Tuesday Morning, November 14, 2006

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@ 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@super 15@ cm@super -3@, 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.@footnote 1@ 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. @FootnoteText@ @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

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 ~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@ 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@ 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@ 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@ 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.@footnote 1@ Additionally, the polar angle control is one of the critical factors for the accurate thickness measurement of SiO@sub 2@ 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@super o@. This study demonstrates that AR-XPS can provide reliable measurement of SiO@sub 2@ 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).

Author Index

Bold page numbers indicate presenter

— A —
Aizenberg, J.: NS1-TuM3, 1
— B —
Baker, S.E.: NS1-TuM6, 1
Blackstock, J.J.: NS1-TuM2, 1
— C —
Celler, G.: NS1-TuM1, 1
— D —
Dementiev, V.: NS1-TuM6, 1
Donley, C.L.: NS1-TuM2, 1
Dziomba, T.: NS1-TuM11, **2**
— E —
Eliseev, E.A.: NS1-TuM9, 1
Eriksson, M.A.: NS1-TuM1, 1
Evans, P.: NS1-TuM1, 1
— H —
Hamers, R.J.: NS1-TuM6, 1
Hinze, P.: NS1-TuM11, 2
Hodes, M.S.: NS1-TuM3, 1
Holscher, H.: NS1-TuM10, **2**
— J —
Jang, J.S.: NS1-TuM13, 2
Jesse, S.: NS1-TuM9, **1**
— K —
Kalinin, S.V.: NS1-TuM9, 1
Kang, H.J.: NS1-TuM13, 2
Kim, K.J.: NS1-TuM13, **2**
Knezevic, I.: NS1-TuM1, 1
Koenders, L.: NS1-TuM11, 2
Kolodner, P.R.: NS1-TuM3, 1
Kranzmann, A.: NS1-TuM11, 2
Krupenkin, T.N.: NS1-TuM3, **1**
— L —
Lagally, M.G.: NS1-TuM1, 1
Lee, J.R.I.: NS1-TuM12, 2
— M —
Matsumoto, K.: NS1-TuM5, **1**
Meulenberg, R.W.: NS1-TuM12, **2**
Moon, D.W.: NS1-TuM13, 2
Morozovska, A.N.: NS1-TuM9, 1
— P —
Park, B.: NS1-TuM1, 1
— R —
Ritter, M.: NS1-TuM11, 2
Rodriguez, B.J.: NS1-TuM9, 1
— S —
Savage, D.E.: NS1-TuM1, 1
Senoner, M.: NS1-TuM11, 2
Stewart, D.R.: NS1-TuM2, **1**
Stickle, W.F.: NS1-TuM2, 1
— T —
Taylor, A.: NS1-TuM3, 1
Terminello, L.J.: NS1-TuM12, 2
Tevaarwerk, E.: NS1-TuM1, 1
Tse, K.-Y.: NS1-TuM6, **1**
— V —
van Buuren, T.: NS1-TuM12, 2
— W —
Warf, P.: NS1-TuM6, 1
Weimann, T.: NS1-TuM11, 2
West, R.: NS1-TuM6, 1
Williams, R.S.: NS1-TuM2, 1
— Z —
Zhang, L.Z.: NS1-TuM6, 1
Zhang, P.: NS1-TuM1, **1**