nat. Acad. Sci. 102, 8838 (2005).

## 9:20am NS2-TuM5 Site-Specific Force spectroscopy and Following Artificial Nanostructuring, S. Morita, Y. Sugimoto, O. Custance, M. Abe, Osaka University, Japan

INVITED

We are developing a novel versatile "Bottom Up Nanotechnology", which can construct artificial nanostructures consisted of many kinds of elements by using a high-resolution and high-performance AFM that can identify chemical species of individual atoms and can manipulate individual atoms even at room temperature (RT). Noncontact AFM image can give different contrast between intermixed two chemical species. However, we cannot identify chemical species of intermixed atoms from atom selective imaging on only one sample without changing relative ratio of intermixed atom species. To achieve more straightforward chemical identification than atom selective imaging at RT, we developed precise atomic force measurement method of individual atoms as a function of tip-sample distance at RT by compensating thermal drift using an atom tracking method and then by averaging reproducibly obtained many site-specific frequency shift curves. Next we converted site-specific frequency shift curve to site-specific force curve. Then, by subtracting long-range force due to Van der Waals force, we obtained short-range atomic force curve of selected individual atoms at RT. Thus we achieved precise force spectroscopy of selected individual atoms at RT. In addition, AFMs under non- and near-contact modes operated at low-temperature (LT) and at room-temperature (RT) were used for investigations of vertical and also lateral atom manipulations of selected single atoms on semiconductor surfaces. Recently, using nearcontact AFM, we found a lateral atom-interchange phenomenon at RT and constructed "Atom Inlay", that is, atom letters "Sn" consisted of 19 Sn atoms embedded in Ge(111)-c(2x8) substrate. Furthermore, we observed atom interchange manipulation phenomena even in Sn/Si(111)-(<sup>3</sup>sr@3x@sr@3), In/Si(111)-(<sup>3</sup>sr@3x@sr@3) and Sb/Si(111)-(7x7) intermixed systems with not only Si but also In (acceptor element in crystal Si) or Sb (donor element in crystal Si) atoms.

## 10:40am NS2-TuM9 Tuning the Self-Assembly of Metal Nanoclusters on Diblock Copolymer Templates, S.B. Darling, A. Hoffmann, Argonne National Laboratory

Polymers and metals are increasingly becoming intertwined in technological systems. In particular, hybrid materials that take advantage of the ability of soft matter to self-assemble and the ability of hard matter to incorporate magnetic, electronic, catalytic, or photonic functionality are of great interest. In this study, we have investigated how spatially selective electron irradiation modifies the diffusion behavior of silver evaporated onto poly(styrene-block-methylmethacrylate) (PS-b-PMMA) films. This diblock copolymer self-assembles into cylinders of PMMA in a PS matrix, where the film thickness has been chosen such that both materials are present at the free interface in an alternating stripe structure. On unexposed films, the Ag selectively adsorbs to the PS domains due to different diffusion mobilities on the two polymeric materials. Exposure to an electron beam affects the polymer blocks in different ways and thereby provides a means to manipulate this motion. Furthermore, this methodology enables a connection between the nanoscopic length scale of diblock copolymer self-assembly with conventional top-down mesoscopic length scales. We performed Kinetic Monte Carlo simulations in order to provide some insight into the diffusion behavior and, hence, the relative significance of polymer-metal and metal-metal interactions.

## 11:00am NS2-TuM10 Selecting and Driving Nanoscale Assembly in Monolayer Films through Tailored Intermolecular Interactions, T.J. Mullen, P.S. Weiss, The Pennsylvania State University

We have developed, applied, and characterized molecules with varied intermolecular interaction strengths that control stability and impart directionality into patterned monolayers. In addition to employing molecules with tailored interactions, we have exploited self- and directed assembly techniques to fabricate, to register, and to functionalize chemical surface structures at the supramolecular 1-100 nm scale. We will highlight specific examples, including applications in molecular electronics and chemical patterning, where specifically tuned molecules were designed, custom self-assembly processing techniques were utilized, and the resulting chemical surface structures were characterized on both the ensemble and the molecular scales.

## 11:20am NS2-TuM11 Exploring and Manipulating Fullerenes at the Single Molecule Level, M.F. Crommie, University of California, Berkeley, U.S.

INVITED

Fullerene molecules provide powerful building blocks for creating nanostructures with unique electronic properties. The flexible nature of these molecules arises from a combination of molecular orbital geometry, Coulomb interactions, and electron-phonon coupling. We have used scanning tunneling microscopy to examine and manipulate these factors in order to explore fullerene molecules in different physical regimes. We find that it is possible to reversibly change the charge and magnetic state of individual C<sub>60</sub> molecules through single-atom doping, and we have spatially mapped electron-phonon interaction strengths over the surface of single fullerene molecules. When fullerenes are packed into 2-dimensional surface assemblies we find that molecular electron-electron and electron-phonon interactions combine to create novel long-range order.

## 12:00pm NS2-TuM13 Electrochemical STM Investigations of Redox-Active Tetrathiafulvalene Islands Embedded in Alkanethiol SAMs, Y. Yokota, A. Miyazaki, K. Fukui, T. Enoki, M. Hara, Tokyo Institute of Technology, Japan

Stochastic apparent height changes in scanning tunneling microscopy (STM) images of self-assembled monolayers (SAMs) have been reported by some groups.<sup>1,2,3</sup> From the viewpoint of molecular electronics, one of the goals of our studies is to control the structure and electronic properties of molecular assemblies by using electric charging effects. We report the electrochemical STM (EC-STM) studies of redox-active tetrathiafulvalene (TTF) islands embedded in n-decanethiol (C<sub>10</sub>SH) SAMs on Au(111).<sup>4</sup> We performed EC-STM measurements of TTF islands of varied sizes at various potentials in 0.05 M HClO<sub>4</sub> solution. While the larger island always protruded from the C<sub>10</sub>SH matrix independent of potential, the smaller island (less than ten molecules) changed their apparent height from flat surface to clear protrusion. This change was reversible against potential change. From the cyclic voltammetry of TTF SAMs, the electronic states of TTF moieties in each STM images were characterized. The behavior of the apparent height change of the smaller island can be explained by the orientational change induced by charging of TTF moieties. Meanwhile, the behavior of the potential-inactive one is associated with a stacking effect of TTF moieties in the large island. These results indicate that not only the oxidation state but

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also the orientation of the electroactive moiety responds to electrochemical potential and that orientational change is also a key factor in designing molecular devices under potential control. Furthermore, we report that the larger the size of the large islands, the higher the apparent height in the STM images, although the physical height of the islands can be assumed to be same. We think that the effective electron transfer between TTF moieties leads to increase of electron-transfer passes, and to the higher apparent height. @FootnoteText@ @footnote 1@ Z. J. Donhauser et al., Science 292, 2303 (2001). @footnote 2@ G. K. Ramachandran et al., Science 300, 1413 (2003).@footnote 3@ R. A. Wassel et al., Nano Lett. 3, 1617 (2003).@footnote 4@ Y. Yokota et al., J. Phys. Chem. B 109, 23779 (2005).

## Plasma Science and Technology

### Room 2009 - Session PS1+MS+NM-TuM

#### Plasma Patterning

**Moderator:** A. Agarwal, University of Illinois at Urbana-Champaign

8:00am **PS1+MS+NM-TuM1 Resolving Gate Patterning Issues at sub 65 nm Technology Nodes**, *T.J. Kropewnicki, C.-C. Fu*, Freescale Semiconductor, Inc.

According to the 2005 edition of the International Technology Roadmap for Semiconductors, the physical gate length of high performance transistors at the 65 nm technology node is expected to be 25 nm in 2007, decreasing to 18 nm at the 45 nm node in 2010. Clearly, these goals present a clear challenge to photolithography and etch, which together are responsible for resolving these features on wafers. In addition to the pure scaling aspects of technology progression are the many additional enhancements such as stressors, which are being used to push the performance of silicon circuits. In certain integration schemes, these stressors add complexity to the transistor gate stack and accelerate photoresist bending and line collapse which cause etch masking, and ultimately variable, uncontrollable line widths. This paper will begin with a brief description of the transistor module process flow, highlighting the new challenges introduced to the gate stack etch at sub 65nm technology nodes. Next, a combination of enhancements in the gate photolithography and etch steps used to address these challenges will be presented. Results from these experiments will show a 50% reduction in across wafer line width variation, and a near 100% reduction in the incidence of polysilicon pattern distortion. Finally, the possible mechanisms for the increased levels of polysilicon pattern distortion seen with advanced transistor modules will be discussed.

8:20am **PS1+MS+NM-TuM2 Plasma Impact on ArF Resist Line Edge Roughness**, *J. Thiault*, LTM / CNRS France, France; *E. Pargon*, LTM / CNRS France; *J. Foucher*, CEA LETI France; *O. Joubert*, *G. Cunge*, LTM / CNRS France, France

As Critical Dimensions for semiconductor devices shrink too few tens of nanometers, the Line Edge Roughness (LER) or Line Width Roughness (LWR) becomes a critical issue because it can degrade resolution and linewidth accuracy that causes fluctuations of transistors performances. ArF resist patterns present a LWR of about 8 nm after lithography that is possibly transferred into the underlayers during plasma processing steps, resulting in a final LWR above the requirements of the International Technology Roadmap for Semiconductors (ITRS, which tolerates a LWR of around 3 nm for the 65nm technology node). In this study, isolated ArF resist patterns have been exposed to different plasma chemistries under identical processing conditions to investigate the impact of the plasma chemistry on the resist LWR. The sidewall roughness characterization has been performed using 3D Atomic Force Microscope (AFM3D) and top view Scanning Electron Microscope (CD-SEM). Experimental results tend to show that when the plasma/resist interaction is strongly chemically driven, such as in O@sub 2@ or SF@sub 6@ plasmas, with no bias applied to the wafer, the resist sidewalls are not smoothen. However, using plasma conditions where the ion bombardment component of the plasma is increased (by applying a bias power to the wafer), a LWR reduction is measured. This trend has been confirmed by exposing resist patterns to chemically inert plasmas such as Ar plasmas. Moreover, we have investigated plasma curing treatment on resist patterns, currently used in semiconductor manufacturing to reinforce the etching resistance of the resist. In this type of plasma (HBr based) where the ion current density is high and ion energy low, we also observe a decrease in LWR. All these trends suggest that the anisotropic ion flux is responsible for the smoothing of the resist sidewall roughness by eroding the bumps present on the resist sidewall.

8:40am **PS1+MS+NM-TuM3 ArF Resist Friendly Etching Technology**, *T. Hayashi, Y. Morikawa, K. Suu*, ULVAC Inc., Japan **INVITED**

The requirements for dry etching technology in semiconductor processes beyond 90 nm node come to be very complicated and difficult. In photolithography, the introduction of ArF resist has started. ArF resists generally have very weak plasma resistance and are deformed in the etching process. However, as for the fundamental mechanism of deformation of resists in dry etching, sufficient discussions have not been done yet. So far ArF resist deformation has been thought to be caused by ion impinging damage. However, in our experiments, the deformation was not found in the high density NLD plasma in lower pressure than 1Pa, which gives relatively higher ion flux to the surface. So if ion energy is the main origin of the ArF resist deformation, then the NLD plasma might give a large resist deformation. Contrary to this expectation, however, the etched profiles in the NLD plasma showed low line edge roughness and almost nothing of striation. Considering these facts, the ArF resist deformation is clearly caused by concerted working of ion impinging damage and subsequent radical reactions at the resist-damaged area. This means if either of ion damage or radical reaction is nothing then the ArF resist deformation is nothing or is suppressed considerably lower. Therefore, the lower pressure process below 1 Pa or the lower reactive radical density process is necessitated. The latter is achieved by using highly effective radical scavenger. Generally H or CO has been used as the scavenger of F atoms. However, Br and I may be more effective as the scavenger, because Br and I react with F atoms and form stable inter-halogen compounds. So use of iodine or bromine contained perfluoro-hydrocarbon compounds like CF3I gives the ArF resist friendly etching process. Actually very smooth etched surfaces were obtained for patterned ArF resist/ARC/CAP/low-k/BARC/Si wafers. This work was partly supported by NEDO (New Energy and Industrial Technology Development Organization) in Japan.

9:20am **PS1+MS+NM-TuM5 Plasma Etching of Nano-Scale, Sub-10nm, Features**, *Y. Zhang, C.T. Black, H.-C. Kim, E.M. Sikorski, T. Dalton*, IBM Research

Features Patterning nano-scale semiconductor features with precision imposes many new challenges for plasma etching. One of the challenges is that as the sizes of nano-scale features shrinking down to the sub-10nm regime, Plasma etching seems to approach to the limits. In this paper, we report the recent results of studying plasma etching of true nano-scale features using two kinds of nano-scale patterns. The first type of samples is diblock copolymer (similar to resist) self assembled nano holes and lines. The second kind samples are self-assembled organosilicate (similar to silicon oxide) nano patterns. With samples pattern, arrays of nano holes or nano lines' dimensions in the range of ~10nm, we studied plasma etching challenges for transferring nano-scale patterns into different materials (silicon, and silicon dioxide) in different plasma chemistries and process conditions. By varying the thickness of masks, the characteristics of aspect ratio dependence vs. "real" etching limits due to the sizes of sub-10nm nano-scale features were studied. The impacts of mask selectivity and line edge roughness (LER) to transferring sub-10nm patterns will be also discussed.

9:40am **PS1+MS+NM-TuM6 Nickel Atom and Ion Density in an Inductively Coupled Plasma with an Internal Coil**, *L. Xu<sup>1</sup>*, University of Houston; *N. Sadeghi*, University Joseph Fourier-Grenoble & CNRS, France; *M.K. Jain, S.C. Vemula, V.M. Donnelly, D.J. Economou, P. Ruchhoeft*, University of Houston  
Nanopantography uses monoenergetic ion beams to enable massively parallel patterning of nano-sized features (e.g. 10 nm dia., 100 nm deep holes etched into Si). Deposition of metal nanodots (e.g. Ni) can have applications such as catalysts for the growth of an orderly array of carbon nanotubes. For this purpose, we have developed an inductive plasma source containing a relatively large fraction of Ni@super +@. A two-turn Ni coil immersed in the plasma generates a Ni-containing Ar plasma. Ni was sputtered both from the negatively self-biased coil and from a Ni target powered by a separate rf power. By adding a trace amount of N@sub 2@, gas temperatures T@sub g@ (= rotational temperatures) were derived from N@sub 2@(C-B) spectra. At low powers (capacitively coupled), T@sub g@ derived from the 0-0 band was erroneously high. This was attributed to energy transfer from Ar metastable atoms to the N@sub 2@ C (@upsilon@=0). At high powers (inductively coupled), both the N@sub 2@ 0-0 and 4-4 bands provided the same reasonable T@sub g@ because electron-impact dominates excitation at high power. Optical emission of Ar at 419.8 nm was used to estimate the plasma density, and was in agreement with values predicted from a global model. Ni densities were

<sup>1</sup> PSTD Coburn-Winters Student Award Finalist

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determined by optical absorption (using a Ni hollow cathode lamp) and were found to increase with pressure and power. The Ni@super +@ densities also increase at higher pressures and powers. Model predictions of Ni@super +@ densities are consistent with metastable Ni@super +@ densities derived from optical absorption. Finally, 50nm dia. Ni islands have been deposited in preliminary nanopantography experiments with the Ni@super +@ beam.

10:40am **PS1+MS+NM-TuM9 Bias Frequency Effect on SOC Film Degradation in sub-45 nm Line and Space Pattern SiO@sub 2@ RIE using S-MAP**, H. Hayashi, K. Kikutani, J. Abe, A. Kojima, T. Oohashi, I. Sakai, T. Ohiwa, Toshiba Corporation, Japan

Sub-45 nm line and space pattern etching of SiO@sub 2@ film was studied using a stacked mask process (S-MAP) which consists of photoresist, spin-on-glass (SOG) and spun-on-carbon (SOC) film stacked structure. Maintaining pattern integrity becomes more challenging with the decrease of pattern size. Reduction of the hydrogen content of SOC, suppressed the fluorination reaction of its C-H bonds during SiO@sub 2@ etching which lead to line pattern wiggling, and as a result, 56 nm line and space pattern etching was realized. This time, the effect of ion energy distribution on SOC degradation in the SiO@sub 2@ etch process was investigated for sub-45 nm line and space pattern etching. The ion energy distribution was varied by dual frequency superimposed (DFS) RIE, using the conditions of 100 MHz rf supply with 3.2 MHz superimposed compared with 100 MHz with 13.56 MHz superimposed. The other SiO@sub 2@ etch conditions were the same, that is, C@sub 4@F@sub 8@ gas chemistry with the same electron density and self-bias voltage (-Vdc) of 6x10@super 10@ cm@super -3@ and 350 V, respectively. As a result, SOC line pattern wiggling was observed in the 3.2 MHz case, but it was suppressed in the 13.56 MHz case, even though the SiO@sub 2@ etch rates were 241 nm/min and 254 nm/min, respectively, and about the same. This shows that, by using DFS RIE with 13.56 MHz superimposed, SOC degradation can be suppressed while maintaining the SiO@sub 2@ etch rate for sub-45 nm line and space pattern etching. The maximum ion energy in the 13.56 MHz case should be lower than that of the 3.2 MHz case under the same -Vdc conditions, because with higher rf frequency, it would have a narrower ion energy distribution. In this way, SOC degradation was suppressed without decrease of the SiO@sub 2@ etch rate. In conclusion, S-MAP combined with 100 MHz/13.56 MHz DFS RIE realized sub-45 nm line and space pattern SiO@sub 2@ etching. @FootnoteText@ @footnote 1@ J. Abe et al., Symp. Dry Process, (2005) 11.

11:00am **PS1+MS+NM-TuM10 High Aspect Ratio (>10:1) Amorphous Carbon Layer Etching Using Soft Etch Capability in a High Frequency Capacitive Coupled Plasma Source Dielectric Etch Chamber**, S. Sung, J. Wang, S. Ma, Applied Materials

Amorphous carbon layer (ACL) such as advanced patterning film (APF) is generally selected as one possible hard mask material for variety of dielectric etching application beyond 65 nm technology nodes to improve the etch process margin from reduction of resist thickness. Most of the APF etching application for DRAM, flash and logic technology are done typically on 1 μm film thickness to enable specific integration scheme of nanotechnology. The challenges of etching high aspect ratio ACL features are bowing prevention during etching, hard mask selectivity and the etch rate improvement for throughput concern. In this paper, high aspect ratio (HAR) contact through ACL layer is developed by soft etching capability using high frequency source plasma etch chamber. This development work was done in the dielectric etch chamber consisting of the superimposed dual bias power and a capacitive coupled source power > 100 MHz, which can be operated in either low density process regime for higher resist selectivity, or in high density process regime for profile control, resist integrity, minimal striations and effective chamber cleaning. With high frequency source power, fast etch rate >6000 Å/min of ACL has achieved with minimum hard mask corner chopping from small plasma self bias. All the process trends are characterized with profile control, hard mask selectivity and etch rate.

11:20am **PS1+MS+NM-TuM11 Chamber and Process Development of High Aspect Ratio Deep Trench Si Etch for DRAM Application below 60 nm**, S. Barth, A. Henke, Qimonda, Dresden, Germany; A. Kersch, Qimonda, Munich, Germany; M. Reinicke, University of Technology, Germany; W. Sabisch, Qimonda, Munich, Germany; J. Sobe, A. Steinbach, S. Wege, Qimonda, Dresden, Germany

For Qimonda's DRAM Technology the deep trench etched into silicon is the base for the capacitor concept. The shrink of lateral dimensions at approximately constant capacity specifications leads to increased deep

trench aspect ratio requirements. Therefore high selectivity to the etch mask and excellent uniformity is needed, especially for technologies below 60nm. In this paper we describe the development of a new DT plasma etch chamber and process to fulfill these requirements. Simulations (an equivalence circuit plasma model and surface reaction models) were combined with in-situ plasma measurement techniques (QMS, high resolution OES, IR absorption spectroscopy, SEERS and Langmuir probe sensor wafers) and technological experiments, to characterize hardware features and process conditions. To achieve high Si etch rate and selectivity, plasma density and electron energy distribution in the plasma bulk, and ion energy distribution on the wafer surface can be optimized through multi frequency cathode excitation. The selectivity is further enhanced by using advanced hard mask materials and combining of etching and deposition process regimes. Excellent uniformity has been achieved by new tool components, e.g., multi zone gas distribution and wafer temperature control. In addition, the etch process chamber includes new features for process control, in-situ wafer surface temperature and trench dept measurement. The equipment and process development was accomplished through close cooperation between Qimonda and the tool supplier.

## Nanometer-scale Science and Technology Room 2016 - Session NS-TuA

### Nanoscale Devices and Detection

Moderator: S. Wind, Columbia University

#### 2:00pm NS-TuA1 Magnetization Reversal by Spin Injection: Ultra-fast and Ultra-small, *Y. Acremann*, Stanford University **INVITED**

Invited Speaker Dan Ralph's talk replaced with Yves Acremann, Stanford University.

#### 2:40pm NS-TuA3 Single-Crystal/Amorphous Si-SiO<sub>2</sub> Multilayer Systems and Devices based on Si Nanomembranes, *W. Peng, M.M. Roberts, F.S. Flack, E.P. Nordberg, D.E. Savage, M.G. Lagally, M.A. Eriksson*, University of Wisconsin-Madison

Silicon-on-insulator (SOI) is widely used in device applications that require single-crystal Si but benefit from the isolation provided by a SiO<sub>2</sub> substrate layer. Multiple layers of single-crystal Si interspersed by (amorphous) SiO<sub>2</sub> are a natural next step for SOI devices. It is impossible to achieve such a system by growth techniques (one cannot grow single-crystal Si on SiO<sub>2</sub>), and multiple bonding steps, such as those used to create conventional SOI, would be prohibitively expensive. We demonstrate a novel method to fabricate such a multilayer Si-SiO<sub>2</sub> system using transferred silicon nanomembranes@footnote 1@ and subsequent oxidation. The surface roughness and interface quality for the multilayer system is similar to prime SOI. We describe devices, including Bragg mirrors and resonant tunneling diodes that take advantage of the properties of repeated layers of single-crystal Si and amorphous SiO<sub>2</sub>. Extremely high reflectivity (~98%) is observed for Bragg reflectors with as few as four Si layers, as expected from the large dielectric contrast between the silicon and silicon-dioxide layers. @FootnoteText@ Supported by NSF, DOE, AFOSR.@footnote 1@ Roberts, M. M. et. al. Elastically relaxed free-standing strained-silicon nanomembranes. *Nature Materials* 5, 388-393 (2006).

#### 3:00pm NS-TuA4 Characterization of Ga-Acceptor Nanoscale Wires in Si, *S.J. Robinson, J.R. Tucker*, University of Illinois at Urbana-Champaign; *T. Schenkel*, Lawrence Berkeley National Laboratory; *T.-C. Shen*, Utah State University

The prospect of using focused ion beams (FIBs) as maskless implants has gained popularity since the inception of the FIB. In particular, nanoscale interconnects and quantum devices could be defined by implantation of dopant atoms into semiconductors. We have used a FEI dual beam Strada 235 FIB at LBNL to fabricate high-density Ga wires in Si substrates to study the transport properties and practicality of these wires. Under similar annealing conditions, we find that these wires have considerably higher sheet resistance than conventional Ga implants and often show non-linear I-Vs. Concurrently, our analysis of the transport properties of these wires shows variable range hopping (in conjunction with the Efros-Shklovskii Coulomb gap) to be the most likely conduction mechanism at low temperatures. Our focus is determining whether high-conductivity, ohmic wires can be realized at liquid He temperatures by varying implant densities and annealing conditions. In addition to transport measurements, we also obtained AFM images to investigate surface morphologies after implantation and annealing. This work is supported by NSF-NIRT and the Molecular Foundry at LBNL.

#### 3:20pm NS-TuA5 Towards Nanowire-Based Thermocouple Arrays, *M.E. Bourg, R.M. Penner*, University of California, Irvine

The ability to accurately measure temperature on the same time scale as a thermal process under investigation is important in many industrial processes. A thermocouple, which consists of a junction between two dissimilar metals, is most often the device used for such measurements. As the thermal mass of the junction decreases, the response time decreases and the spatial resolution increases. Therefore, junctions containing nanowires should lead to faster response times and increased spatial resolution. In order to initially investigate this, we prepared nanowire-thin film (NWTF) junctions consisting of electrodeposited nanowires and an evaporated film. These devices measure temperature accurately and reproducibly, and have a response time of 2ms. For comparison, the response time of a 0.81mm type J thermocouple was 12s. To improve upon the enhancements shown by NWTF devices, nanowire-nanowire junctions were also fabricated. The characterization of these thermocouple arrays by SEM, EDS, and thermal measurements will be presented.

#### 3:40pm NS-TuA6 Electrical Properties of Graphene Ribbon Transistors, *Z. Chen*, IBM T.J. Watson Research Center; *X. Du*, Rutgers the State University of NJ; *M.J. Rooks, Ph. Avouris*, IBM T.J. Watson Research Center

Carbon nanotube has caught much attention due to its exceptional physical properties and potential for future nano-electronic applications. Graphene, a single layer of graphite, has similar chemical composition and crystal structures as the carbon nanotube, while at the same time shows semi-metallic behavior. It is proposed that, with intentional size and edge control graphene ribbons can be fabricated and viewed as an unrolled single wall carbon nanotube. The important question however is, whether the right boundary conditions can be introduced to generate quantization and result in semiconducting band gaps. Here we present a study on the electrical properties of the graphene ribbon transistors with various widths. Edge modification and measurements under different conditions will be discussed as well.

#### 4:00pm NS-TuA7 Superconducting Proximity Effect in Graphene, *X. Du, E.Y. Andrei*, Rutgers University; *A.F. Hebard*, University of Florida

Graphene, a single atomic layer of graphite, has drawn great interest recently both in its unique physical properties and potential for electronics applications. Graphene is an ideal 2D semimetal, with linear dispersion (Dirac Fermion). When in contact with superconductor, many interesting questions arise, in terms of 2D superconductivity and Andreev reflection at the superconductor/relativistic Dirac Fermion interface. Here we present experimental study on gate controlled superconductor/graphene devices. Electric field dependent superconducting proximity effect and Andreev reflection will be discussed.

#### 4:20pm NS-TuA8 Catalytic Nanodiode; Chemical Sensing of Gas Phase Catalytic Reaction by using Hot Electron Flows at Metal-Oxide Interface, *J.Y. Park*, University of California, Lawrence Berkeley National Laboratory; *J.R. Renzas, A.M. Contreras*, University of California, Berkeley; *G.A. Somorjai*, University of California, Lawrence Berkeley National Laboratory

Atomic or molecular processes in metals can generate a pulse of hot electrons with kinetic energy of 1-3 eV, and mean free path of the range of ~10 nm. The flow of these hot electrons are directly measured during the platinum catalyzed oxidation of carbon monoxide across a Pt-TiO<sub>2</sub>@sub 2@ and Pt-GaN Schottky nanodiode that were constructed from Pt film, oxide layers, and Ohmic contact pads. The thickness of Pt film used as the catalyst was 5 nm, less than the electron mean free path, resulting in the ballistic transport of hot electrons through the metal. By heating the nanodiodes in He, we could measure the thermoelectric current that is in the opposite direction to the flow of the hot electron current. We found that the chemi-current was well correlated with the turnover rate of CO oxidation separately measured by gas chromatography, suggesting the possibility of application as chemical sensors with high sensitivity. Chemi-current measured through the metal-oxide interface remains stable for over several hours and is reversible upon temperature change. The influence of the flow of hot charge carriers on the chemistry at the oxide-metal interface and the turnover rate in the chemical reaction will be discussed.

#### 4:40pm NS-TuA9 Hydrogen Detection NEMS Devices Fabricated from Tunable Microstructure Pd-Ta-X Nanocomposites, *C. Gilkison, C. Ophus, R. Mohammadi*, University of Alberta, Canada; *Z. Lee, V. Radmilovic, U. Dahmen*, NCEM, Lawrence Berkeley National Laboratory; *D. Mitlin*, University of Alberta, Canada

Macro and micro-scale portable Pd-based resistive hydrogen sensors are considered an established technology. However, because hydrogen needs to both dissociate at the Pd surface and diffuse into the material in a significant quantity, existing portable Pd sensors suffer from relatively slow response times coupled with the requirement of high hydrogen partial pressures for detection. The difficulty in achieving nano-scale sensors, which are intrinsically faster and more sensitive, is related to the difficulty of reproducibly depositing, patterning, etching and releasing very thin Pd lines with high surface to volume ratios. We have developed a family of new thin films based on the Pd-Ta-X (where X is one or more alloy additions) system that have an amorphous-nanocrystalline microstructure. This microstructure is unique and results in materials with exceptional properties including little or no deposition shadowing effects, near atomic level smoothness, very high nanoindentation hardness coupled with ductility, a tunable elastic modulus, metallic conductivity and the ability to still dissociate and absorb hydrogen. These unique features of the Pd-Ta-X nanocomposites have allowed us to fabricate resistive hydrogen sensors with nano-scale width and thickness, but a meter-scale total length, all contained within a micron scale device on a silicon substrate. The

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combination of a short diffusion length and ultra-high surface to volume ratio has resulted in exquisite detection sensitivity and a very fast response time in these devices. The sensors are strong enough to be partially released from their substrates effectively doubling their surface area to volume ratio, and are easily functionalized to be hydrophobic. We were also able to use these alloys to fabricate the first generation of fully released single-anchored nanometer scale cantilevers, to be used in both static and resonance gas detection mode.

# Wednesday Morning, November 15, 2006

## Nano-Manufacturing Topical Conference Room 2018 - Session NM+NS+NNT-WeM

### International Developments in Nanoimprint Lithography

**Moderator:** S. Matsui, University of Hyogo, CREST JST, Japan

8:00am **NM+NS+NNT-WeM1 Nanoimprint: Diversity in Materials, Processes and Application**, *H. Schift*, Paul Scherrer Institute (PSI), Switzerland; *J. Ahopelto*, VTT Centre for Microelectronics, Finland; *A. Kristensen*, Technical University of Denmark; *C.M. Sotomayor Torres*, Tyndall National Institute, Ireland; *M. Tormen*, INFN-TASC, Italy **INVITED**

Replication by molding offers more than high resolution and mass fabrication aspects. By creating a 3D structure by mechanical displacement of material, the patterning of a range of specific functional materials becomes possible, without losing their chemical properties. Furthermore this ability can be used to fabricate complex structures, e.g. by building up devices with imbedded channels. Many of these aspects play a role within the European Integrated Project NaPa. @footnote 1@ Its aim is not only the further development of emerging nanopatterning methods, including processes, tools, and materials, but also a range of applications, which go far beyond the development of a next generation nanolithography for chip manufacturing. Nanoimprint lithography of thermoplastic materials remains the technology with the imminent highest potential, because of its ability to integrate different materials and functionalities, and by its compatibility with other mass fabrication processes such as injection molding and roll embossing. @footnote 2@ Typical applications can be seen in the topological and chemical patterning of templates for displays and cell growth, the integration of fluidic and optical functionalities in a lab-on-a-chip, and patterning of special polymers intended for polymer electronics and optics. This is achieved by using new thermoplastic polymers, or by incorporating dyes and nanoparticles with specific functionalities, e.g. for optical devices. Throughput is enhanced by using heatable stamps, and flexibility by a step and stamp approach. This toolbox, ranging from mold fabrication to analytical tools, is a prerequisite to establish nanoimprint as a future core technology in the heart of Europe. @FootnoteText@ @footnote 1@ EC-funded project NaPa (Emerging Nanopatterning Methods). URL: <http://www.phantomsnet.net/NAPA/index.php.@footnote 2@> *H. Schift* and *A. Kristensen*. In Handbook of Nanotechnology, *Bhushan B. ed.*, 2nd edition, publisher Springer Verlag, Germany (2006).

8:40am **NM+NS+NNT-WeM3 Nanoimprint Lithography: Technology, Applications and Commercialization**, *S.Y. Chou*, Princeton University **INVITED**

Since the proposal of nanoimprint lithography (NIL) as a low-cost high-throughput sub-10-nm manufacturing method in 1995, @footnote 1@ the field has been growing rapidly in research, applications and commercialization in the past 11 years. The talk will present author's bird-eye view in these areas, as well as future potential and new directions. Particularly, the talk will address (a) NIL progresses in minimum feature size (6 nm half-pitch), printing areas (over 50 in-sq), alignment (sub-20 nm), pattern shapes (2D and 3D), materials and masks, (b) NIL applications in different disciplines, such as nanoscale electronics (nano-MOSFETs and SRAMs), photonics, displays, data storage (optical and magnetic), biotech, chemical synthesis and advanced materials, and (c) commercialization of NIL technologies. The talk will conclude that despite the success of NIL in its first 10 years, it is still just a beginning and we haven't seen anything yet! NIL will serve as an enabling ubiquitous manufacturing engine to propel future research in nanotechnology, future manufacturing of micro/nanostructures, and hence future discoveries and commercialization in nanotechnology. @FootnoteText@ @footnote 1@ *S. Y. Chou*, *P. R. Krauss*, and *P. J. Renstrom*, *Appl. Phys. Lett.*, 67 (21), 3114 (1995)

9:20am **NM+NS+NNT-WeM5 Nanoimprint and Nanopatterning in Japan**, *H. Hiroshima*, National Institute of Advanced Industrial Science and Technology (AIST), Japan **INVITED**

In a while after Prof. Chou had revealed the potential of nanoimprint a decade ago, a few groups in Japan started studying on nanoimprint technology. But now many groups use nanoimprint and notice the ability. People can feel the nanoimprint fever in Japan as indicated by the nanoimprint and nanoprint technology conference (NNT) 2005 held in Nara. In Japan, many nanotechnology projects run under support by the government; however, nanopatterning using nanoimprint is strangely excluded. One of recent topics on research situation is that Nanoimprint

Process Solutions (NiPS) Consortium was established in April 2005. The group of Dr. Maeda in national research institute of advanced industrial science and technology (AIST) plays a central role in the consortium and provides technical supports to the NiPS members. Another is that a nanoimprint technology study group, whose chair person is Dr. Komuro in AIST, was born in April 2006 as a valuable by-product of NNT2005. There are three powerful groups in the study group; one is the group of Prof. Matsui at university of Hyogo that shifted its focus from thermal nanoimprint to room-temperature nanoimprint; another is the group of Prof. Hirai at Osaka prefecture university that fabricates fluid devices using an improved reversal nanoimprint; and the other is our AIST group "nanoimprint manufacturing technology group" that covers high-temperature nanoimprint onto glass materials, roller nanoimprint and also UV-nanoimprint. In my presentation, I should like to introduce outlines on the activities of these groups and to show the diversity of research and development of the nanoimprint technology in Japan.

10:40am **NM+NS+NNT-WeM9 Metrology for Nanoimprint Technologies: Needs and Prospects**, *C.L. Soles*, *H.W. Ro*, *Y. Ding*, *H.J. Lee*, *R.L. Jones*, *A. Karim*, National Institute of Standards & Technology **INVITED**

Nanoimprint is a next generation lithography candidate with enormous potential not just for the semiconductor industry, but also a wide range of emerging technologies. This is because nanoimprint combines a fine patterning resolution, comparable to e-beam lithography, with a high throughput and low cost tool. However, there are several aspects of the nanoimprint process that significantly differ from state of the art optical lithography currently used for high volume nanofabrication and these differences introduce new metrology challenges unique to the nanoimprint process. Equally as important, the potential increase in the patterning resolution of nanoimprint over current patterning technologies threatens to exacerbate many of the existing metrology challenges presently facing optical lithography. In this presentation we review the unique and the non-unique metrology challenges facing nanoimprint lithography. These metrology needs come from interactions with both industrial and academic practitioners of nanoimprint through international workshops and panel discussions. We will present an overview of the progress being made to meet these metrology needs. Specific examples from our own research in developing novel X-ray scattering and reflectivity measurements to facilitate the nanoimprint processes will be highlighted.

11:20am **NM+NS+NNT-WeM11 Large Area Ultraviolet Nanoimprint Lithography Applicable to Flat Panel Display**, *E.-S. Lee*, Korea Institute of Machinery & Materials, Korea **INVITED**

Nanoimprint lithography (NIL) is known as an emerging lithography with a resolution of less than 10 nm, having a high throughput and low cost compared to the conventional photolithography. In this talk, we present very large area (> 20 inch) Ultraviolet Nanoimprint Lithography (UV-NIL) process as a breakthrough strategy for flat panel display industry. As thin film transistor liquid crystal display (TFT-LCD) manufacturers in Korea ramp up output of TV panels from advanced factories in anticipation of strong demand, the excess supply in the market is leading to steep price declines especially for TV use panels. Succeeding generation plants use larger glass substrates, thereby boosting output and helping manufacturer's lower production costs. Falling LCD panel prices may be tough for LCD manufacturers to swallow. There is something in creating profit besides investment in upgrading generation. That can only be possible by converting process facilities to use advanced process technology. This talk will summarize activities for the development of large area UV-NIL at KIMM to date and in detail. Very large area UV-NIL process and its apparatus for the TFT-LCD pattern will be presented.

## Nanometer-scale Science and Technology

### Room 2016 - Session NS+SS+TF-WeM

#### Nanotribology and Nanomechanics

**Moderator:** J. Harrison, U.S. Naval Academy

8:00am **NS+SS+TF-WeM1 Nanotribological Properties of Diamond-Like Carbon Thin Films: The Effect of Annealing on Nanoscale Adhesion and Friction**, *D.S. Grierson*, *A.R. Konicek*, *A.V. Sumant*, *K. Sridharan*, *R.W. Carpick*, UW-Madison

Developing micro- and nano-scale devices with contacting or sliding parts continues to be challenging due to the poor tribological performance of conventional materials at the micro/nanoscale. The surface-to-volume ratio at small scales is high, and therefore materials with low nanoscale

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adhesion, friction and wear are needed to reduce tribological failures. Additionally, in applications where materials are subjected to cyclic thermal loadings, such as nanoscale thermomechanical data storage, the structure and tribological properties must remain stable. Diamond-like carbon (DLC) thin films have exceptional physical, chemical and tribological properties at the macroscale and are promising candidates for tribologically robust micro/nanoscale devices. We have studied the surface chemistry and nanotribology of undoped, Si-containing, and F-containing DLC, and investigated how annealing these films at 300°C in air affects these properties. We used the XANES (x-ray absorption near-edge spectroscopy), a surface-sensitive probe of the core-hole perturbed local density of unoccupied states, to understand the evolution of the surface chemistry and bonding. The  $\text{sp}^3/\text{sp}^2$  ratio is increased by the addition of Si but not by F. The Si-containing DLC shows increased thermal stability. Atomic force microscopy (AFM) with DLC-coated AFM tips was used to conduct self-mated nanotribology experiments. The AFM results indicate that all DLC films exhibit adhesion on the order of van der Waals forces ( $\sim 0.03 \text{ J/m}^2$ ), and the nanoscale adhesion and friction on the Si-containing DLC are not affected by the thermal annealing. This indicates that DLC films, particularly those doped with Si, are highly promising for nanoscale thermomechanical device applications.

**8:20am NS+SS+TF-WeM2 Spectromicroscopy of Tribochemistry: X-PEEM Characterization of Wear vs. Humidity for Ultrahard Carbon Films, A.R. Koniczek, D.S. Grierson, A.V. Suman, UW-Madison; N.N. Naguib, O. Auciello, J.A. Carlisle, Argonne National Lab; T.A. Friedmann, J.P. Sullivan, Sandia National Labs; J. Birrell, Advanced Diamond Tech.; P.U.P.A. De Stasio, R.W. Carpick, UW-Madison**

The outstanding tribological performance of carbon-based films can be seriously affected by variations in humidity. To explore the tribochemical origins of this behavior, self-mated interfaces of tetrahedral amorphous carbon (ta-C) and ultrananocrystalline diamond (UNCD) films were subjected to fretting wear in dry N<sub>2</sub> with relative humidity values of 0%, 25% and 50%. Relative friction was measured during fretting, and the resulting wear tracks were examined with atomic force microscopy (AFM) and X-PEEM-XANES (X-ray PhotoElectron Emission Microscopy combined with X-ray Absorption Near-Edge Structure) spectromicroscopy. X-PEEM is capable of discerning chemical and bonding contrast at high spatial resolution, readily distinguishing between the wear track and the unworn film. For both films, friction increases as the relative humidity decreases. However, there are opposing trends in the behavior of graphitization and oxidation due to wear. For ta-C, as the relative humidity is decreased there is an increase in both graphitization and oxidation. This implies that the harsher wear environment has more heavily modified the carbon bonds compared to the wear at a higher relative humidity, inducing graphitization and oxidation. In contrast, the self-mated UNCD interface showed a comparative decrease in graphitization and oxidation in the wear track as the relative humidity was decreased. We will discuss changes observed in both friction and the chemical signature of the surface as the amount of relative humidity in the environment is varied. We will also discuss the relation between the amount of relative humidity in the environment and the level of graphitization that occurs in the wear track. @FootnoteText@ This work was partially supported by the US Department of Energy, BES-Materials Sciences, under Contract W-13-109-ENG-38.

**8:40am NS+SS+TF-WeM3 The Role of Atomic Corrugation, Crystal Orientation, and Surface Chemical Bonding in the Nanotribology of Carbon-Based Systems, R.W. Carpick, University of Wisconsin-Madison INVITED**

A key challenge for nanotechnology lies in developing an understanding of nanotribology, particularly for materials with outstanding tribo-mechanical properties such as carbon-based films. Frictional slip and wear in these materials can be manifested in unique and surprising ways. As a fundamental example, we show that nanoscale friction can exhibit clear transitions from smooth sliding to single slips and then multiple slips. The slips are directly correlated with the atomic lattice of the sample, in this case pure graphite. The observation of the transition to multiple slips is new, and is understood by considering the competition between the stiffness of the interatomic interfacial potential and the elastic stiffnesses of the contacting materials and the force sensor itself. The transition to smooth sliding with ultralow dissipation in open air is observed for the first time, and atomic-scale stick-slip is observed for interfaces orders of magnitude larger than any previously tested. Atomic-scale stick-slip may therefore be a far more prevalent phenomenon than currently appreciated. We have also extensively studied the nanotribological behavior of other carbon-based systems, including single crystal and

nanocrystalline diamond. The atomic structure of the surface, verified by detailed surface spectroscopy, critically affects friction and adhesion. Hydrogen termination is particularly effective in reducing friction and adhesion to the limit of van der Waals' interactions. Friction and adhesion are also affected by the crystal orientation. For larger tips, continuum mechanics models of contact area can be applied to understand the interfacial mechanics of these nano-scale contacts, as evidenced by the observation of direct proportionality between friction and contact area, a phenomenon known as "interfacial friction". By using smaller tips coated with a carbonaceous film, the limits of continuum mechanics are explored and discussed.

**9:20am NS+SS+TF-WeM5 Influence of the Solvent Environment on the Contact Mechanics of Tip-Sample Interactions in Friction Force Microscopy of Self Assembled Monolayers, T.J. Colburn, G.J. Leggett, University of Sheffield, UK**

The application of friction force microscopy (FFM) to the characterisation of surface composition and nanoscale tribological phenomena requires an adequate understanding of the tip-sample contact mechanics. We present new data that show that the properties of the liquid medium influence not only the strength of the frictional interaction in FFM, but also the nature of the contact mechanics model that describes the tip-sample interaction. FFM measurements have been carried out on self assembled monolayers of dodecanethiol (C@sub 10@CH<sub>3</sub>) and mercaptoundecanoic acid (C@sub 10@COOH) in a variety of liquid media using tips functionalised with alkanethiols (chemical force microscopy). In perfluorodecalin, a liquid with a low dielectric constant, the friction-load relationship fits the behaviour predicted using the Johnson-Kendall-Roberts model for like pairs of interacting molecules, and the Derajuin-Muller-Toporov model for unlike molecules. In contrast, measurements in ethanol, a liquid with a larger dielectric constant, obey Amontons' law (i.e. the friction force is linearly proportional to the load). These findings suggest that single asperity contact mechanics are observed in media with low dielectric constants, where dispersion forces are very strong and frictional interactions are adhesion-controlled. In media with large dielectric constants, sliding is not adhesion-controlled and linear friction-load behaviour is observed.

**9:40am NS+SS+TF-WeM6 Indentation of Individual and Multiple Multi-Walled Carbon Nanotubes with AFM, H.W. Yap, R.W. Carpick, University of Wisconsin-Madison**

There has been a recent surge of theoretical interest in the axial compression of MWCNTs, but only a limited number of experimental studies have been conducted. The study of axial compression of MWCNTs sheds light on their mechanical properties and has interesting implications in the fabrication of CNT - based composites. Lakes et al.@footnote 1@ have shown that macroscopic tubes have better damping properties in the post kink - buckled, snap-through regime, which is marked by a force drop with increased compression. Waters et al.@footnote 2@ and Qi et al.@footnote 3@ have indented an array of vertically aligned MWCNTs but have not observed force drops indicating these snap-through instabilities. Here, a stiff AFM cantilever with a microsphere tip attached to it is used to indent and compress a film of vertically-aligned MWCNTs with aspect ratio of  $\sim 35$ . These CNTs are grown with dc plasma-enhanced hot filament chemical vapor deposition from an anodized alumina nanopore template. This is the first use of spherical probe AFM to compress aligned CNTs, though this method has been widely used in indenting biological and polymeric materials. Continuous cycles of loading and unloading to large strains were performed, and both large drops and finer relaxations in the loading portions of the force curves of a collection of MWCNTs were observed with a high degree of reproducibility. The drops are likely signatures of collective kink buckling instabilities seen in individual CNTs. The deformations, while nonlinear, are mostly elastic and reversible, as demonstrated by SEM images of the spherical tip and the sample after many cycles of deformation. We will discuss the implications of these results in the design of composites that take advantage of buckling instabilities for high damping applications. @FootnoteText@ @footnote1@ Lakes et al., Phil. Mag. Lett., 81, 95 (2001). @footnote2@ Waters et al., Appl. Phys. Lett., 85, 1787 (2004) @footnote3@ Qi et al., J. Phys. Mech. Sol., 51, 2213 (2003).

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10:40am **NS+SS+TF-WeM9 Measurements and Modeling of Shear Modulus of Multiwalled Tungsten Disulfide Nanotubes**, *I. Kaplan-Ashiri, S.R. Cohen, N. Apter, H.D. Wagner, R. Tenne*, Weizmann Institute of Science, Israel; *G. Seifert*, Technische Universität Dresden, Germany; *R. Shneck, D. Barlam*, Ben Gurion University, Israel

Recent investigations of the mechanical properties of inorganic fullerene have highlighted their unique properties. Although the bending modulus has been measured on single nanotubes, the shear modulus is much smaller and technically more difficult to ascertain. The S-W-S sandwich structure of individual layers of these inorganic nanotubes distinguish them chemically and mechanically from carbon nanotubes. The spacing of 6.18Å between layers is comprised of the tungsten disulfide entity, and a van der Waals gap. Direct measurements of the shear modulus of single tubes could elucidate how the tube mechanics are influenced both by the gap, and the interaction between the adjacent dichalcogenide atomic layers. Measurements were made in a scanning probe microscope (SPM) using a method similar to that described by Wu et al.: Nanotubes were dispersed on a grid structure consisting of trenches of depth 200 nm and width 500 nm. Scanning electron microscopy (SEM) was used to locate nanotubes which were appropriately aligned, and suspended over such a trench. These nanotubes were then glued to the substrate at the trench edge with amorphous carbon. The SPM was subsequently used to bend the suspended nanotubes from the side with the SPM probe by amounts ranging between 5 and 100 nm, while recording the lateral force. The results were analyzed by applying the bending equation using the known value of the bending modulus to extract shear modes. Results were further compared with both density functional calculations, and finite element analysis modeling, providing insights into mechanical interactions between the layers. @FootnoteText@ @footnote 1@ I. Kaplan-Ashiri, et al, Proc. Nat. Acad. Sci. 103, 523 (2006).@footnote 2@ B. Wu, et al, Nature Mat. 4, 525 (2005).@footnote 3@ Yu et al, Science 287, 147 (2000).

11:00am **NS+SS+TF-WeM10 Deformation Behavior of Low-Density Nanoporous Dielectrics**, *S.O. Kucheyev, P.M. Bythrow, T.F. Baumann, C.A. Cox, Y.M. Wang, T. van Buuren, A.V. Hamza*, Lawrence Livermore National Laboratory; *J.E. Bradby*, The Australian National University

Understanding deformation behavior of nanoporous glasses has recently regained tremendous interest in the community, primarily due to its importance for the development of a new generation of low-k dielectrics. Aerogels are sol-gel-derived nanoporous dielectric materials formed by nanometer size particles randomly interconnected into a solid network with a large degree of porosity and very high surface area. For these materials, the density can be varied from the theoretical maximum density (of a full density solid) to extreme cases of very high porosities (~99 %). Thus, aerogels represent a very attractive model system for studying the deformation mechanisms in nanoporous dielectrics. In this presentation, we discuss the use a combination of sound velocity measurements and nanoindentation with large spherical indenters (1-2 mm diameter) to study the deformation behavior of alumina and tantalum aerogels with porosities up to 99%. In particular, we focus on how the deformation behavior is affected by (i) the average density of monoliths, (ii) the morphology and connectivity of nanoligaments, and (iii) the crystallographic phase. Results show that all of the above parameters can strongly affect the mechanical properties of nanoporous solids. Based on our results, we discuss the underlying deformation mechanisms and demonstrate an effective way to control mechanical properties of the nanoporous solids that can be synthesized with ligaments having a quasi-two-dimensional shape, such as platelets, ribbons, or leaflets. Work at LLNL was performed under the auspices of the U.S. DOE by the University of California, LLNL under Contract No. W-7405-Eng-48.

11:20am **NS+SS+TF-WeM11 Nanoporous Au - a 3D Network of Ultra-High Strength Nanowires**, *J. Biener, A.M. Hodge, J.R. Hayes, M. Duchaineau, L.A. Zepeda-Ruiz, A.V. Hamza, F. Abraham*, Lawrence Livermore National Laboratory

Recent mechanical studies on nanoporous gold (np-Au) have revealed that the yield strength of this material is almost one order of magnitude higher than predicted by scaling equations developed for open-cell foams. The higher-than-expected yield strength seems to be linked to the nanoscale morphology of np-Au which can be best described as a three-dimensional network of ultra-high strength Au nanowires. Here, we compare experimental results with molecular dynamics simulations to elucidate the nature of the high yield strength of nanoporous gold. This work was performed under the auspices of the U.S. Department of Energy by

University of California, Lawrence Livermore National Laboratory under contract of No.W-7405-Eng-48.

11:40am **NS+SS+TF-WeM12 Superconductivity Dependent Friction of Water, Nitrogen and Superheated He Films Adsorbed on Pb(111)**, *J. Krim, M. Highland*, North Carolina State University

The ability to predict sliding friction in adsorbed film systems underlies a vast range of topics in physics and nanotechnology, spanning the origins of static friction to the design of atomic-scale automobiles. One still hotly debated topic in this area is the degree to which electronic effects contribute to friction. In order to explore this issue, we have performed a quartz crystal microbalance study of sliding friction levels in nitrogen, water and superheated helium films adsorbed on Pb(111)@footnote 1@ and also self-affine fractal Pb substrates alternating in and out of the superconducting state. Reductions in friction upon entry into the superconducting state are greater for nitrogen than helium, consistent with a recent theory that linked electronic friction to adsorbate polarizability. Repetitive cycling of the externally applied magnetic field is observed to reduce overall friction levels.@footnote 2@ @FootnoteText@ @footnote 1@ M. Highland and J. Krim, Phys. Rev. Lett., in press (2006)@footnote 2@ Work supported by NSF and AFOSR.

12:00pm **NS+SS+TF-WeM13 In Situ Quantitative TEM Nanoindentation of Individual Nanoparticles and Nanoscale Materials**, *Z. Shan, A.M. Minor*, Lawrence Berkeley National Laboratory; *S.A. Syed Asif, O.L. Warren*, Hysitron Inc.

Monitoring the microstructure evolution while simultaneously measuring the stress and strain information at the nanometer level has been a long standing goal for material scientists. Here we show that by incorporating a miniature capacitive transducer into a TEM holder the load-displacement response (force resolution better than 0.5 Ån and displacement resolution better than 1 nm) can be achieved inside a TEM during the in situ nanoindentation process. A wide range of materials have been examined using this technique, such as Al thin films, single crystal Ni and Cu, Au and Cd metallic nanoparticles, and hollow shell-structured nanoparticles. The preliminary results will be reported and the physical insight derived from these results will be discussed. In particular, our results demonstrate unique insight into the initial deformation processes during the nanoindentation of metals and the analysis of elastic moduli and plasticity in nanoparticles.



## Nanometer-scale Science and Technology

### Room 2016 - Session NS+NM-WeA

#### Nanolithography and Patterning

Moderator: S.W. Pang, The University of Michigan

2:00pm **NS+NM-WeA1 Direct Deposition of Ordered Polymer Nanostructures in UHV via thermal Dip-Pen Nanolithography**, *P.E. Sheehan, M. Yang, A.R. Laracuent*, Naval Research Laboratory; *B.A. Nelson, W.P. King*, Georgia Tech; *L.J. Whitman*, Naval Research Laboratory

In thermal Dip Pen Nanolithography (tDPN) a heated atomic force microscope cantilever controls the deposition of a solid ink, acting like a nanoscale soldering iron. tDPN has several advantages over conventional DPN. Control over writing is greatly improved deposition may be turned on or off and the deposition rate easily changed without breaking contact with the surface. In addition, imaging with a cool tip does not appear to contaminate the surface, thereby allowing in situ confirmation of the deposited pattern. Finally, tDPN can deposit a range of materials that are immobile at room temperature from semiconductors to insulators to metals. Thermal DPN is particularly suited to the deposition of polymers. To date, mylar, MEH-PPV, and poly(3-dodecylthiophene) [PDDT] have all been successfully deposited. PDDT is of particular interest as a conducting polymer with great potential for use in organic electronic devices. Using tDPN, well-ordered PDDT nanostructures have been deposited on silicon oxide and gold surfaces with layer-by-layer thickness control. By adjusting the tip heating power and the writing speed, we can vary the polymer thickness from a single monolayer (~2.6 nm) to tens of monolayers with lateral dimensions below 100 nm. Moreover, the morphology of the nanostructure suggests that the polymer strands are aligned along the path of the AFM tip. Unlike conventional DPN inks, the low vapor pressure of solvent-free polymers allows deposition in Ultra High Vacuum (UHV). We have deposited in UHV single monolayers of highly-ordered PDDT nanostructures on clean Si(001)-(2x1). The electronic and crystallographic properties of these structures will be discussed.

2:20pm **NS+NM-WeA2 Multiplexed Electroless Nanopatterning of Metallic Arrays Via Scanning Probe Lithography**, *S.A. Backer*, University of California, Berkeley; *M. Rolandi*, University of California, Lawrence Berkeley National Laboratory; *D. Okawa*, University of California, Berkeley; *J.M.J. Fréchet*, University of California, Lawrence Berkeley National Laboratory

Direct surface patterning of metallic species is essential for the fabrication of ever smaller nanostructures, such as electrodes, magnetic data storage devices and specific catalytic sites for localized chemical reactions. Conventional lithographic techniques based on radiation can achieve remarkable resolution and pitch. However, limitations lie in the multiplexing capabilities with the need of a separate alignment and lithography step for each metal deposited. We present a multiplexed lithographic technique based on the localized surface modification of silicon using an atomic force microscope (AFM) in fluid. The sharp tip of the AFM is used to create pinhole defects in the thin oxide present on the silicon surface. Spontaneous electroless deposition of the metal ions dissolved in the fluid occurs at the defect sites creating metallic islands as small as tens of nanometers. We demonstrate the fabrication of high pitch arrays of as many as thousands of islands of Au, Co, Sn and Fe. By consecutively exposing a sample to various metallic salts in solution, nano islands composed of different metals can be deposited in a single patterning session without needing to realign the sample or change the probe. This allows for the fabrication of multiplexed interdigitated arrays of nanostructures that could be used for orthogonal catalysis.

3:00pm **NS+NM-WeA4 High-Density Gigabit Patterning of Sub-100nm Circular Hole/Dot Arrays by Nanoimprint Lithography**, *W. Hu*, Stanford University, U.S.A.; *R.J. Wilson, L. Xu, S.J. Han, S.X. Wang*, Stanford University High throughput and cost-effective patterning of sub-100nm periodic features is of great interest for science and engineering. In this work we present results from patterning high-density arrays of circular holes with 100nm diameters at 300nm pitch, or with 50nm diameters at 100nm pitch, throughout an area of 1 square cm. We use 75K molecular weight polymethyl methacrylate (PMMA) resist and thermal nanoimprint lithography. Several issues are addressed which arise during patterning and characterization, such as pattern distortion control during debonding and PMMA damage induced by Au metallization for scanning electron microscopy (SEM). We further demonstrate the fabrication of dense gigabit magnetic nanodot arrays using PMMA and polymethyl glutarimide (PMGI)

resist bi-layers and a lift-off process. Metal residuals with different shapes are found to be associated with variable resist processing, metallization, and lift-off conditions and are removed by an ion mill process. Additionally we show that the nanodot diameter can be tuned by using oxygen plasma ashing. Finally, the magnetic properties of nanodot arrays are shown to be readily characterized by alternating gradient magnetometry (AGM).

3:20pm **NS+NM-WeA5 Advances in Nanostructure Fabrication Technology**, *G. Willson, M. Stewart*, The University of Texas, Austin  
**INVITED**

The drive to manufacture cheaper and more efficient semiconductor devices has inspired remarkable improvements in imaging materials science and high resolution patterning technology for several decades. Billions of dollars have been spent in efforts to devise methods and materials that enable the printing of ever smaller transistors. Microelectronic devices in full scale production today have minimum features in the range of 70-90nm and fully functional CMOS transistors with 10nm gates have been characterized. The lithographic process that has been used to generate these "nano-structures" is becoming extremely expensive and the process window or process latitude for producing them is shrinking. The cost of the imaging process threatens the economics of the semiconductor manufacturing industry. A single efficient factory equipped to produce the next generation of devices is projected to cost as much as the gross national product of countries the size of Hungary. Imprint lithography, a lower cost, high resolution patterning technology is emerging as a potential adjunct to photolithography. Some view it as a truly disruptive patterning technology. Imprint lithography loosely defines a set of techniques that includes several forms of embossing; stamping and molding that show great promise as low cost methods for producing nanostructures. These techniques take many different forms each of which has its own special applicability. The technique we call Step and Flash Imprint Lithography (S-FIL) is designed to allow the fabrication of high resolution, high aspect ratio images that can be aligned with precision. The process accurately replicates arbitrary shapes as small as 10nm. The process can be used to define structures with very small widths but unlike photolithography, it can be used to produce very small three dimensional structures via simultaneously controlling variations in depth. A progress report on modern patterning technologies will be presented with emphasis on imprint lithography.

4:00pm **NS+NM-WeA7 Photolithography Beyond the Diffraction Limit**, *G.J. Leggett*, University of Sheffield, UK

A scanning near-field optical microscope (SNOM) coupled to a UV laser has been used to pattern self-assembled monolayers (SAMs) of photosensitive adsorbates. Using this approach, which we call scanning near-field photolithography (SNP), it is possible to achieve a resolution of 9 nm (nearly 15 times smaller than the conventional diffraction limit) in a SAM of alkanethiols adsorbed on gold. In conjunction with a new etchant, mercaptoethylamine in ethanol, SNP-patterned SAMs have been used as resists to fabricate gold nanostructures as small as 10 nm that have sharp edge definition. Gold nanowires, 60 nm wide, have been fabricated by selective exposure of bilayers of thiol-stabilised gold nanoparticles, followed by rinsing, in a simple two-step process. The principal criterion for achieving high resolution using SNP is the excitation of a specific photochemical reaction in a group distributed with monolayer coverage on a solid surface. This encompasses a broad range of materials. This is illustrated using monolayers of chloromethylphenyl siloxanes adsorbed on silicon dioxide, which may be selectively converted to aldehydes or to carboxylate functionalities to which biological molecules may be attached. DNA nanoarrays have been fabricated that consist of 70 nm DNA spots at 500 nm spacings. These have a density 40 000 times greater than existing DNA chips, while remaining readable by far-field optical methods. Finally we demonstrate the feasibility of carrying out nanophotolithography on aluminium surfaces. Monolayers of aryl azide terminated phosphonic acid adsorbates, which exhibit substantially enhanced ambient stability compared to alkanethiol SAMs, have been formed and selectively functionalised using SNP, in a process 50 - 100 times faster than the alkanethiol patterning process. These illustrations indicate that SNP is a powerful, flexible and straightforward technique for the execution of specific surface chemical transformations.

# Wednesday Afternoon, November 15, 2006

4:20pm **NS+NM-WeA8 SPM Nanolithography of ZrN Thin Films: Nitrogen-Enhanced Growth and Hollow Oxide Feature Formation**, *N. Farkas, E.A. Evans, R.D. Ramsier*, The University of Akron; *J.A. Dagata*, National Institute of Standards and Technology

A systematic study of the scanning probe microscope (SPM) oxidation of sputter-deposited ZrN thin films is presented. Based on data covering an extensive range of parameters such as exposure time, voltage, humidity and nitrogen content of the sputtering plasma, we propose a four-stage oxidation kinetics model for ZrN. We find that the heights of the oxide features are significantly larger when the films are prepared with substantial nitrogen content in the deposition plasma, whereas small nitrogen flow rates yield oxidation rates similar to Zr films. In the intrinsic part of the SPM oxidation, the oxide density increases until the volume of the oxide is about twice that of the consumed ZrN. Further oxide growth is sustainable, and in fact faster yet controlled, as the system crosses over from the space charge limited to a nitrogen-enhanced growth regime. Most striking and different from other material systems is that high-voltage SPM oxidation of ZrN generates micrometer high features that are an order of magnitude higher than the thickness of the films. Selective etching of the oxides and nitrides reveals that as the oxidation reaches the ZrN/silicon interface delamination occurs resulting in hollow oxide feature formation through stress-induced plastic flow. Interpretations of the underlying processes and film properties responsible for the unique behavior of ZrN in all regimes are provided along with an explanation for the observed non-linear voltage dependence.

4:40pm **NS+NM-WeA9 High Resolution Electron Beam Induced Etching of Extreme Ultraviolet (EUV) Lithography Mask Films**, *M.G. Lassiter, P.D. Rack*, University of Tennessee; *T. Liang*, Intel Corp.

The application of EUV wavelengths for lithography is emerging as a viable technology to extend the production of small feature sizes for integrated circuits and nanotechnology. In order to make EUV lithography commercially viable, production of the mask requires the repair of defects found during the mask inspection. Traditionally, the highest resolution mask repair technology involves using a focused ion beam (FIB) of Gallium ions to physically sputter away material for etching and to induce dissociation of a precursor gas to deposit material for repairs. Some of the Gallium ions are implanted into the surface of the mask and leave undesirable effects such as reduced transmission of clear area repairs in photomasks and reduced reflection of the clear area of EUV masks. The use of electron beam induced processes for the repair of EUV masks has recently been developed as an alternative to FIB repair of masks. The electron beam induces the dissociation of a precursor gas to cause a reaction at the surface of the mask. This reaction either deposits material or causes the etching of the mask material, depending upon the type of precursor used and the substrate material. This work focuses on the latter case of electron beam induced etching (EBIE). Electron beam provides superior spatial resolution than that of the FIB, so the EBIE process can be more effective in small mask feature repairs. Also, the electron beam will not damage the mask materials because of the relatively small mass of the electron and relatively low energy used in the electron beam. This work characterizes the process of high resolution EBIE of Tantalum Nitride based EUV mask films. The effects of electron beam parameters such as accelerating voltage, beam current, and the scanning parameters are investigated. Furthermore, the use of various precursor gases is examined, and the effects on spatial resolution, etching rate, and selectivity against other materials are determined.

## Nanometer-scale Science and Technology

### Room 2020 - Session NS-WeA

#### Nanotube Devices and Processes

Moderator: D. Carr, Sandia National Lab

2:00pm **NS-WeA1 2006 AVS Peter Mark Award Lecture - Biofunctionalized Single-Walled Carbon Nanotubes: Purification, Properties, and Devices**, *M.C. Hersam*<sup>1</sup>, Northwestern University

INVITED

The utilization of single-walled carbon nanotubes (SWNTs) in large quantities for electronics, optoelectronics, biosensors, and medical applications will require SWNTs of the same physical structure, electronic type, and band gap. Since current methods of synthesis produce SWNTs with polydisperse structure and properties, post-production purification is highly desirable. Here, we demonstrate a scalable method for enriching

SWNTs by diameter and electronic structure using density gradient centrifugation. Importantly, this technique does not require covalent modification of the SWNTs, which can significantly degrade their outstanding electronic, optical, and mechanical properties. Furthermore, density gradient centrifugation is compatible with SWNTs of arbitrary length. To date, we have demonstrated purification in aqueous density gradients using a variety of noncovalent biofunctionalizations including sodium cholate, sodium dodecyl sulfate, and DNA. Atomic force microscopy analysis confirms enrichment by diameter and reveals SWNTs of various lengths between 0.1 and 1 micron. Furthermore, optical absorption and photoluminescence spectroscopy verify improvements in the monodispersity of SWNT optical properties. Finally, thin film field effect transistors (FETs) have been fabricated using SWNTs that are enriched by metal or semiconducting electronic type. The purified semiconducting SWNT thin films yield FET on/off ratios that are 10,000 times higher than their metallic counterparts. Overall, this non-destructive and scalable separation strategy is expected to impact a variety of applications for SWNTs where monodisperse structure and electronic properties are essential.

2:40pm **NS-WeA3 Recent Advance on the Synthesis and Device Applications of Aligned Carbon Nanotubes**, *C. Zhou*, University of Southern California

INVITED

We present a novel nanotube-on-insulator (NOI) approach to produce high-yield nanotube devices based on aligned single-walled carbon nanotubes. First, we managed to grow aligned nanotube arrays with controlled density on crystalline, insulating sapphire substrates, which bear analogy to industry-adopted silicon-on-insulator substrates. Based on the nanotube arrays, we demonstrated registration-free fabrication of both top-gated and polymer-electrolyte-gated field-effect transistors with minimized parasitic capacitance. In addition, we have successfully developed a way to transfer these aligned nanotube arrays to flexible substrates. Our approach has great potential for high-density, large-scale integrated systems based on carbon nanotubes for both micro- and flexible electronics.

3:20pm **NS-WeA5 Size Effects on Electrical Contacts to Nanotubes and Nanowires**, *F. Leonard*, Sandia National Laboratories; *A.A. Talin*, Sandia National Laboratories, 94550

Metal-semiconductor contacts play a key role in electronics. Here we show that for quasi-one-dimensional structures such as nanotubes and nanowires, side contact with the metal only leads to weak band re-alignment, in contrast to bulk metal-semiconductor contacts. Schottky barriers are much reduced compared with the bulk limit, and should facilitate the formation of good contacts. However, the conventional strategy of heavily doping the semiconductor to obtain ohmic contacts breaks down as the nanowire diameter is reduced. The issue of Fermi level pinning is also discussed, and it is demonstrated that the unique density of states of quasi-one-dimensional structures makes them less sensitive to this effect. Our results agree with recent experimental work, and should apply to a broad range of quasi-one-dimensional materials.

3:40pm **NS-WeA6 Electrical Contact Metallization to Self-Assembled Large-Scale Carbon Nanotube Arrays**, *J.H. Bak, B.Y. Lee, S.S. Hong, S. Hong, Y.D. Park*, Seoul National University, Korea

We report on the investigation of various metallization schemes to electronically probe self-assembled large-scale carbon nanotube arrays. Reliable and highly reproducible metallization scheme to realize ohmic contacts to single wall carbon nanotubes (swCNT) by Palladium and Gold metallic thin films is an important technological step for the realization of swCNT-based nanoelectronics and their applications as well as self assembled large scale CNT arrays being technological attractive for mass-production of swCNT-based devices. Although electrical contacts to individual swCNT by Cr, Ti and Pd have enabled the observations of ballistic electron transport in swCNTs,<sup>1</sup> the systematic study of metallization to self assembled large scale CNT arrays and the elucidation of its electrical properties have been limited. Large-scale CNT arrays are selectively patterned via directed-assembly strategy on areas defined by self-assembled monolayer patterns.<sup>2</sup> Metallization and electrical properties of resulting contacts are studied by patterning submicron contact electrode on 2 $\mu$ m wide CNT array elements by e-beam lithography followed by the e-beam evaporation of contact metals of which contact spacing is varied from 300nm to 2 $\mu$ m. Contact spacings greater than 2 $\mu$ m represent mixed nanotube junctions properties. We found Pd contacts show both ohmic and nonohmic contact behavior whereas Cr and Ti metallization result in nonohmic contacts. <sup>1</sup>FootnoteText@ <sup>2</sup>Footnote

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

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1@ D. Mann et al., Nano Letter 3, 1541 (2003).@footnote 2@ S. Rao et al., Nature 425, 36 (2003).

4:00pm **NS-WeA7 Devices Based on Individual Carbon Nanotubes**, *S. Roth, J. Meyer, D. Obergfell, S. Sahakalkan, M. Kaempgen*, Max Planck Institute for Solid State Research, Germany

**INVITED**

Combining advanced semiconductor technology, synthetic organic chemistry, and high resolution transmission electron spectroscopy it is possible to prepare electronic and electromechanical devices based on individual single-walled carbon nanotubes and to fully characterize these devices by electrical and mechanical investigations and electron diffraction. We present examples of ultrafine electrochemical electrodes, T-junction transistors, peapod transistors, electromechanical acceleration sensors, and a torsional pendulum. The state of the art of industrial applications is reviewed.

4:40pm **NS-WeA9 Damage-free Surface Modification of Carbon Nanotubes using Advanced Neutral Beam**, *K. Okumura, Y. Sato, K. Tohji, S. Samukawa*, Tohoku University, Japan

In an effort to realize carbon nanotube (CNT) FET, it is necessary to modify electric characteristic of grown CNTs by using plasma process. However, the conventional plasma process damages CNTs because charged particles and ultraviolet photons are irradiated to the CNTs. As a result, the CNT FET could not be practically fabricated using conventional plasma processes. Here, we have proposed damage free surface modification by using our developed neutral beam to resolve the problems and to practically fabricate the CNT FET without any damages. Neutral beam can almost eliminate irradiation of charged particles and ultraviolet photons to CNTs. In this study, we irradiated Ar plasma and Ar neutral beam to single-walled carbon nanotubes (SWCNTs). Raman spectra confirmed that the intensity ratio of D-band/G-band in SWCNTs irradiated by neutral beam was still kept at the same as that with no beam irradiation. Conversely, the intensity ratio of D-band/G-band was drastically increased by conventional plasma irradiation. Transmission electron microscopy could also confirm that SWCNTs did not have any damages after the neutral beam irradiation, whereas SWCNTs was destroyed by conventional plasma irradiation. Neutral beam process is very promising candidate for future CNT FET fabrication processes.

## Nanometer-scale Science and Technology Room 2016 - Session NS+BI-ThM

### Biological and Molecular Applications of Nanoscale Structures

**Moderator:** H.G. Craighead, Cornell University

8:00am **NS+BI-ThM1 A Nanometer-scale Gene Chip: Detecting Single Molecules of DNA using a Silicon Nanopore, G.L. Timp**, Beckman Institute  
**INVITED**

We describe a prospective strategy for reading the encyclopedic information encoded in the genome: using a nanopore in a membrane formed from an MOS-capacitor to sense the charge distribution in a single molecule of DNA. In principle, as DNA permeates the capacitor-membrane through the pore, the electrostatic charge distribution polarizes the capacitor and induces a voltage on the electrodes that can be measured. The sub-nanometer precision available through silicon nanotechnology facilitates the fabrication of this nanometer-scale gene chip, and molecular dynamics provides us with a means to design it and analyze the experimental outcomes. Double-stranded DNA is a highly charged, unusually stiff polymer. And so, the nano-electromechanics of the molecule profoundly affect the design of this detector. Consequently, we have explored the electromechanical properties of DNA using an electric field to force single molecules through synthetic nanopores in ultra-thin silicon membranes. At low electric fields  $E \approx 0.5 \text{ nm}^{-1}$ , while double-stranded DNA only permeates pores with a radius  $r \approx 1.5 \text{ nm}$  because the diameter of the double helix is about  $2 \text{ nm}$ . For pores

8:40am **NS+BI-ThM3 Single DNA Molecules Stretched in Electrospun Polymer Fibers, L.M. Bellan, J.D. Cross, E.A. Strychalski, J.M. Moran-Mirabal, H.G. Craighead**, Cornell University

We have deposited electrospun polyethylene oxide (PEO) fibers containing isolated single stretched DNA molecules. The ability to stretch single molecules of DNA is desirable for single-molecule sequencing techniques and also allows the study of the behavior of DNA molecules undergoing various forces. Electrospinning is a popular technique for quickly and easily depositing micro- and nanoscale diameter fibers from a variety of materials, and has recently been used in several studies as a method for assembling nanoscale particles and molecules. In the present study, a dilute concentration of fluorescently labeled lambda phage DNA molecules was added to the water solvent used to mix the PEO electrospinning solution. The solution was used to produce isolated nanofibers using the scanned electrospinning technique. The DNA molecules were stretched in-flight in the electrospinning jet and remained stretched when the fluid jet solidified into fibers. These fibers were deposited on coverslips and imaged using fluorescence microscopy. The embedded DNA molecules were seen as single lines of fluorescence ranging from under 3 microns to 19 microns, which is the extended length of the lambda DNA molecule at the base pair to dye labeling ratio used. The variation in length is thought to be due to variations in the electrospinning jet fluid dynamics. By tuning the process parameters we were able to obtain a distribution of stretched lengths with a mode of  $\sim 7$  microns. We also observed chain scission in some cases. Given the long relaxation time of DNA in the polymer solution and the high strain rates present in electrospinning jets, both stretching and sporadic chain scission are expected. Current work is focused on mechanical manipulation of the resulting fibers and DNA molecules embedded therein.

9:00am **NS+BI-ThM4 Polypyrrole Based Nano-Electrode Arrays Produced by Colloidal Lithography, P. Lisboa, A. Valsesia, P. Colpo, F. Rossi**, European Commission, Institute of Health and Consumer Protection, Italy

The implementation of sensor platforms providing high sensitivity of detection is a crucial step for the design of the new analytical device generation for biosensor developments. Electrochemical sensors have shown a high potential for this challenge. A drastic increase of sensitivity of bio-event detection<sup>1</sup> has been proven by designing platform with active/non-actives region at nanoscale. Besides, the electrochemical sensitivity can be as well enhanced by using nano-electrode arrays that increase mass transport rate.<sup>2</sup> Polypyrrole (PPy) is a good candidate to fulfil these requirements. Its preferential material for bio-analytical electrochemistry based sensor thanks to its excellent biocompatibility and higher conductivity, together with the possibility of being functionalised with biological relevant functional groups.<sup>3</sup> In this work PPy nano pillars were fabricated by electrochemically growing PPy in a nano-template of gold nano-seeds produced by colloidal

lithography. Atomic force Microscopy and Scanning Kelvin Microscopy demonstrated that PPy grown only inside the conductive gold seeds, creating conductive nano pillars surrounded by an insulating material. The nano-structured surfaces were studied by Cyclic Voltammetry using hexacyanoferrate and the typical sigmoidal shape voltammogram of nanoelectrodes was obtained.<sup>2</sup> The nanoelectrode character of the surfaces is a promising feature to improve the already high sensitivity of biosensors based in nanostructured surfaces. The functionalisation of the nano pillars with bio-active chemical functions gives the possibility of protein immobilization in specific nano-areas which is promising for the production of an array of nano-sensors.<sup>1</sup> @FootnoteText@<sup>1</sup> @footnote 1@A. Valsesia, P. Colpo, P. Lisboa, M. Lejeune, T. Meziani, F. Rossi, Langmuir 2006, 22, 1763.<sup>2</sup> @footnote 2@D. Arrigan, The Analyst, 2004, 129, 1157.<sup>3</sup> @footnote 3@P. Lisboa, D. Gilliland, G. Ceccone, A. Valsesia, F. Rossi, Applied Surface Science 2005, In Press.

9:20am **NS+BI-ThM5 Biofunctionalized Nanoshells for Biological Detection, J.E. Van Nostrand**, Air Force Research Lab; C.S. Levin, Rice University; J.M. Slacik, Air Force Research Lab; J.D. Hartgerink, N.J. Halas, Rice University; M.P. Kadakia, Wright State University; R.R. Naik, Air Force Research Lab

The ability to detect chemical and biological agents is arguably one of the highest priority technical challenges today. The capability to obtain specific information at and near single-molecule resolution is the ultimate goal in chemical detection. Recent advances in surface-enhanced spectroscopy have demonstrated that Surface Enhanced Raman Scattering (SERS) displays spectrum enhancements of several orders of magnitude when molecules are adsorbed onto metallic surfaces. Metallic nanostructures because of their plasmonic properties are attractive SERS substrates, in particular, nanoshells and nanorods. Combining the specificity of biomolecular recognition with these nanostructures might lead to increased sensitivity and selectivity. Localization of biological recognition motifs to the surface of these nanostructures will lead to large signal enhancements when bound to its target. Nanoshells will be functionalized with capture elements (peptide ligands and antibodies) and these biofunctionalized nanoshells will be tested for their ability to detect microorganisms using SERS.

9:40am **NS+BI-ThM6 Characterization of Silver Nanoparticles Films for the Development of TPB Biosensors, J. Wolstenholme**, Thermo Electron Corporation, UK; K. Bonroy, G. Borghs, F. Frederix, IMEC, Belgium; R.G. White, Thermo Electron Corporation, UK

Metal nanoparticle films have been subject of much research, primarily due to their interesting optical and electronic properties and due to their high surface-to-volume ratio. According to numerous studies, nanoparticle films are promising as precursors for metallic films, as catalysts and especially as sensing substrates for the development of novel biosensors such as the Transmission Plasmon Biosensor (TPB). This type of biosensor is based upon the optical properties of silver and gold nanoparticles which are used to sense the specific target molecules in a complex matrix. Hereby, the nanoparticles are to be functionalized with self-assembled monolayers (SAMs) of thiols or disulfide molecules. These monolayers form a covalent bond with the gold or silver surface and can have appropriate functional groups to allow the attachment of specific bioreceptors. For surface plasmon based biosensors such as TPB, gold nanoparticles are most frequently used due to their chemical stability and their relatively simple preparation. Nevertheless, various studies and models predict a much higher sensitivity using silver nanoparticles as TPB sensing substrate. However, at this moment the drawbacks for using silver films as sensing substrates are their instability due to the formation of an oxide layer. The latter will negatively influence the formation of well-organized SAMs of thiols and the subsequent functionalization of the particles with specific bioreceptors. In this paper, we describe the FTIR, the XPS and angle resolved XPS characterization of the multilayered TPB sensing substrates, comprising quartz, silanes, silver nanoparticles and thiol molecules. In this study, we compared the thickness of the silane layer, the oxidation ratio of the silver films and the density of the thiol SAM for both silver nanoparticle films and continuous silver films. Our study indicates that the deposition of SAMs decreases the formation of oxides on the silver nanoparticle films.

10:40am **NS+BI-ThM9 Surface Nanostructuring using Colloidal Particles for Improved Biocompatibility, C.J. Nonckreman, P.G. Rouxhet, Ch.C. Dupont-Gillain**, Université Catholique de Louvain, Belgium

Nanostructured surfaces offer new perspectives in different fields of application, including the design of biomaterials (implants, catheters, blood

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bags). The aim of this work is to create model surfaces presenting bimodal roughness characteristics (scales of 500 nm and 50 nm) using colloidal lithography. An appropriately designed nanoroughness is expected to modulate the effect of the surface chemical composition for controlling the interactions of cells and tissues with materials. Colloidal lithography was performed using adsorption of cationic polymers and adhesion of negatively charged colloidal particles. Polyallylamine hydrochloride was used to confer a positive charge to a glass substrate. On this conditioned surface, a layer of colloids (polystyrene latex) was formed owing to electrostatic attraction. Sequential steps of polycation adsorption and particle adhesion were applied on the substrate, which was then analyzed by scanning electron microscopy. Adjustment of conditions for incubation solutions (concentration, pH and ionic strength), rinsing and drying were tested in order to produce a high surface coverage with colloids and to minimize their aggregation. Thereby, a range of surface structures was obtained: layer of particles with a diameter of 470 nm, layer of particles with a diameter of 65 nm, bimodal roughness made by particles with a diameter of 65 nm on the top of particles with a diameter of 470 nm. The obtained surfaces are conditioned by adsorption of compounds which make them protein repellent, in particular Pluronic F68, a block copolymer of polypropylene oxide and polyethylene oxide. The surfaces finally obtained are being tested with respect to plasma protein adsorption, in particular competitive adsorption of fibrinogen and albumin, and to biocompatibility.

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## Nanometer-scale Science and Technology Room 2016 - Session NS-ThA

### Nanoscale Material Processing

**Moderator:** S. Jesse, Oak Ridge National Laboratory

#### 2:00pm NS-ThA1 Controlled Chemical Mechanical Polishing of Polysilicon and Silicon-Dioxide for Nanostructures, V. Joshi, A.O. Orlov, G.L. Snider, University of Notre Dame

In this paper, we report experimental results of silicon dioxide (SiO<sub>2</sub>@sub 2@) and polysilicon chemical mechanical polishing (CMP) to produce nanoscale features with very smooth surfaces. The sizes of the features polished ranged from 20 to 500 nm. Nanostructures were defined by e-beam lithography and reactive ion etching (RIE). For polysilicon polishing, the nanostructures were defined in ZEP-520A (positive tone e-beam resist) and pattern was transferred to the oxide substrate through RIE (CHF<sub>3</sub>@sub 3@/O@sub 2@) plasma. These etched nanostructures (70 nm deep trenches) were conformally filled with LPCVD polysilicon, and polished using a Logitech CDP system. The slurry used was Semi-Sperse P1000 from Cabot Microelectronics. Similarly for oxide CMP study, nanostructures were patterned on Si substrate using HSQ (negative tone e-beam resist) as the etch mask. These nanostructures in Si (~100 nm height) were covered with thick layer of PECVD SiO<sub>2</sub>@sub 2@. Polishing was done to planarize the sample and expose the Si structures. CMP-1150 slurry from Ektech was used for oxide CMP. The polished structures were studied using SEM (cross-sections) and AFM (surface). We report controllable CMP to realize ~20 nm thick layers after polishing. The RMS roughness of the polished surfaces was ~ 0.3 nm. Better control of the CMP process (2 nm/min removal rate) was demonstrated by using diluted slurry or pure DI water as the CMP slurry.

#### 2:20pm NS-ThA2 Properties of Carbon-Based Nanostructures Grown by Low-Pressure Plasma Enhanced Chemical Vapor Deposition, J.B.O. Caughman, L.R. Baylor, R.A. Kisner, K. Korsah, Oak Ridge National Laboratory

The role of the plasma in the growth of carbon-based nanostructures is being determined by relating plasma conditions to the physical and electrical properties of carbon nanofibers and nanosheets. Nanofibers have a cylindrical cone-like structure while nanosheets have a planar sheet-like structure. Forests of vertically aligned nanofibers and nanosheets have been grown using an inductively coupled plasma source operated from 30 to 100 mTorr. The plasma is composed of hydrogen and either acetylene or methane as the carbon source. The plasma conditions are determined by using mass spectroscopy and optical emission spectroscopy. Dilute mixtures of carbon containing gases in hydrogen lead to well-formed nanostructures, while excessive carbon in the plasma leads to an increase in amorphous carbon deposition. The presence of a nickel catalyst layer prior to deposition leads to nanofiber growth, while no catalyst leads to nanosheet growth. Substrate bias plays an important role in the formation of nanofibers (by controlling the physical etching component during deposition), while having a detrimental effect on nanosheet growth. Field emission from the material, as measured with a scanning probe, is found to be strongly dependent on the details of the nanostructure. Sparse forests or well defined nanosheet edges give the lowest threshold fields. Details of the effect of plasma properties and substrate conditions (bias and temperature) on the electrical/physical properties of the nanofibers will be presented.\* @FootnoteText@ \*Oak Ridge National Laboratory is managed by UT-Battelle, LLC, for the U.S. Dept. of Energy under contract DE-AC05-00OR22725.

#### 2:40pm NS-ThA3 Prospects and Processing of Soft Materials, G.G. Malliaras, Cornell University

INVITED

During the past two decades dramatic advances have been achieved in the performance of organic semiconductor devices. Light emitting diodes, transistors, solar cells, and several other devices can now be fabricated from organic semiconductors and are at various stages of commercialization. This fast-paced progress, which highlights the technological potential of soft materials, is largely a result of advances in our ability to deposit and pattern high quality films. To make this possible, traditional deposition and patterning techniques had to be adapted, and new ones had to be developed. Moreover, the integration of organic semiconductors with biologically-relevant materials, which share some of the processing challenges, promises the development of novel sensors with

unmatched capabilities. I will discuss recent advances in the processing of soft materials and the opportunities they have created.

#### 3:20pm NS-ThA5 Synthesis of Suspended Single-Walled Carbon Nanotubes on Tips of Silicon Nanostructures Fabricated by Plasma Etching, C.H. Weng, H.C. Su, W.Y. Lee, C.H. Tsai, K.C. Leou, National Tsing Hua University, ROC

Suspended single-walled carbon nanotubes (su-SWNTs) have attracted a great deal of attention recently. This is because (1) the perturbation from substrates can be minimized to allow probing the natural properties of SWNTs; and (2) nano-devices using su-SWNTs exhibit enhanced performance. In this work, we present a method to synthesize su-SWNTs selectively suspended on tips of silicon based nanostructure (Si-ns) templates formed by anisotropic hydrogen plasma etching. Conventional thermal chemical vapor deposition technique was employed to synthesize the SWNTs. Nano-sized islands or particles as formed from multi-layered thin-films (Mo/Fe/Al) at high temperature are served as the catalytic nanomasks (CNMs) during plasma etching, where the remaining CNMs left at tips directly become the catalysts for SWNTs growth. As revealed by characterizations using scanning electron microscopy and resonance Raman spectra, su-SWNTs form networks interconnecting ns-Si templates. This technique allows us to assemble two different nanomaterials, i.e., ns-Si and su-SWNTs, together as a building block for future possible applications. (Work supported by the grant from the National Science Council of the ROC.)

#### 3:40pm NS-ThA6 Fabrication of Thin Silicon Nanodisk for Quantum Effect Devices Using Cl Neutral Beam Etching and Ferritin Iron Core Mask, T. Kubota, T. Hashimoto, Tohoku University, Japan; M. Takeguchi, National Institute for Materials Science, Japan; Y. Uraoka, T. Fuyuki, Nara Institute of Science and Technology, Japan; I. Yamashita, Matsushita Electric Industrial Co., Ltd. and Nara Institute of Science and Technology, Japan; S. Samukawa, Tohoku University, Japan

Miniaturization of semiconductor devices will require new fabrication processes to realize nanometer-scale structures without process damages. To realize a nanometer-scale structure, we used ferritin iron core (7nm in diameter) as a uniform and high-density template, and our developed neutral beam (NB) etching process for damage-free etching. We have fabricated "nanodisk", a nanometer-thick disk-shaped silicon structure. Thin oxide layer of about 1nm was grown on Si(100) wafer and then 4-nm-thick Si layer was deposited. The Si layer was etched by using Cl NB with ferritin iron core as an etching mask. There were two problems which had to be solved to fabricate thin nanodisk. (1) The native oxide layer on thin Si layer causes enlarging the diameter of nanodisk. (2) The oxide layer must be remained while Si layer has to be completely etched. To solve these problems, two step NB etching process was performed. High-energy neutral beam was used for the first step to breakthrough the native oxide and low-energy neutral beam was used for the second step to obtain high etching selectivity to underlying-SiO<sub>2</sub>@sub 2@. To monitor the proceeding of atomic layer etching, XPS measurement was utilized. We could successfully observe the fabrication of silicon nanodisk (2nm in thick and 10nm in diameter) through cross-sectional TEM. Such nanometer-scale structure would be applied to quantum effect devices. A part of this work was supported by "Nanotechnology Support Project" of the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan. This study was supported by Leading Project of Ministry of Education, Culture, Sports, Science and Technology.

#### 4:00pm NS-ThA7 Cool Plasma Functionalization of Nanodiamond Particles, G.E. McGuire, M.A. Ray, O. Shenderova, W.M. Hooke, A. Martin, T. Tyler, G. Cunningham, V. Grishko, International Technology Center

An atmospheric pressure dielectric barrier glow discharge (APDBGD) plasma system has been developed at ITC. It was demonstrated that the APDBGD plasma system is a powerful tool for the surface functionalization of nanodiamond particulates. Nanodiamond functionalization was performed in minutes using plasma discharges generated with nitrogen and fluorine containing gases. The chemical bonds formed between reactive species generated in the plasma and nanodiamond surfaces were confirmed by FTIR and XPS analysis. Following plasma fluorination, XPS analysis revealed a relatively high concentration of F on the nanodiamond surface, up to 4-6wt%. Based on FTIR spectra analysis of several types of the initial ND produced by different vendors, it was demonstrated that plasma treatment of ND results in removal of particular surface groups (such as OH- and C=O), as well as in the formation of a variety of carbon-fluorine types of bonding (such as CF, CF@sub 3@(CF@sub 2@), C=CF@sub 2@) depending on the surface chemistry of the initial ND. Stable

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colloidal solutions of anisole with 0.5% by mass of nanodiamond were formed using these fluorinated nanodiamond particles, which are intrinsically hydrophilic and otherwise easily sediment in non-polar organic solvents. The degree of functionalization with a specific surface group depends on the surface chemistry of the initial nanodiamonds as well as on the type of gas used for plasma generation. Particularly, the use of CF<sub>4</sub> gas results in more efficient nanodiamond fluorination than SF<sub>6</sub>.

4:20pm **NS-ThA8 ZnCdSe Ternary Nanophosphors**, *H.-J. Lee*, University of Florida; *H. Yang*, Hongik University, South Korea; *S.Y. Seo*, *P.H. Holloway*, University of Florida

ZnCdSe quantum dots have been synthesized by a high temperature colloidal method using trioctylphosphine oxide (TOPO) solutions. Oleic acid complexes of Cd and Zn were used for the metal sources in reactions that produced air-stable nanoparticles of CdSe and ZnSe, respectively. Minimal alloying was achieved during a one-step synthesis process that led to a ZnSe shell on a CdSe core. Reaction temperatures below 250C led to less alloying as evidenced by spectral shifts. The kinetics of alloying were slower at 250C as compared to quantum dots synthesized at 320C. Smaller nanocrystals were obtained at the lower growth temperature due to a smaller critical radius of nucleation, leading to a large nucleation rate. ZnCdSe nanorods were also synthesized from CdSe/ZnSe coreshell nanorods via a solution thermal alloying process at 270~280C. CdSe nanorods were prepared using tetradecylphosphonic acid (TDPA)/TOPO surfactants, and a ZnSe shell was grown on CdSe nanorods at 180~190C. These nanorods were characterized as a function of alloying time using X-ray diffraction, Raman, and transmission electron microscopy. The kinetics of alloying and properties of ZnCdSe nanophosphors will be discussed.

4:40pm **NS-ThA9 Multiphysics Modeling of Electrochemical Structuring Using Complex Tool Electrodes**, *J.A. Kenney*, *G.S. Hwang*, University of Texas at Austin

Electrochemical machining using ultra-short voltage pulses has been used to modify electrically-active substrates at the micron length-scale without causing thermal damage or affecting crystal structure, unlike other machining technologies. However, this technique suffers from relatively slow processing speeds. In order to increase the speed, the use of complex tool 'template' electrodes has been suggested. Here we examine the relationship between pulse duration and feature separation on the resulting substrates using a transient charging simulation of the behavior of the pulse coupled with a feature profile evolution simulation employing the level set method.

## Surface Science

### Room 2004 - Session SS2+NS+TF-ThA

#### Tribology

**Moderator:** J. Krim, North Carolina State University

2:00pm **SS2+NS+TF-ThA1 Investigation of the Tribology of Diamondlike Carbon and SAMs using Molecular Dynamics**, *J. Harrison*, *P. Mikulski*, *G. Gao*, *J. Schall*, United States Naval Academy

INVITED

Examination of the Tribology of DLC and SAMs using Molecular Dynamics@footnote 1@ The development of micron-sized devices, such as microelectromechanical devices, for terrestrial and space applications has prompted the need for protection of the surfaces of these devices. Amorphous carbon films, diamondlike carbon, and self-assembled monolayers (SAMs) are all possible candidates for the passivation and lubrication of these devices. The fundamental problem associated with controlling friction is a lack of understanding of the underlying atomic-scale processes that govern both friction and wear. Over the past several years, we have performed extensive molecular dynamics simulations using the REBO@footnote 2@ and the AIREBO@footnote 3@ potentials aimed at understanding the atomic-scale mechanisms of friction in hydrocarbon systems. We have examined the contact forces present at the interface of a nominally flat, DLC tip and model alkane SAMs during sliding. We have also examined the effects of tip roughness on the contact friction. In addition, we have done simulations that have analyzed the tribological, mechanical, and transport properties of amorphous carbon films and diamondlike carbon films with various compositions. Some of our recent results will be discussed. @FootnoteText@ @footnote 1@ This work was supported by The Air Force Office of Scientific Research under contracts F1ATA04295G001 and F1ATA04295G002 (The Extreme Friction MURI) and by The Office of Naval Research (N00014-06-WX-20205).@footnote 2@ D.

W. Brenner, Shenderova, O. A., Harrison, J. A., Stuart, S. J., Ni, B., and Sinnott, S. B., *J. Phys. C* 14, 783 (2002).@footnote 3@ S. J. Stuart, Tutein, A. B., and Harrison, J. A., *J. Chem. Phys.* 112, 6472 (2000).

2:40pm **SS2+NS+TF-ThA3 Interfacial Force Microscopy of Viscous Water on Hydrophilic Surfaces**, *M.P. Goertz*, *R.C. Major*, *X.-Y. Zhu*, University of Minnesota; *J.E. Houston*, Sandia National Laboratories

The hydration of surfaces is important to many fields and its effect on system behavior has been studied for decades, yet the detailed origins of the forces involved are still under debate. We use interfacial force microscope (IFM) to measure the viscosity of water thin films with nanometer thickness on hydrophilic surfaces, including silica and carboxylic acid terminated alkanethiol self-assembled monolayers. We obtain the viscosity from this interfacial water from three different measurements: shear force measurement for a water meniscus formed between a tip and the hydrophilic surface under ambient conditions; shear force measurement for the interfaces under water; and repulsive, draining-force measurements of the two approaching interfaces immersed in water. In all three types of measurements, we obtain effective viscosities more than 10@super 6@ times greater than that of bulk water for interfacial separations on the nanometer scale. The experiments clearly show that the extent of the interphase layer and its level of viscosity depend sensitively on the strength of hydrophilic interaction, gradual degrading after the surfaces have been in water for a couple of hours. Thus, it is clear that a strong water/surface interaction gives rise to an increases in water-water bonding near the two surfaces and it is this increased level of bonding that is disturbed by the lateral tip motion resulting in the observed viscosity increase.

3:00pm **SS2+NS+TF-ThA4 Design of Environmentally Friendly Lubrication Systems through Surface Grafting of Ultrahydrophilic High-density Polymer Brushes**, *A. Takahara*, *M. Kobayashi*, Kyushu University, Japan; *K. Ishihara*, The University of Tokyo, Japan; *A. Suzuki*, *M. Kaido*, Toyota Motor Co., Ltd., Japan

The hydrophilic polymer brushes of poly(2,3-dihydroxypropyl methacrylate) (poly(2)) and poly(2-methacryloyloxyethyl phosphorylcholine) (poly(3)) were prepared by surface-initiated atom transfer radical polymerization of (2,2-dimethyl-1,3-dioxolan-4-yl)methyl methacrylate (1) and (3) on the initiator-immobilized silicon wafer. A large water contact angle hysteresis was observed for poly(2). Extremely low water contact angle and low contact angle hysteresis of poly(3) against water were observed. Neutron reflectivity measurements at water/poly(3) brush interface revealed that the poly(3) is highly stretched to the bulk water phase. Frictional properties of the poly(2) and (3) brushes were characterized by sliding a glass ball probe on the polymer brush surfaces in various solvents under the load of 0.49 N at a sliding velocity of 90 mm/min. Friction coefficient of poly(3) brush was lower than that of poly(2) in water. This can be ascribed to the presence of phosphorylcholine in poly(3). Further low friction coefficient was observed, even in air, by sliding a poly(3) brush immobilized glass ball against poly(3)-immobilized silicon wafer.

3:20pm **SS2+NS+TF-ThA5 Mechanical Properties of Alkanethiol Monolayers on Gold: A Force Spectroscopy Approach**, *G. Oncins*, *C. Vericat*, *F. Sanz*, University of Barcelona, Spain

Since the discovery of the self-assembling process of alkanethiol molecules on gold, these monolayers have been a matter of increasing interest not only from the point of view of basic surface science, but also due to their many applications in nanotechnology.@footnote 1@ Their mechanical properties have been widely studied by several techniques, especially by scanning probe microscopy.@footnote 2@ The growth mechanism of these monolayers on gold in vacuum has been followed by in situ Scanning Tunneling Microscopy (STM).@footnote 3@ Atomic Force Microscopy (AFM) has proved to be suitable to study the topography and mechanical properties of alkanethiol monolayers under compression.@footnote 4@ In this contribution, we used Force Spectroscopy to study the behavior of alkanethiol monolayers deposited on a (111) monocrystalline gold surface in an electrolyte aqueous solution. The analysis of the force curves shows that the tip indents the monolayer in a stepped way and that the force at which these stepped events have place depends on the compactness of the monolayer. Thanks to newly developed models to calculate the mechanical constants of monolayers on hard substrates,@footnote 5@ we were able to calculate the different values of Young's modulus for alkanethiol monolayers of different compactness and chain length. @FootnoteText@ @footnote 1@J. C. Love, L. A. Estroff, J. K. Kriebel, R. G. Nuzzo and G. M. Whitesides, *Chem. Rev.*, 2005, 105, 1103.@footnote 2@R.W. Carpick, M.

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Salmeron, Chem. Rev., 1997, 97, 1163. @footnote 3@G.E. Poirier, Langmuir, 1999, 15, 1167. @footnote 4@E. Barrena, C. Ocal, M. Salmeron, J. Chem. Phys., 2000, 113(6), 2413. @footnote 5@E.K. Dimitriadis, F. Horkay, J. Maresca, B. Kachar, R.S. Chadwick, Biophys. J., 2002, 82(5), 2798.

## 3:40pm SS2+NS+TF-ThA6 Observations of Microslip in Realistic Microscopic Contacts with Combined Nanoindentation and Quartz Microbalance, B. Borovsky, St. Olaf College; A. Booth, Grinnell College

For emerging technologies such as micromachined devices, it is increasingly important to understand the frictional properties of microscale sliding contacts and how these may be optimized with ultrathin lubricant films. While high sliding speeds and multiple contact points characterize realistic systems of interest, most theoretical and experimental studies of small contacts do not access this physical regime. We have therefore used a combined indenter probe and quartz crystal microbalance (QCM) to study the contact between a sapphire sphere and a polycrystalline gold electrode undergoing transverse shear at speeds near 1 m/s. The contacts were lubricated with monolayer octadecanethiol films. We find that both the elastic and dissipative components of the interaction are best described by the microslip model for reciprocating interfaces. We directly observe a 60% reduction in the tangential stiffness as the interface undergoes a spontaneous transition from stuck to slipping. For a mostly-slipping interface, the frequency and bandwidth shifts of the QCM are proportional to each other and track changes in the contact radius, as derived from the normal contact stiffness. Furthermore, we observe shear loss tangents over 0.4, more than 100 times larger than expected for no-slip conditions. We will show that our results are consistent with force-equilibrium microslip theory @footnote 1,2@ by proposing a simplified dynamic model of hysteresis effects in microslip, based on the driven harmonic oscillator. Research supported by NSF, Research Corporation, and Hysitron, Inc. @FootnoteText@ @footnote 1@R. D. Mindlin, W. P. Mason, T. F. Osmer, and H. Deresiewicz, Proceedings of the First U. S. National Congress of Applied Mechanics, 1951, pp. 203-208. @footnote 2@K. L. Johnson, Contact Mechanics, Cambridge University Press, NY, 1985, pp. 216-230.

## 4:20pm SS2+NS+TF-ThA8 The Effect of Filling and Temperature on the Mechanical Responses of Carbon Nanotubes, S.-J. Heo, S.B. Sinnott, University of Florida

It is well known that carbon nanotubes (CNTs) have fascinating electrical, optical, chemical, and mechanical properties that differ from the properties of macroscale carbon materials such as graphite or diamond. As a result of these properties, CNTs are being considered as candidate materials for MicroElectroMechanical System (MEMS)/NanoElectroMechanical System (NEMS) components. It is therefore worthwhile to study the mechanical behavior of CNTs to better understand how they might fit in with the mechanical property requirements of MEMS/NEMS. To facilitate this better understanding, we have explored two different mechanical responses of CNTs, to bending and compression, using classical molecular dynamics simulations. The second generation reactive bond order potential is used to model the short-range covalent interactions and a Lennard-Jones potential is used to model the long-range van der Waals interactions. In particular, we have modeled a three-point bend test to explore the mechanical responses of the single walls CNTs, single-walled CNTs filled with C60, double-walled CNTs, and triple-walled CNTs. A compression test has also been done on these same systems. Filling the single-walled CNTs, or increasing the number of inner shells in the case of multi-walled CNTs, is predicted to increase both the bending strength and the maximum buckling force. We have also investigated the effect of temperature on the mechanical responses of the CNTs. On the whole, higher temperatures are predicted to lower the bending strength of the CNTs. This work is supported by the National Science Foundation funded Network for Computational Nanotechnology (EEC-02288390).

## 4:40pm SS2+NS+TF-ThA9 Assessing nanomechanical properties and nanoscratch resistance of Me-ZrN and ZrN thin films using Atomic Force Microscope, D.M. Mihut, J. Li, S.R. Kirkpatrick, University of Nebraska-Lincoln; S. Aouadi, Southern Illinois University; S.L. Rohde, University of Nebraska-Lincoln

The present study aims at getting a better understanding of the correlation between nanomechanical properties (nanohardness (H), elastic modulus (E), H/E and H<sup>3</sup>/E<sup>2</sup> ratio) and nanotribological properties (resulting from nanoscratch measurements) for three groups of Me-ZrN thin films (Inconel-ZrN, Cr-ZrN and Nb-ZrN) and ZrN thin films. Nanomechanical and nanotribological properties for Me-ZrN and ZrN thin films deposited by DC unbalanced magnetron sputtering were investigated using atomic force microscope (AFM) interfaced with a Hysitron Triboscope. The elastic

recovery of thin films under a normal load applied during nanoindentation was evaluated and correlated with elastic recovery of thin films under a dynamic load applied during nanoscratch measurements and with other mechanical properties in order to predict the thin film composition that will better serve for wear resistant applications.



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## Nanometer-scale Science and Technology Room 3rd Floor Lobby - Session NS-ThP

### Nanoscale Science and Technology Poster Session

#### NS-ThP1 Scanning Tunneling Microscopy of Dinuclear Metal-Complex Molecules, *S. Guo, Z.Q. Wei, S.A. Kandel*, University of Notre Dame

Scanning tunneling microscopy is employed to study the adsorption of dinuclear metal-complex molecules on a variety of substrates under ultra-high-vacuum conditions. These investigations are focused on the applicability of such molecules in quantum cellular automata-based molecular devices. These large molecules can show surprisingly strong interactions with the underlying substrate, leading to stable binding at room temperature and, in some cases, molecular orientation commensurate with the surface lattice. Direct molecular manipulation using the STM tip provides additional information about stability and binding, as well as indicating the feasibility of simple molecular device assembly.

#### NS-ThP5 Electronic Conduction of the Titanium Silicide Nanowires Grown on Si(111) Surface, *R. Stiefel, T. Soubiron, B. Grandier, D. Deresmes, L. Patout, D. Stievenard*, Institut d'Electronique, de Microelectronique et de Nanotechnologie, France

Scanning tunneling microscopy (STM) and scanning tunneling spectroscopy (STS) are two experimental techniques very suitable for the study of fundamental mechanism of electronic transport in low dimensional systems such as nanowires. Due to their unusual physical properties the nanowires represent a promising candidate for future nano devices. In order to achieve this goal, the understanding of their unusual quantum properties is necessary. Using STM/STS techniques we have investigated the electronic transport of the self-assembled titanium silicide nanowires in ultrahigh vacuum conditions in the 77 - 4 K temperature range. By varying the temperature we observed a transition from a semiconductor behavior at 4 K, characterized by the presence of a well defined energy band gap, to a metallic one at 77 K where the band gap vanishes. We explain this change by the different role of the silicon surface states in the charge transfer mechanism of the system formed by TiSi<sub>2</sub> nanowires and the silicon substrate. The semiconductor behavior observed at low temperatures is a consequence of the Schottky barrier formation at the interface between the nanowires and the silicon substrate, when the silicon surface states are frozen.

#### NS-ThP6 Silver Nanowires Grown on Titania Films by Thermal or Photo Induced Reduction, *C.Y. Huang*, National Dong Hwa University, Taiwan, R.O.C.; *T.K. Chen*, National Dong Hwa University, Taiwan, R.O.C., Taiwan, ROC; *H.Y. Tang*, National Dong Hwa University, Taiwan, R.O.C.; *M.S. Wong*, National Dong Hwa University, Taiwan, R.O.C., ROC

Silver nanowires were synthesized simply by thermal or photo reduction of silver nitrate solution on anatase TiO<sub>2</sub> films without any capping and reduction agent. The silver nanowires were 1-50  $\mu\text{m}$  in length and 20-150 nm in diameter. Several parameters including reduction temperature, light energy and intensity, reduction time and the concentration and amount of the silver nitrate solution affected the formation of silver nanostructures. Scanning electron microscopy, transmission electron microscopy, X-Ray diffraction, and UV-Visible spectroscopy were used to characterize the silver nanostructures. The results provided a better understanding of the growth mechanism of silver nanowires formed by this simple route.

#### NS-ThP7 Nano-Pocket Array for Protein Immobilization, *K.J. Kim, W.-J. Kim, D.H. Ha, S.I. Park*, Korea Research Institute of Standards and Science; *W.S. Yun*, Korea Research Institute of Standards and Science, Korea; *H.J. Kang, S.J. Chung*, Korea Research Institute of Bioscience and Biotechnology

We report on the preparation of nanoscale pockets on a solid surface for protein immobilization. The nano-pockets were fabricated by e-beam lithography followed by reactive ion etching. Diameter and depth of the nano-pockets were both in the range of 30-150 nm. For some samples, thin gold layer was formed at the bottom of the pocket. The nano-pocket was first treated by either aminoalkylsilane or aminoalkylthiol depending on the material in the pocket. The pocket was then treated by succinic anhydride, NTA (Nitriloacetic acid), and Ni<sup>2+</sup>. His-tagged proteins such as GFP (green fluorescence protein) and PTP (protein tyrosine phosphatase) were then immobilized in the pocket by Ni-NTA. The fluorescence intensity of GFP and

the catalytic activity of PTP were measured at intervals of a day to examine the effect of the nano-pocket on the protein stability.

#### NS-ThP8 Imaging and Polarization Dynamics in Ultrathin Ferroelectric PVDF Copolymers by Piezoresponse Force Microscopy, *B.J. Rodriguez, S. Jesse*, Oak Ridge National Laboratory; *J. Kim, S. Ducharme*, University of Nebraska; *S.V. Kalinin*, Oak Ridge National Laboratory

Ferroelectric polyvinylidene fluoride (PVDF) is widely used as a piezoelectric material because of its outstanding electromechanical properties. High-quality monolayer-thin films of PVDF and its copolymers are fabricated using a Langmuir-Blodgett technique, enabling applications such as all-polymer field effect transistors, polymer ferroelectric random access memories, and flexible ferroelectric electronic components. In order to realize the full potential of PVDF, significant progress must be made in nanoscale characterization of the structure of PVDF films. Here, the local structure, electromechanical activity, and polarization switching of ultrathin ferroelectric films of PVDF copolymers are studied by piezoresponse force microscopy (PFM). PFM imaging of PVDF thin films reveals ferroelectric domain sizes of less than 40 nm at a resolution below 5 nm. The combination of vertical and lateral PFM data reveals that the polar axes of the molecules are not orthogonal to the substrate. Local hysteresis loop measurements and local domain writing are performed to establish the potential of PVDF films for data storage and electronic applications. Unlike traditional ferroelectric thin films, PVDF exhibits switching on the order of milliseconds, which is attributed to a fundamental difference in the switching mechanism as compared to perovskite ferroelectrics. In PVDF, a significant change in molecular geometry, as opposed to dipole switching, is required to change the polarization orientation. To address the dynamic polarization behavior in PVDF, spatially resolved mapping of piezoelectric response is performed using switching spectroscopy PFM (SS-PFM). Lastly, the potential advantages of liquid PFM to minimize surface damage are discussed. These results provide a complementary view of structure, ferroelectric properties, and domain dynamics in PVDF on the nanoscale.

#### NS-ThP9 Effects of Deforming of Carbon Nanotubes (CNTs) on Electrical Conductivities, *Z. Xiao, M. Saafi*, Alabama A&M University; *W. Li*, GE Global Research Center

Dielectrophoresis (DEP) method was used to deposit, align, and assemble carbon nanotubes (CNTs) to bridge cross a pair of electrodes. Highly-purified HiPCO-grown single-walled carbon nanotubes (CNTs) from Carbon Nanotechnologies, Inc. (CNI) were used for the investigation. The Single-walled carbon nanotubes were ultrasonically dispersed in toluene, sodium dodecyl sulfate (SDS), and DI water. The CNT-containing solution was then dropped onto a pair of electrodes, which were patterned on a polyimide thin film coated on the oxidized silicon substrate, and an ac voltage was applied to the electrodes for generating an inhomogeneous electric field to attract CNTs onto the electrodes. Photolithography and e-beam lithography and lift-off techniques were used to fabricate the pair of electrodes, which are opposite to each other and has a gap of 1  $\mu\text{m}$  or 3  $\mu\text{m}$ . Each electrode is 50  $\mu\text{m}$  long and 1  $\mu\text{m}$  or 3  $\mu\text{m}$  wide. The flexible polyimide thin film was coated on the substrate with a spin coater using PI 2525 from HD Microsystems, and had a thickness of about 10  $\mu\text{m}$ . The polyimide film, together with the fabricated device on it, was finally stripped off from the substrate for the measurement of electrical conductivities versus the bending of polyimide thin films, which resulted in the deforming of CNTs. The current-voltage characteristics of CNTs depend on the form of CNTs [Z. Yao, et al, "Carbon nanotube intramolecular junctions", Nature 402, 273-276 (1999)]. The fabricated CNT Devices and the effects of deforming of carbon nanotubes (CNTs) on the electrical conductivities will be reported in the conference.

#### NS-ThP10 Fabrication of Suspended Single-Walled Carbon Nanotube on Vertically-Aligned Carbon Nanofiber Templates and Electrical Characteristics, *Z.G. Wu, C.H. Weng, H. Lin, W.Y. Lee, C.H. Tsai, K.C. Leou*, National Tsing Hua University, ROC

We have developed a new method to directly synthesize suspended carbon nanotubes (su-SWNTs) crossing the tips of vertically-aligned carbon nanofibers (CNFs) which were sharpened by energetic argon plasma. Via this approach, here we demonstrate the fabrication of electronic devices of novel su-SWNTs-on-CNFs structure and investigate the nano-sized contact between su-SWNT and CNFs as well as the electrical characteristics. Highly n-doped (phosphorous) poly-Si, due to its similarity to p-type single-crystalline Si substrate we used before, is demonstrated a good choice of under-layered electrodes for the fabrication of su-SWNTs-on-CNFs electronic devices. The electrical

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characteristics, including two (source/drain), three (source/drain/bulk-gate), and four (source/drain/bulk-gate/local-gate) terminal measurements reveal the electronic device features, such as hysteresis-free electrical transport, negative differential conductance, etc. Furthermore, we use multiple nano-probes to exert electrostatic forces, and in-situ observe the deformation of su-SWNTs in a scanning electron microscope to investigate the contact between su-SWNT and CNFs. @FootnoteText@ @footnote 1@C. H. Weng, W. Y. Lee, Z. Y. Juang, K. C. Leou, and C. H. Tsai, *Nanotechnology* 17, 1 (2006).

**NS-ThP11 Cutting of Multiwalled Carbon Nanotube Tip of Atomic Force Microscope, D.-H. Kim**, KRIS, Korea; *K.Y. Jung*, NanoFocus Inc., Korea; *J. Choi, S.J. Ahn, B.C. Park*, KRIS, Korea

We demonstrate cutting a multi-walled carbon nanotube attached on an atomic force microscope tip by flowing direct current through the tube, as a method to precisely control nanotube tip length. Mechanical contact was made at the target cutting position between a sharp metal wire (electrode 1) and a nanotube on silicon mother tip (electrode 2), and then a voltage was applied to electrode 1 until a current flowed through and cut the nanotube. Nanotubes were reproducibly cut at the contact point with the wire, and as the results we could achieve 30 nm as the cutting precision, and 100 nm as the shortest nanotube protrusion. We found that the process happened at either current ranges: higher than 100  $\mu$ A and as small as 1  $\mu$ A. High current-process happens fast and is attributed to unimolecular decomposition reaction, while small current-process is a slow chemical etching, being activated by the current.

**NS-ThP12 Diffusion Characteristics of Gases through Y-shaped Carbon Nanotubes using Molecular Dynamics Simulations, J. Myers, S.-J. Heo, S.B. Sinnott**, University of Florida

In the modern pharmaceutical and chemical industries, solutions of extremely high purity are needed. Current filtration methods are reaching the limits of their abilities, so new filters must be developed. One possible filter is a Y-shaped carbon nanotube (Y-tube). By changing the sizes of the arms of the Y-tube, custom molecular filtration is theoretically possible. Through the use of classical molecular dynamics simulations, these hypotheses will be tested. These particular simulations use Y-tubes of different sizes and chiralities to determine the effect of these factors on gaseous diffusion and separation. Gas reservoirs of methane and an isobutane/methane mix are allowed to diffuse through each Y-tube. In the case of the isobutane/methane reservoir, it is hypothesized that the larger molecule, isobutane, will not diffuse down the smallest arm. The overall goal of this research is to gain a fundamental understanding of diffusion through Y-shaped carbon nanotubes to support and guide future studies. This research is supported by a National Science Foundation grant through the Network for Computational Nanotechnology (grant no. EEC-02288390).

**NS-ThP13 Conductance Plateau Length of Au Single-Atom Contacts, R. Suzuki, S. Kurokawa, A. Sakai**, Kyoto University, Japan

We have measured the length distribution of  $1G@sub 0@$  ( $G@sub 0@$  is the quantum unit of conductance) plateaus of Au single-atom contacts in UHV at room temperature. It is found that the distribution has a slowly decaying tail and can be well fitted by a power-law behavior. The exponent little depends on the bias and takes a value close to -1. Thus, the  $1G0$  plateau length, which is the lifetime of Au single-atom contacts, obeys the so-called Zip's law. We also found that the average plateau length decreases with increasing the bias but increases when measured in air. Probably, gas adsorptions on Au single-atom contacts in air act to improve their stability.

**NS-ThP14 Self-Organized Growth of Magic Planar Ag Nanopucks, Y.P. Chiu**, Academia Sinica and National Sun Yat-Sen University, Taiwan, Taiwan, ROC; *Y.S. Ou, Y.R. Chang*, Academia Sinica and National Taiwan University; *C.M. Wei, C.S. Chang, T.T. Tsong*, Academia Sinica, Taiwan

Detailed calculations based on ab initio density functional theory have been made to study the formation of two-dimensional metal nanostructures. An analysis with metal nanostructures arranged in different shapes for a chosen size has been made to investigate how the size and shape effects related to electronic confinement influence the stability. Furthermore, based on the calculations of the second differences in total energy, it is shown that for Ag and Au two-dimensional geometries can exist in free space. In the report, the noble self-organized growth of magic planar Ag nanopucks on Pb islands has been observed in experiment by STM. According to the binding energy per atom as function of the nanopuck size, it clearly indicates that when the Ag nanopuck grows to a certain size, the geometrical effect takes hold from the electronic effect as

the major attribute, which drives the Ag nanopucks towards well defined hexagonal crystalline structures. The transition takes place between around 30 and 60 atoms.

**NS-ThP15 The Effect of Relative Humidity on Atomic Force Microscopy Local Oxidation of Silicon Nitride Film for Mask Fabrication, H.F. Hsu, C.W. Lee**, National Chung Hsing University, Taiwan, R.O.C.

Atomic force microscope (AFM) induced local oxidation on silicon nitride thin film of silicon substrate which is in contact mode is presented. This local anodic oxidation was subjected to its kinetics and mechanism in varying relative humidity. The kinetic results are observed to have a logarithmic relationship of oxide height versus voltage pulse duration [ $h=L@sub c@ln(t/t@sub 0@)$ ] and an exponential decay relationship of the growth rate to oxide height [ $dh/dt=R@sub 0@exp(-h/L@sub c@)$ ]. Both the onset time  $t@sub 0@$  and the characteristic decay length  $L@sub c@$  increase during experiments at lower relative humidity and the lateral oxidation growth rate decreases. As the result, we can fabricate the high-aspect-ratio of the oxide by controlling the ambient humidity. Due to the large etching selectivity in various etchants between  $Si@sub 3@N@sub 4@$ ,  $SiO@sub 2@$  and Si, an AFM patterned silicon nitride film mask with different size open windows can be made to fabricate nanoscale structure on silicon substrate.

**NS-ThP16 Evaluation of Pattern Profiles Replicated by Nanoimprint Using AFM with Carbon Nanotube Tip, J. Igaki, K. Nakamatsu**, University of Hyogo, Japan; *K. Tone*, Meisyo Co., Japan; *T. Nishimura*, SII NanoTechnology Inc., Japan; *S. Matsui*, University of Hyogo, Japan

Nanoimprint lithography (NIL) is a very useful technique to make nanostructure devices with low cost and high throughput. A various resist polymers such as PMMA are used as replicated materials for NIL. The observation of replicated pattern profile is usually carried out after cleaving a pattern by scanning electron microscopy (SEM). However, it is difficult to use this method to accurately position the cleavage for a cross-section of a specific pattern with nano dimensions. In contrast, atomic force microscope (AFM) evaluation enables non-destructive measurement without contamination or irradiation damage inevitable in SEM evaluation. Therefore, evaluation of replicated pattern using AFM has potential advantages. In this paper, we evaluated profiles of  $SiO_2$  mold (200 nm-thick  $SiO_2$  on Si) and PMMA replicated pattern on a Si substrate by AFM using carbon nanotube (CNT) as a probe. By using carbon nanotube as a probe, it enables obtaining a high resolution and high aspect pattern profile. First, a  $SiO_2$  pattern as a thermal NIL mold was fabricated by EB lithography and dry etching process. Next, PMMA patterns were replicated by thermal NIL process using  $SiO_2$  mold.  $SiO_2$  mold was pressed into PMMA layer at 120°C at a set press-pressure of 20 MPa. Finally, the shapes of  $SiO_2$  mold and PMMA replicated pattern were evaluated by AFM with CNT tip (L-trace, SII NanoTechnology Inc.). The pitch, line width, height and angle of gradient of the  $SiO_2$  mold were 600 nm, 221 nm, 198 nm and 82°, respectively. The pitch, line width, height and angle of gradient of the PMMA replicated pattern pressed at 120 °C were 604 nm, 226 nm, 194 nm and 82°, respectively. In this way, the shapes of  $SiO_2$  mold and the PMMA replicated pattern were well evaluated by AFM with CNT tip.

**NS-ThP17 Nanoimprint Mold Repair by Focused-Ion-Beam Etching and Deposition, M. Okada, K. Nakamatsu, S. Matsui**, University of Hyogo, Japan

Nanoimprint lithography (NIL) in recent years is beginning to attract much attention from many industrial fields because of the advantages to provide various nanostructure devices with a high-throughput and low cost. In particular, mold is the key element in imprinting because the mold patterns transferred directly in replication materials. Therefore, repair techniques are required for the NIL mold. However, there are only a few papers reporting on the repairing of the NIL mold. In this paper, we demonstrate repair of a NIL mold with defects of protrusions and hollows by focused-ion-beam (FIB) technology. A beneficial advantage in use of FIB technology is that mask repair can be achieved with high-resolution because of the fine diameter of FIB of 5 nm. We performed repairing of a defective NIL mold using a commercial FIB apparatus with  $Ga^+$  ion-beam operating at 30 kV. Defects of protrusions contained in NIL mold were successfully repaired by FIB etching. Hollow defects of NIL mold were also repaired by FIB chemical vapor deposition (CVD) using  $C_{14}H_{10}$  gas as a precursor. Diamond-like carbon thin layer deposited by FIB-CVD plugged the hollow defects. The mold patterns repaired by FIB etching and CVD were successfully imprinted in PMMA resin on a Si substrate, and then the PMMA imprinted patterns were transferred into Si substrate by  $CF_4$  reactive-ion-etching while the patterns were used as the etching mask. The results of repairing a defective

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NIL mold, and replicated patterns imprinted using the repaired mold will be presented at the conference.

**NS-ThP18 Mechanical Characteristics of Metal-Containing Nanosprings Fabricated by Combination of FIB-CVD and Sputter Coating.** *K. Nakamatsu, K. Kanda, Y. Haruyama*, University of Hyogo, Japan; *T. Kaito*, SII NanoTechnology, Japan; *S. Matsui*, University of Hyogo, Japan

Three-dimensional (3D) nanoscale components such as spring structures are necessary to build a high performance nanoelectromechanical system. To form the 3D nanostructures, we have proposed the use of focused-ion-beam chemical vapor deposition (FIB-CVD). FIB-CVD promises to generate various 3D nanostructures, and has an efficient capability to provide them of various materials. In our previous experiments, diamond-like carbon (DLC) nanosprings were fabricated by FIB-CVD. The DLC nanosprings showed a unique property to expand and contract as flexibly as macro-scale springs. However, the DLC springs lack electrical characteristics because of the high resistivity. In this paper, we investigate mechanical properties of sputter-coated metal FIB-CVD nanosprings. Metal-containing springs are useful for the nanoelectrical applications due to its low resistivity. Although metal-containing springs can be grown by FIB-CVD, there is a serious problem that undesired materials such as C and O are also incorporated. Therefore, we propose here the combination of FIB-CVD and metal sputtering to fabricate the pure metal nanosprings. The fabrication is as follows. (1) A DLC nanospring was fabricated by FIB-CVD using C14H10 gas as a precursor. (2) O<sub>2</sub> reactive-ion-etching was performed to remove a large amount carbon formed on a core region of Ga. Only the core region of Ga without deformation of a coil structure remained after this process. (3) Metal was coated onto the nanospring by sputter coating. We selected several materials as the metal for the sputter coating on the nanosprings. Metal thin layer was successfully coated onto a nanospring by sputter coating. The metal-coated nanosprings were able to expand and contract mechanically. A spring constant of the metal sputter coated nanosprings were able to be obtained from Hooke's law. The mechanical characteristics of the several kinds of sputter-coated metal nanosprings will be presented at the conference.

**NS-ThP19 Development of the Integrated Manipulation System using 3-D Nano-Manipulator Fabricated by the Focused-Ion-Beam Chemical-Vapor-Deposition.** *R. Kometani, K. Kanda, Y. Haruyama*, University of Hyogo, Japan; *T. Kaito*, SII NanoTechnology Inc., Japan; *S. Matsui*, University of Hyogo, Japan

Nano-factory is necessary to carry out the manipulation, manufacturing and analysis by high-accuracy. For the nano-factory creation, the high-performance nano-tools such as manipulator, emitter, sensor and so on are required. Therefore, we have been researching the nano-tool with the three-dimensional (3-D) structure by using the focused-ion-beam chemical-vapor-deposition (FIB-CVD) for giving the high-performance to the nano-tools. FIB-CVD is the key technology for 3-D nano-structure fabrication. This time, we established the integrated manipulation system by using 3-D nano-manipulator fabricated on the tungsten probe by FIB-CVD. 3-D nano-manipulator have 4-fingers, made of the diamond-like carbon, for catching the target with various structures flexibly. Gap distance of fingers was about 500 nm. Driving force of the 3-D nano-manipulator is the repulsive force. So the fingers of the 3-D nano-manipulator can open by applying voltage from the tungsten probe to fingers. It was possible to control the finger opening of the 3-D nano-manipulator with about 0.5 nm/V. And a tungsten probe as a monopole electrode was connected with a commercial piezo manipulator to control the position of the 3-D nano-manipulator. This result indicates that highly accurate control of the 3-D nano-manipulator can be carried out in the nano-factory. Furthermore, the manipulation of the glass ring as nano-parts was succeeded by using integrated manipulation system having the 3-D nano-manipulator in the vacuum chamber of FIB-CVD-system. These results indicate that our integrated manipulation system having the 3-D nano-manipulator is very useful tool to use in the nano-factory.

**NS-ThP20 Controlled Growth of Nanostructured Complex Oxides by Pulsed Laser Deposition Nanostenciling.** *C.V. Cojocaru, O. Gautreau, C. Harnagea, F. Rosei, A. Pignolet*, Université du Québec, Canada

We have investigated an alternative approach to conventional patterning, namely nanostenciling. Along with its suitability for direct patterning of complex materials, we demonstrated its unique flexibility in combining different functional material films on various substrates. Among functional materials, the complex oxides family displays a range of interesting properties and useful response to various stimuli such as electric, magnetic, and stress fields. These properties, including piezoelectricity,

ferroelectricity or high-temperature superconductivity have been shown to be directly related to the structural quality and thus to the details of the fabrication processes of the grown thin films. We will report how various features drawn in miniature microfabricated masks (nanostencils) can be successfully transferred directly to the surface in the form of nanostructures of functional complex oxides (e.g. SrRuO<sub>3</sub>, BaTiO<sub>3</sub>, BiFeO<sub>3</sub>) by pulsed-laser deposition (PLD), and will discuss in detail the advantages of the technique. The process is rapid, resist-less and does not interfere with the structures natural growth dynamics. On one hand, this approach allows organizing the structures in the desired architectures; on the other it conserves their individual functionality (e.g. ferroelectricity at the nanoscale proven by piezoresponse force microscopy). We will also discuss the application of a nanoscale multi-level-stenciling approach that gives the opportunity for rapid prototyping of functional heterostructures composed of stacks of different materials (e.g. metal-oxide-metal and multiferroic structures grown by sequential depositions through nanostencils). This new patterning technique is extremely attractive for research (e.g. investigation of size effects on the functional properties of small structures of various materials) but also shows a great potential for the parallel fabrication and patterning of a large variety of materials.

**NS-ThP22 Evaluation of Immobilization Techniques on the Performance of Nanocrystalline TiO<sub>2</sub>-Polymer Composite Films.** *E. Cruz*, Sandia National Laboratories - Lawrence Berkeley National Laboratory, ALS division; *B.A. Simmons*, Sandia National Laboratories

Titanium dioxide (TiO<sub>2</sub>) has been used extensively over the years as a very effective photocatalyst capable of purifying water from organic contaminants. In a slurry of free particles and liquids, however, it is complex and costly to remove the titanium dioxide from the purified water effectively. This study investigates the immobilization of titanium dioxide on a variety of robust mesh structures with numerous techniques that can be used repeatedly for water purification operations. Various deposition methods and substrate combinations are presented and evaluated in terms of degradation efficiency of a mock pollutant. Scanning electron microscope (SEM) micrographs reveal the morphological characteristics of the TiO<sub>2</sub>-matrix support and the nature of the solid-liquid interface. Mechanical stability and robustness of the TiO<sub>2</sub> support structures will also be discussed as a function of lifetime and durability. In our experiments, a flow-thru cell is used to contain the TiO<sub>2</sub>-matrix support. A pump-manifold system is then utilized to introduce the mock contaminated water that contains the dye methyl orange. This flow thru cell is irradiated at 300-nm, 350-nm and 419-nm wavelengths at different time intervals with different incident fluxes of light. Methyl orange degradation is evaluated using UV-Vis spectroscopy as a measure of the overall photocatalytic activity of the immobilized TiO<sub>2</sub>. We have also investigated the impact of doped TiO<sub>2</sub> in terms of activity and wavelength dependence. The immobilized TiO<sub>2</sub> films were observed to be very efficient decontamination substrates.

**NS-ThP23 Synthesis and Optical Properties of Indium Oxide Nanocrystal Chains Grown by Thermal Oxidation Method.** *T.S. Ko, C.P. Chu, H.G. Chen, T.C. Lu, H.C. Kuo, S.C. Wang*, National Chiao Tung University, Taiwan

We reported the synthesis of indium oxide nanocrystal chains on silicon substrates by using thermal evaporation of pure indium at 0.1 torr of nitrogen atmosphere. Indium nanocrystal chains were grown through vapor-liquid-solid process by using gold nanoparticles as catalyst on native silicon dioxide. The composition of the nanocrystal was identified by energy dispersion spectrum and the result showed the existence of indium oxide compound. Scanning electron microscopy investigations showed indium oxide nanocrystal chains were formed. The nanocrystal chains comprised many well-shaped octahedron nanoparticles with their size ranging from 20 nm to 100 nm connected each other to form a network. Both high resolution transmission electron microscopy and diffraction pattern revealed that the indium oxide nanocrystal chains were single crystalline grown along [111] direction. Photoluminescence study of indium oxide nanocrystal chains exhibited main photoemission at 544 nm. Such self-assembled nanocrystal chains should have great potential for applications of novel optoelectronic devices and nano-photonics.

**NS-ThP24 Evaluation of Resolution for Free-Space-Wiring Fabricated by FIB-CVD.** *C. Minari, R. Kometani, K. Nakamatsu, K. Kanda, Y. Haruyama*, University of Hyogo, Japan; *T. Kaito*, SII NanoTechnology, Japan; *S. Matsui*, University of Hyogo, Japan

Focused-ion-beam chemical-vapor-deposition (FIB-CVD) is the effective technology to fabricate 3-D nanostructure. FIB-CVD is promising in many fields such as mechanical device, optical device and bio device. Thus it is

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very important to refine FIB-CVD as 3-D nanostructure fabrication technology. This time, we examined the growth mechanism by FIB-CVD. We fabricated various line structures by changing a scan speed and an ion beam current with a single scanning of Ga<sup>+</sup> ion beam. Acceleration voltage was 30kV, and phenanthrene (C<sub>10</sub>H<sub>14</sub>) was used as a source gas to deposit Diamond Like Carbon (DLC). First, we examined the resolution of DLC line patterns on Si substrate. It was observed that DLC linewidth became smaller, as Ga<sup>+</sup> ion beam scan speed quickened. This is attributed to the Gaussian profile of Ga<sup>+</sup> ion beam. The tip part of beam profile is effective to deposit at faster scan speed, on the other hand, at slower scan speed, both the tip and tail parts of beam profile are contributed. A 30nm linewidth was achieved on Si substrate at the scan speed of 0.8μm/s with a 0.1pA beam current (calculated beam diameter: 5nm). Next, we evaluated resolution of DLC free-space-wiring grown from the top of DLC wall made by FIB-CVD. A DLC line grows in space by controlling the scan speed, and performed as a free-space-wiring. When the beam scans slowly at a scan speed of 0.05μm/s using a beam current of 0.4pA (calculated beam diameter: 10nm), a free-space-wiring grows up from the top of DLC wall. In contrast, when the beam scans quickly at the scan speed of 0.07μm/s, a free-space-wiring grows downward from the top of DLC wall. In this experiment, the minimum linewidth of downward free-space-wiring was about 50nm and that of upward free-space-wiring was about 100nm.

**NS-Thp25 Annealing Effect of Electronic Properties for Tungsten Wires Fabricated by FIB and EB-CVD, A. Ozasa, K. Nakamatsu, R. Kometani, K. Kanda, Y. Haruyama, University of Hyogo, Japan; T. Kaito, SII NanoTechnology Inc.; S. Matsui, University of Hyogo, Japan**

We present the temperature dependence of electrical properties for tungsten wires fabricated on four terminal Au electrodes by 30 kV FIB-CVD and 5 kV EB-CVD using W(CO)<sub>6</sub> source gas. The gap of center electrodes is 1μm. The tungsten wires has 4.8-μm-long and 300-nm linewidth. The tungsten wires fabricated by FIB-CVD contains Ga that is implanted by Ga ion beam irradiation. The tungsten wires deposited by FIB-CVD contain about 86% C, 0.6% O, 13% Ga and 0.3% W, which were measured by SEM-EDX. On the other hand, the deposited tungsten wires by EB-CVD contain about 96% C, 3% O and 1% W. We measured the temperature dependences of the resistances of the wires deposited by EB and FIB-CVD with a four-terminal method at 300-673 K. The measurement results indicate that the resistance decreased by annealing temperature raised for all wires deposited by FIB and EB-CVD. The resistance of tungsten wires deposited by FIB-CVD flatly decreased from 2.7 @ohm@ cm to 1.0 @ohm@ cm with increasing annealing temperature from 300 K to 673 K. On the other hand, the result of tungsten wires deposited by EB-CVD dramatically decreased from 144 k@ohm@ cm to 3.2 k@ohm@ cm with annealing temperature rise from 300 K to 673 K. Moreover, we examined in detail I-V measurement for both FIB and EB-CVD wires. As a result, non-linear I-V characteristics were observed for FIB and EB-CVD annealed wires. Especially, EB-CVD wires annealed over 673 K indicates significant semiconductor I-V characteristics.

**NS-Thp26 Topographical Studies of Nanoscale Secondary Structure of Electrochemically Anodized Aluminum Surface, T. Sugiyama, H. Kato, S. Takemura, Y. Watanabe, H. Matsunami, Y. Takarai, M. Izumiya, A. Ishii, T. Hiramatsu, N. Nanba, Kanto Gakuin University, Japan; O. Nishikawa, M. Taniguchi, Kanazawa Institute of Technology, Japan**

An electrochemical anodization technique was applied for fabrication of surface nanoscale structure on Al. Sub-micron to nanometer scale surface structure is a prospective candidate for highly functional nanometer scale devices, especially in the field of inorganic-organic hybrid devices and molecular devices as a nano-pattern template. In the present work, the surface was cleaned with several stages of wet cleansing process using Semico Clean and pure water under ultrasonic waves before applying anodization techniques to Al plate. The process of anodization was conducted by applying a positive voltage to Al plate as an anode in a H<sub>2</sub>SO<sub>4</sub> solution with various concentration. The anodized Al and Cu surfaces processed under different conditions were investigated by dynamical force microscopy (DFM) and scanning tunneling microscopy (STM). A crater-array pattern was observed in a flat area. The diameter size of the crater was 100 nm to 200 nm, linked each other making a scaled pattern with the clear-cut contour, covering wide range area with 5 micrometer square. In the scaled pattern, the linked craters covered up-and-down hills with several nanometer height difference. Focusing on a crater, small crater with several tens nanometer diameter was created in the large crater. A detailed image of Al surface of another sample prepared under a different condition showed the nanoscale secondary structure in the crater-array pattern so that 20 nm to 30 nm-sized holes were arrayed in a 100 nm-sized

crater, which is quite a unique pattern as a nanoscale template. Furthermore, copper phthalocyanine (PcCu) molecular layer deposition was conducted on scaled pattern of Al surface by wet and dry techniques. Characteristic PcCu configuration was confirmed by DFM, XPS, and FTIR measurements. This work was supported by High-Tech Research Center Project aided by MEXT. The authors would like to thank Dr. M. Shigeno, SII Nano-Technology Co. Ltd for a technical assistance.

**NS-Thp27 Etching of Ion-implanted Silicon Nitride and Oxide in a Weakly Anisotropic Dry Etch Process, P.K. Subramanian, AMD Inc.; S. Panda, M.P. Belyansky, IBM Microelectronics**

Etching silicon nitride films deposited over other (e.g., silicon oxide) films is frequently used in different process schemes in the semiconductor industry. Thus, etch selectivity and throughput of the etch process are important factors. Two commonly available unit processes, ion implantation and rapid thermal anneal can be used to tailor the etch rate of various nitride films. In this study, we report nitride film etch rates after anneal-only, implantation-only and implantation combined with anneal. We use different types of nitride films - deposited by LPCVD, RTCVD and PECVD techniques. It is shown that along with composition and structure, the intrinsic stress of these films affects the etch rates. Correlations of the etch rates with these basic film properties is discussed. Ion implanted silicon nitride (LPCVD, RTCVD and PECVD) films exhibit higher etch rates in weakly anisotropic dry etch compared to as-deposited films. Similar effect has been observed during wet etch of implanted nitride in some earlier studies. The observed increase in etch rate depends on both the concentration and the composition of implanted species. The implanted and unimplanted films were annealed at 950C and the etch rates determined and correlated with the stress in the films. The behavior of implanted SACVD LTO (low temperature oxide) and high temperature oxide (HTO) under the same implant and etch conditions are also studied. The etch rate dependence of LTO on implant conditions is not significant under the process conditions explored. The etch rate of implanted films after anneal is equivalent to that of as deposited films. Thus, the etch selectivity between nitride and oxide films can be tailored to meet process design requirements.

**NS-Thp28 Fabrication and Characterization of Reproducible SERS Substrates for Trace Chemical Detection, Sevnur Komurlu, S. Yilmaz, G. Ertaş, S. Suzer, Bilkent University, Turkey**

Gold nanoparticles are of fundamental interest and technological importance because of their applications in surface-enhanced Raman scattering (SERS). Gold nanoparticles having size range from ca. 5 to 100 nm have been prepared in three steps. Firstly, small spherical particles (seeds) of diameters ca. 2 nm were prepared by varying the ratio of gold ion concentration to citrate/NaBH<sub>4</sub> concentration. Secondly, larger particles were formed by a seed-mediated growth where small particles produced by the above technique were exploited as seeds and fresh Au(III) ions were reduced onto the surface on the seed particles by citrate ions. This seeding method repeated one more time and these nanoparticles were deposited onto amine-terminated substrates, which were obtained by modifying glass surfaces with 3-aminopropyltrimethoxysilane (APS). Such substrates possess amine surface functionality and uniform morphology, which can offer abundant and easily accessible binding sites for nanoparticles. SERS substrates consisting of gold nanoparticles deposited on glass slides are used to examine SERS effect of amino acids; lysine, histidine and aspartic acid. We use XPS to verify the deposition of the Au nanoparticles on APS substrate and amino acids on gold nanoparticles by recording the Au4f and N1s signals before and after immobilization steps. In addition, by controlling the charging/discharging via external voltage stress we can induce additional spectral separation of the XPS peaks of nitrogen in two different forms (amino acid and APS) present on the same surface.

**NS-Thp29 Electronic Detection of AFP by Si-based Field Effect Transistor, N.H. Lee, H.J. Park, W.-J. Kim, Korea Research Institute of Standards and Science; H.Y. Yu, S. Lee, Electronics and Telecommunications Research Institute, Korea; H.-T. Kim, Kumoh National Institute of Technology, Korea; W.S. Yun, Korea Research Institute of Standards and Science, Korea**

Lithographically-defined silicon nanowires were used in the detection of a cancer marker protein: alpha fetoprotein (AFP). Doped Si nanowires of 20 nm in thickness, 50 nm in width, and 10 @micron@ in length were fabricated on an insulating silicon oxide surface by e-beam lithography. On the surface of the nanowires, anti-AFP was immobilized by a series of chemical reactions. A 1¼-fluidic channel was then assembled onto the chip of the nanowires. When an aqueous solution of AFP (about 10 ng/ml in

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concentration) was introduced, the electric conductance along the nanowire changed its value by a factor of 2, which was attributed to the change in the local electric field felt by the semiconducting nanowire upon specific antibody-antigen binding. Since the Si nanowires in our work were defined by a lithographic technique, the fabrication of arbitrary-shaped and/or highly-integrated biosensors should become quite straightforward.

**NS-ThP30 Near IR Electroluminescent Studies of ZnS Photonic Crystal, E.S. Law**, University of Florida, Alachua; *P.H. Holloway*, University of Florida; *N. Shepherd*, University of North Texas

The effects of a photonic crystal on outcoupling of light from an alternating current thin film electroluminescent (ACTFEL) device is being studied. The ACTFEL device consists of a thin film of ZnS doped with erbium that was sputter deposited onto a glass substrate with an indium-tin-oxide (ITO) transparent conducting electrode and with an aluminum-titanium-oxide (ATO) thin insulating layer. Al top electrodes were vapor deposited on the ZnS:Er phosphor. For ZnS:Er, strong emission is observed at 1550nm. The photonic crystal was created in the ACTFEL device by etching away a hexagonal lattice of 264 nm diameter holes with 660nm lattice spacing. This pattern is expected to enhance the emission intensity perpendicular to the plane of the device. The photonic crystal pattern was created with electron-beam lithography using a PMMA resist over a complete ACTFEL device. A  $\text{Cl}_2/\text{Ar}$  reactive ion etch was used to etch through the Al top electrode layer with the PMMA as a mask. A  $\text{CHF}_3/\text{H}_2$  reactive ion etch was used to etch through the ZnS:Er phosphor, with the Al layer now acting as the mask. The emissions of ACTFEL devices with and without the photonic crystal will be compared using an optical spectrometer.

## Nanometer-scale Science and Technology Room 2016 - Session NS-FrM

### Nanowires

**Moderator:** S.E. Mohney, The Pennsylvania State University

8:20am **NS-FrM2 Double-Sided Epitaxy on Si Nanowire Ribbons\***, *C.S. Ritz, F.S. Flack*, University of Wisconsin, Madison; *Y. Zhang, F. Liu*, University of Utah; *M.G. Lagally*, University of Wisconsin, Madison

We study the ultrahigh-vacuum chemical vapor deposition growth of SiGe and Ge 3D islands on free-standing compliant Si nanowire ribbons. The Si template layer of a Si-on-insulator (SOI) sample with a thick buried oxide (BOX) is thinned down to the order of 20 nm via thermal oxidation and HF etching. This thin template layer can then be patterned into nanowire ribbons with widths less than 100nm along different crystallographic directions and connected at both ends. Reactive ion etching is used to remove the unwanted Si down to the BOX. Prior to SiGe growth a final HF dip is used to etch away this exposed oxide and undercut the Si ribbons, leaving them freestanding. CVD allows us to grow 3D islands on both the top and bottom surfaces of the Si ribbon. @footnote 1@ We observe a strong anticorrelation between islands grown on the top and bottom of the freestanding regions, as well as ordering of islands into rows along the ribbon edges. This latter ordering also exhibits additional dependence on the crystallographic orientation of the edge. Modeling suggests that this anticorrelation takes place to minimize the total strain energy and that growth on thinner ribbons induces and enhances correlation. In addition to the ordering of the dots, we also see global bending of the Si nanoribbon (because it is connected at both ends like a bridge) due to the elastic strain induced by the 3D islands. The islands affect the local electronic structure of the wire, which in concert with the ordering, makes double-sided epitaxy on nanowire ribbons a new approach to creating superlattice nanowires. @FootnoteText@ \*Supported by DOE.@footnote 1@Clark S. Ritz, Frank S. Flack, Michelle M. Roberts, Donald E. Savage, Yu Zhang, Feng Liu, Max G. Lagally, Double-sided growth of ordered SiGe islands on ultra-thin Si, in preparation.

8:40am **NS-FrM3 Silicidation and Oxidation of Silicon Nanowires**, *B.Z. Liu, Y. Wang, S.M. Dilts, J.M. Redwing, T.S. Mayer, S.E. Mohney*, The Pennsylvania State University

Metallization and oxidation are two important processes in the fabrication of transistors on silicon wafers. Forming metal contacts and gate dielectrics is equally important when integrating silicon nanowires into functional devices, but the processes have not been studied as extensively to date. We have used transmission electron microscopy to investigate the silicidation of silicon nanowires using Pt, Pd, Fe, Co, and Ni. Metal films were deposited on the silicon nanowires and annealed between 200 and 750°C for 15 min to 15 h. Sections of the silicon nanowires were successfully converted uniformly into silicides by controlling the ratio of metal to Si, annealing temperature, and ambient. We have also investigated the kinetics of oxidation of undoped and doped silicon nanowires. Dry thermal oxidation of the as-grown silicon nanowires was carried out at temperatures between 700 and 900°C with trichloroethane flowing. Differences in the oxidation kinetics between the silicon nanowires and silicon wafers are discussed. The electrical properties of the thermally grown oxide shell were investigated as well. The thermally grown oxide shell on the silicon nanowires has been successfully utilized as a gate dielectric that improves the operational stability of silicon nanowire field effect transistors.

9:00am **NS-FrM4 Large Area, Dense Si Nanowire Array Chemical Sensors**, *A.A. Talin*, Sandia National Laboratories, 94550; *L.L. Hunter, B. Rokad, F. Leonard, B.A. Simmons*, Sandia National Laboratories

We present a simple top down approach based on nanoimprint lithography to create dense arrays of silicon nanowires over large areas. Metallic contacts to the nanowires and a bottom gate allow the operation of the array as a field-effect transistor with very large on/off ratios. The carrier mobility extracted from transconductance measurement exceed the values reported for chemically synthesized Si nanowires with similar dimensions. When exposed to ammonia gas or cyclohexane solutions containing nitrobenzene or phenol, the threshold voltage of the field-effect transistor is shifted, a signature of charge transfer between the analytes and the nanowires. The threshold voltage shift is proportional to the Hammett parameter and the concentration of the nitrobenzene and phenol analytes. For the liquid analytes considered, we find binding energies of 400 meV,

indicating strong physisorption. Such values of the binding energies are ideal for stable and reusable sensors.

9:20am **NS-FrM5 Controlling the Width of Self Assembled Nanowires on the Si(001) Surface**, *J. Nogami, Y. Cui, J. Chung, D. Grozea*, University of Toronto, Canada; *C. Ohbuchi*, National Institute for Materials Science, Japan

Many low dimensional structures arise from self-assembly when depositing metals onto silicon surfaces, including both quantum dots and quantum wires. One class of these objects are rare earth silicide nanowires that grow on Si(001).@footnote 1@ In any self assembled nanostructure, one of the critical issues is control over feature size. In the case of metals deposited on the Si(111) surface, the 7x7 reconstruction has been used as a template to create clusters with a very narrow size distribution, where the metal atoms are confined to one half of the reconstruction unit cell.@footnote 2@ We present STM data that shows it is possible to use a metal induced 2x7 reconstruction of Si(001).@footnote 3@ to narrow the width distribution of Dy silicide nanowires. The interaction between the reconstruction and the nanowires is more complex than in the 7x7 case since nanowire growth induces local changes in the 2x7 periodicity. We will suggest possible mechanisms for the templating phenomenon. @FootnoteText@ @footnote 1@ J. Nogami et al, Phys. Rev. B 63 (2001) 233305.@footnote 2@ J. Jia et al, Appl. Phys. Lett 80 (2002) 3186.@footnote 3@ B.Z. Liu and J. Nogami, Surf. Sci. 540 (2003) 136.

9:40am **NS-FrM6 Synthesis and Characterization of Aligned III-Nitride Nanowire and Heterostructure Nanowire Arrays**, *G.T. Wang*, Sandia National Laboratories; *A.A. Talin*, Sandia National Laboratories, 94550; *J.R. Creighton*, Sandia National Laboratories; *D. Werder*, Los Alamos National Laboratory; *E. Lai, P.P. Provencio*, Sandia National Laboratories

Nanowires based on the direct bandgap semiconductor Group III nitride (AlGaInN) materials system have attracted attention as potential building blocks in nanophotonics, nanoelectronics, and sensing. We have employed a metal-organic chemical vapor deposition process to synthesize highly aligned arrays of single-crystalline GaN nanowires in a standard cold-wall rotating disk reactor on 2-inch diameter sapphire and GaN substrates without the use of a template. SEM and TEM analysis indicate that the nanowires share a common growth direction and have aligned facets. Interestingly, the majority of the nanowires do not have a catalyst droplet at the tip, suggesting the growth may differ from the standard vapor-liquid-solid process. Building on this technique, we have also been able to synthesize radial heterostructure nanowire arrays consisting of a GaN cores and various III-nitride shell materials, including AlN, InN, and AlGaInN. The GaN and heterostructure nanowires were probed using a Sandia-developed platform which allows us to correlate the morphological, optical, and electrical properties of a statistically relevant number of nanowires. Results from 3D STEM tomography and spatially resolved photoluminescence and cathodoluminescence studies will also be presented. We have found that the growth conditions, particularly temperature, have a strong effect on the structural, optoelectronic, and electrical properties of the nanowires. Additionally, the choice of substrate and the catalyst preparation play critical roles in the density, uniformity, and alignment of the nanowire arrays. Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

10:00am **NS-FrM7 Epitaxially Grown III-V Nanowires for Quantum Device Applications**, *L. Samuelson*, Lund University, Sweden **INVITED**

In this talk I will survey progress during the last couple of years in the controlled growth of epitaxially nucleated nanowire structures, obtained via local catalytic activation of growth. From an application point of view, progress in the technologies which allow control of geometrical dimensions and positioning of nanowire structures and devices is of great importance, as is the recently demonstrated ideal formation of III-V nanowires epitaxially grown on silicon substrates. From the perspective of designing and realizing complex device structures, by which quantum phenomena can be utilized, the formation of axial as well as radial heterostructures within a nanowire is of special interest and I will give different examples of how this has been achieved. Finally, I will present a couple of quantum device families that we have been working on in the last year, such as wrap-gate field-effect transistors and multiple-barrier storage devices.

# Friday Morning, November 17, 2006

10:40am **NS-FrM9 Electrical Characterization of Nanowires with the LEEPS Microscope**, *D.H. Weber, A. Beyer, A. Götzhäuser*, University of Bielefeld, Germany

We introduce the LEEPS microscope as a tool for the electrical characterization of nanowires. We have contacted single nanowires in the LEEPS electrically and have measured the conductivity. In addition the LEEPS image itself represents the electrical properties. The LEEPS (Low Energy Electron Point Source) microscope is a transmission electron microscope with electron energies from 20 eV to 200 eV. These electrons are emitted by a field emission tip with a radius in the atomic range. Because the electrons have a high spatial coherence the resulting detector image is an interference pattern which includes structural as well as electrical and magnetic information of the object. The LEEPS image includes features which are very typical for either conductive or nonconductive nanowires. The interference pattern of conductive nanowires appears much brighter referring to the image background as the interference pattern of a nonconductive nanowire. With a sharp manipulation tip as a movable electrode a single nanowire can be contacted electrically and the I/U curve is measured. Contacting and measurement can be observed with the LEEPS microscope subsequently. To take the contact resistance into account, length dependent measurements have been performed. We present I/U curves of single nanowires (i.e. ZnO) as well as a comparison of LEEPS images of conductive and nonconductive wires (i.e. ZnO, Cu, Bi and Al@sub 2@O@sub 3@, polymers).

11:00am **NS-FrM10 Germanium Nanocrystals and Nanowires: Morphological Control, Surface Characterization, and Applications**, *H. Gerung, L.J. Tribby*, University of New Mexico; *T.N. Lambert*, Sandia National Laboratories; *N. Andrews*, University of New Mexico; *T.J. Boyle*, Sandia National Laboratories; *C.J. Brinker, J.M. Oliver, S.M. Han*, University of New Mexico

The use of Ge@sup 0@ nanocrystals (NCs) and nanowires (NWs) for advanced materials applications are of great interest; however, complex synthetic routes have hindered their use and development. We have recently developed a simple route to produce Ge@sup 0@ NCs and NWs from the reduction of Ge@sup +2@ precursors at 300°C and 1 atm Ar without using metal catalysts and without producing salt byproducts. Taking a molecular design approach, we tailor the Ge-ligand bond and the ligand steric hindrances to control Ge@sup +2@ precursors' reactivity. More reactive Ge@sup +2@ precursors yield Ge@sup 0@ NCs, while less reactive alkoxide precursors yield Ge@sup 0@ NWs. X-ray diffraction and transmission electron microscopy show that both NCs and NWs are in cubic phase. The surface of these Ge@sup 0@ NCs and NWs can be further functionalized to prevent spontaneous oxidation, which is useful for both optical and biological applications. The resultant Ge@sup 0@ NCs are optically active, as demonstrated by the presence of photoluminescence in both visible (around 450 nm) and infrared region (around 1300 nm). Ge@sup 0@ NCs also display highly nonlinear optical behavior; the experimentally measured two photon absorption coefficient ranges from 1190 to 1940 cm<sup>2</sup>/GW. We also demonstrate that water-soluble Ge@sup 0@ NCs are stable for months. Preliminary investigations on the use of Ge@sup 0@ NCs as a biological probe reveal that dinitrophenol (DNP)-decorated Ge@sup 0@ nanocrystals readily bind to anti-DNP IgE receptors on mast cells, while maintaining high cell viability comparable to the control cells. The details of synthesis, optical properties, and biofunctionalization will be presented. The authors acknowledge generous support from NSF CAREER (DMR-0094145). 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.

11:20am **NS-FrM11 Fabrication of Tin Oxide Nanowires Based Nano-Micro Sensor for Room Temperature Hydrogen Detection**, *S. Deshpande, A. Karakoti, G. Lande, H.J. Cho, S. Seal*, University of Central Florida

Randomly oriented tin oxide nanowires, with diameters on the order of 100 nm, were grown on Si substrate using thermal evaporation technique. The thermodynamic and kinetic factors leading to the evolution of tin oxide nanowires from vapor solid transformation during the process is evaluated. These nanowires were successfully incorporated into micro-electro-mechanical (MEMS) device. Hydrogen (H<sub>2</sub>) at ppm-level has been successfully detected at room temperature using the present 1-dimensional tin oxide based MEMS sensor. The device showed rapid and reversible resistance changes upon periodic exposure to hydrogen gas (less than 900ppm concentration). The one dimensional geometry of tin oxide is responsible for the device response. Effect of aspect ratio of the nanowires

on diffusion of hydrogen molecules in the tin oxide nanowires, effect of catalyst adsorption on nanowire surface and corresponding effect on sensor properties has been studied in detail.

11:40am **NS-FrM12 Atomic Level Analysis of Carbon Nanotubes and Graphite Nanofibers by the Scanning Atom Probe**, *O. Nishikawa, M. Taniguchi*, Kanazawa Institute of Technology, Japan

Utilizing the unique capability of the scanning atom probe (SAP) carbon nanotubes (CNT) and graphite nanofibers (GNF) are mass analyzed detecting individual field evaporated ions. The analyzed CNTs are the mixture of single and multiple wall nanotubes (SWCNT and MWCNT). A minute lump of densely intertwined CNT was mounted at the apex of a tungsten tip with silver paste. The GNFs are grown on a nickel substrate. The mass analysis of the CNT and GNF by the SAP was conducted by applying DC and pulsed voltages to the specimen at room temperature. The mass resolution of the SAP is better than 1000. It has been reported that most CNTs and graphite contain a significant amount of hydrogen and oxygen. In this study we focused at the correlation between the C-C binding and the local concentration of hydrogen and oxygen in CNTs and GNFs. Field evaporation of metal proceeds atom-by-atom base because the metallic binding is uniform and non-directional. On the other hand the covalent bonds are directional and non-uniform. Accordingly, non-metallic specimens are mostly field evaporated as cluster ions. Furthermore, strongly bound clusters are field evaporated as multiply charged ions. An interesting finding is the detection of many large carbon cluster ions. The number of carbon atoms is not random but quite characteristic such as 11, 17, 19 and so on. The mass peaks of doubly and triply charged ions are very sharp without a tail indicating strong C-H bonds. On the other hand the mass peaks of singly charged cluster ions have a tail. This implies that the C-H bonds are weak and the C-H cluster are dissociated before entering the ion detector. Detection of the large cluster may imply that the CNT is field evaporated as a nano-size graphene sheet and not as a carbon chain. The electronic state of the specimens and the correlation between the size of cluster and the ratio of the number of carbon atoms to that of hydrogen atoms will be discussed.

**Bold page numbers indicate presenter**

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- Sugimoto, Y.: NS2-TuM5, 9
- Sugiyama, T.: NS-ThP26, 28
- Sullivan, J.P.: NS+SS+TF-WeM2, 15
- Sumant, A.V.: NS+SS+TF-WeM1, 14; NS+SS+TF-WeM2, 15
- Sung, S.: PS1+MS+NM-TuM10, 11
- Suu, K.: PS1+MS+NM-TuM3, 10
- Suzer, S.: NS-ThP28, 28
- Suzuki, A.: SS2+NS+TF-ThA4, 23
- Suzuki, R.: NS-ThP13, 26
- Syed Asif, S.A.: NS+SS+TF-WeM13, 16
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- Takahara, A.: SS2+NS+TF-ThA4, 23
- Takarai, Y.: NS-ThP26, 28
- Takeguchi, M.: NS-ThA6, 22
- Takemura, S.: NS-ThP26, 28
- Talin, A.A.: NS-FrM4, 30; NS-FrM6, 30; NS-MoA8, 6; NS-WeA5, 18
- Tang, H.Y.: NS-ThP6, 25
- Tang, M.: NS-MoA7, 5
- Taniguchi, M.: NS-FrM12, 31; NS-ThP26, 28
- Taylor, A.: NS1-TuM3, 7
- Tenne, R.: NS+SS+TF-WeM9, 16
- Terminello, L.J.: AS+BI+NS+NM-MoM3, 1; NS1-TuM12, 8
- Tevaarwerk, E.: NS1-TuM1, 7
- Textor, M.: AS+BI+NS+NM-MoM5, 1
- Thiault, J.: PS1+MS+NM-TuM2, 10
- Timp, G.L.: NS+BI-ThM1, 20
- Tohji, K.: NS-WeA9, 19
- Tone, K.: NS-ThP16, 26
- Tormen, M.: NM+NS+NNT-WeM1, 14
- Tour, J.M.: NS2-TuM3, 8
- Tribby, L.J.: NS-FrM10, 31
- Tsai, C.H.: NS-ThA5, 22; NS-ThP10, 25
- Tse, K.-Y.: NS1-TuM6, 7
- Tsong, T.T.: NS-ThP14, 26
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- Tyler, T.: NS-ThA7, 22
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- Uraoka, Y.: NS-ThA6, 22
- V —
- Valesia, A.: AS+BI+NS+NM-MoM10, 1; NS+BI-ThM4, 20
- van Buuren, T.: AS+BI+NS+NM-MoM3, 1; NS+SS+TF-WeM10, 16; NS1-TuM12, 8
- Van Nostrand, J.E.: NS+BI-ThM5, 20
- Vemula, S.C.: PS1+MS+NM-TuM6, 10
- Verdaguer, A.: NS-MoA1, 5
- Vericat, C.: SS2+NS+TF-ThA5, 23
- W —
- Wagner, H.D.: NS+SS+TF-WeM9, 16
- Wang, G.T.: NS-FrM6, 30
- Wang, J.: PS1+MS+NM-TuM10, 11
- Wang, J.J.: NS-MoA2, 5
- Wang, S.: NS-MoA2, 5
- Wang, S.C.: NS-ThP23, 27
- Wang, S.X.: NS+NM-WeA4, 17
- Wang, Y.: NS-FrM3, 30
- Wang, Y.M.: NS+SS+TF-WeM10, 16
- Warf, P.: NS1-TuM6, 7
- Warren, O.L.: NS+SS+TF-WeM13, 16
- Watanabe, Y.: NS-ThP26, 28
- Weber, D.H.: NS-FrM9, 31
- Wege, S.: PS1+MS+NM-TuM11, 11
- Wei, C.M.: NS-ThP14, 26
- Wei, Z.Q.: NS-ThP1, 25
- Weimann, T.: NS1-TuM11, 8
- Weiner, D.: NS-MoM9, 3
- Weiss, P.S.: NS2-TuM10, 9
- Weng, C.H.: NS-ThA5, 22; NS-ThP10, 25
- Werder, D.: NS-FrM6, 30
- West, R.: NS1-TuM6, 7
- Wheeler, D.: AS+BI+NS+NM-MoM4, 1
- White, R.G.: NS+BI-ThM6, 20
- Whitman, L.J.: NS+NM-WeA1, 17
- Willey, T.M.: AS+BI+NS+NM-MoM3, 1
- Williams, R.S.: NS1-TuM2, 7
- Willson, G.: NS+NM-WeA5, 17
- Wilson, R.J.: NS+NM-WeA4, 17
- Wittmann, M.: NS-MoM4, 3
- Wolstenholme, J.: NS+BI-ThM6, 20
- Wong, M.S.: NS-ThP6, 25
- Wu, Z.G.: NS-ThP10, 25
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- Xiao, Z.: NS-ThP9, 25
- Xu, L.: NS+NM-WeA4, 17; PS1+MS+NM-TuM6, 10
- Xu, T.: NS-MoA5, 5
- Y —
- Yamashita, I.: NS-ThA6, 22
- Yang, H.: NS-ThA8, 23
- Yang, M.: NS+NM-WeA1, 17
- Yap, H.W.: NS+SS+TF-WeM6, 15
- Yilmaz, S.: NS-ThP28, 28
- Yoder, N.L.: NS2-TuM4, 9
- Yokota, Y.: NS2-TuM13, 9
- Yoo, W.J.: NS-MoA9, 6
- Yu, H.Y.: NS-ThP29, 28
- Yun, W.S.: NS-ThP29, 28; NS-ThP7, 25
- Z —
- Zepeda-Ruiz, L.A.: NS+SS+TF-WeM11, 16
- Zhang, G.: NS-MoA9, 6
- Zhang, H.: NS-MoA7, 5
- Zhang, J.: NS2-TuM3, 8
- Zhang, L.Z.: NS1-TuM6, 7
- Zhang, P.: NS1-TuM1, 7
- Zhang, Y.: NS-FrM2, 30; PS1+MS+NM-TuM5, 10
- Zhao, L.Y.: NS-MoA4, 5
- Zhao, X.: NS-MoA2, 5
- Zhou, C.: NS-WeA3, 18
- Zhu, L.: NS-MoM12, 4
- Zhu, M.: NS-MoA2, 5
- Zhu, X.-Y.: SS2+NS+TF-ThA3, 23
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