Wednesday Morning, October 27, 1999

Electronic Materials and Processing Division
Room 6C - Session EM+NS-WeM

Nano-characterization of Molecules, Materials, and Devices

Moderator: R.S. Goldman, University of Michigan

8:20am **EM+NS-WeM1 Homoepitaxy on AlSb(001): Novel Reconstructions and Their Implications for Nucleation and Growth**, ***W. Barvosa-Carter,*** HRL Laboratories*; A.S. Bracker, J.C. Culbertson, B.V. Shanabrook, B.R. Bennett, L.J. Whitman,* Naval Research Laboratory*; N. Modine,* Sandia National Laboratories*; H. Kim, E. Kaxiras,* Harvard University

Strained-layer heterostructures involving the 6.1 Å family of III-V semiconductors (InAs, GaSb, and AlSb) are being investigated for use in a growing number of high-frequency and infrared devices. The structure of the interfaces in these heterostructures can be critical to device performance, and device optimization will ultimately require precise and reproducible control over surface morphology during growth. To accomplish this level of morphological control, models are being developed which relate process parameters to surface roughness. These models require a detailed understanding of the relevant surface reconstructions and the mechanisms by which epitaxy proceeds. Using MBE, RHEED, and STM (performed at NRL), combined with first-principles theoretical calculations, we have discovered a novel (4x3) reconstruction on the nominally "(1x3)" AlSb(001) growth surface. This new reconstruction is different than those previously proposed for this surface and, surprisingly, includes mixed III-V dimers in the top layer of the reconstruction. The presence of surface Al atoms close to their natural lattice sites leads to nucleation and growth mechanisms that are fundamentally different than for III-As systems. We have also studied AlSb homoepitaxy as a function of coverage. The relationship between the observed reconstructions, island structure, island distributions, and possible growth modes will be discussed.

8:40am **EM+NS-WeM2 A New Point Projection Microscope for the Holographic Imaging of Single Macromolecules**, ***A. Eisele,*** *B. Völkel, A. Glenz, B. Jäger, A. Gölzhäuser, M. Grunze,* Universität Heidelberg, Germany

In Low Energy Electron Point Source microscopy the spatial coherence of electrons from point sources can be utilized to image single molecules. A molecular object is positioned ~100 nm in front of the source and interference patterns between the part of the electron's wave function that scatters at the object and the part that passes by unscattered are recorded.@footnote 1@ Structural information on the molecule can then be obtained by numerical reconstruction of the hologram.@footnote 2,3@ We have built a new microscope for the recording of holograms at high magnification (@>=@10@super 6@) and under the minimization of critical disturbances like vibrations and alternating magnetic fields. In the presented instrument projection microscopy can be interleaved with in-situ preparation of the source via field emission / field ion microscopy. The microscope has been tested by the imaging of single DNA molecules that were deposited on thin structured siliconmembranes. Numerical reconstructions of the obtained holograms show corrugated strands with a diameter of ~2 nm. @FootnoteText@ @footnote 1@ H.-W. Fink, W. Stocker, and H. Schmid, Phys. Rev. Lett. 65, 1204 (1990) @footnote 2@ H. J. Kreuzer, K. Nakumura, A. Wiezbicki, H.-W. Fink, and H. Schmid, Ultramicroscopy 45, 381 (1992) @footnote 3@ A. Gölzhäuser, B. Völkel, B. Jäger, M. Zharnikov, H.J. Kreuzer, M. Grunze, J. Vac. Sci. Technol. A16(5), 3025 (1998)

9:00am **EM+NS-WeM3 Single Molecule Vibrational Spectroscopy with a Variable Temperature STM**, ***L.J. Lauhon,*** *W. Ho,* Cornell University **INVITED**

The ultimate sensitivity for vibrational spectroscopy is the detection of a single bond. The vibrational spectrum of a single molecular adsorbate carries information about the effects of the local environment on chemical bonding. Such effects are the basis of important processes such as catalysis. Single bond sensitivity was recently demonstrated by using a scanning tunneling microscope to perform inelastic electron tunneling spectroscopy (STM-IETS) on a single acetylene molecule.@footnote 1@ We have extended this technique to other molecules at temperatures from 8 K to 60 K in an effort to both better understand and widen the applicability of STM-IETS. Two 'tunneling-active' vibrational modes have been identified for CO adsorbed on Cu(001) and Cu(110). The effects of monatomic steps and coadsorbed potassium on the vibrational spectra, including peak shifting and quenching, were found to be local in nature. The increase in the vibrational peak width with temperature was measured up to 40 K, beyond which thermal diffusion prevented STM-IETS spectra from being recorded. STM-IETS was also performed on pyridine and benzene adsorbed on Cu(001). Though these molecules differ only in the substitution of a nitrogen atom for one C-H group, their bonding geometries and vibrational spectra are very different. Achieving the spatial limit of nanotechnology depends on the ability to perform chemistry on the atomic scale. To this end, tunneling electrons were used to dissociate individual pyridine and benzene molecules. The adsorbtion geometries of the reaction products differ from the parent molecules and lead to changes in the vibrational spectra which provide insights into the identities of the reaction products and the tunneling mechanism. The extension of STM-IETS to new functional groups, including larger molecules, will also be discussed. @FootnoteText@ @footnote 1@ B. S. Stipe, M. A. Rezaei, and W. Ho, Science Vol. 280, p. 1732 (1998).

9:40am **EM+NS-WeM5 Characterization of Electronic Materials and Devices by Scanning Probe Microscopies**, ***C.C. Williams,*** *V. Zavyalov, L. Klein,* University of Utah*; J. Kim,* Korea Advanced Institute of Science and Technology **INVITED**

Several studies of the electrostatic properties of oxides and silicon devices have been performed by the Scanning Capacitance Microscope (SCM) and the Electrostatic Force Microscope (EFM). The SCM provides a method for measuring topographical and electrical roughness of thin oxides, surface charge and local carrier and dopant density in semiconductors. On thin oxides, the SCM reveals a nanometer scale variation in the "electrical thickness" of the oxide. The thickness variations seen by SCM have been compared with topographical (AFM) and surface potential measurements by EFM in UHV. Surface potential variations of order 5 mV are observed on the same spatial scale as the thickness variations seen by the SCM. Calculations show that the measured surface potential variations correspond to less than one electron per tip area (30 nm radius). Single MOSFET devices have been imaged in cross-section under active electrical bias by SCM. The images provide a measure of the distribution of the carriers in a device under bias. Finally, a new technique will be described for detecting the transfer of a single electron between a SPM tip and surface.

10:20am **EM+NS-WeM7 Mapping Composition and Electrostatic Potential in Devices**, ***A. Ourmazd,*** *A. Orchowski, W.-D. Rau, P. Schwander,* IHP, Germany **INVITED**

An electronic device is, in essence, a microscopically varying electrostatic potential surface, which steers the charge carriers between the device's terminals. Until recently, there were no means for directly measuring the electrostatic potential distribution in the bulk of devices. It is now possible to map the electrostatic potential in two dimensions by electron holography. Maps of deep submicron transistors have been obtained with nanometer spatial resolution and 0.1V sensitivity. The electrostatic potential surface can be tailored by changes in composition and/or doping. It is often important to separate the two effects. Electron holography alone, however, cannot distinguish between them; they both change the electrostatic potential. QUANTITEM, on the other hand, is sensitive to compositional changes only. Efforts are under way to combine the results from electron holography and QUANTITEM, in order to separate the effects of composition and doping, with first encouraging results.

11:00am **EM+NS-WeM9 Failure Analysis of Sub 1/4-Micron Contacts by Means of TEM-EELS**, ***F. Yano,*** *Y. Nakamura, T. Aoyama, Y. Mitsui,* Hitachi Ltd., Japan

Although TEM-EELS (Transmission Electron Microscope-Electron Energy Loss Spectroscopy) has practically been used for elemental analysis for nanometer area, its full potential, we believe, is achieved when it is used for chemical analysis just like ESCA. This paper uncovers our experience of thermally stable contact development, in which our advanced TEM-EELS@footnote 1@ has revealed reactions in the contact during thermal process, which have detrimentally increased its resistivity.@footnote 2,3@ The contact holes are filled with sputtered Ti, which is annealed to form TiSi@sub 2@, CVD-TiN and CVD-W. Alathough the contact resistivity was low enough just after contact processes, it became higher after thermal process. The resistivity was varied 10@super 2@ to 10@super 6@ @ohm@ depending on the process conditions. Chemical analysis of loss energies showed that Si substrate in the contact is fully covered with SiO@sub 2@ in the fatal worst case. Even somewhat better cases, TiSi@sub 2@ formation was partial and still SiO@sub 2@ was formed. In other cases, TiO@sub x@ was also observed. These oxidized layers clearly increased resistivity. However, the mechanism of oxidation was unclear,

especially the origin of the oxygen was, because neither oxygen nor water was applied. To clarify the mechanism, we made a model, in which residual TiCl@sub x@ on CVD-TiN forms titanium acid gel (TiO@sub x@(H@sub 2@O)@sub n@) which works as a water reservoir. During thermal process, the water in the gel is released, which goes through TiN grain and finally oxidizes silicon at the interface of TiN and Si substrate. To prove this model, the relation between the amount of residual Cl and the contact resistivity was measured. The results supported the model above, i.e., the more Cl observed, the higher the resistivity is. Based on this mechanism, all cases of high resistivity failures in the thermally stable contact were explained. Chemical analysis by TEM-EELS will be a key technique for failure analysis of 1/4-micron devices and after. @FootnoteText@ @footnote 1@ T. Sekiguchi, et al., Jpn. J. Appl. Phys., vol. 37 (1998) L694. @footnote 2@ Y. Mitsui, et al., Ext. Abst. IEDM (1998) 329.@footnote 3@ Y. Nakamura, et al., Proceedings of Advanced Metallization Conference, Colorado (1998) 661.

11:20am **EM+NS-WeM10 Applications of AFM/SCM in Imaging Implant Structures of Semiconductor Devices**, ***K.-J. Chao***, *J.R. Kingsley, R.J. Plano, X. Lu, I.D. Ward,* Charles Evans & Associates

The scanning capacitance microscopy (SCM) has been widely used to investigate the two-dimensional carrier profile of semiconductor devices. In this work, SCM is used to investigate several different semiconductor devices. First, one commercially purchased integrated circuits (IC) device was cross-sectioned and polished for the SCM investigation. Implant structures near the gate were clearly resolved. Second, two semiconductor devices, one was good and the other was failed, were prepared by cross-sectioning and then followed by polishing. Implant profiles of similar structures on both devices were revealed by SCM. As compared with the good device, the thickness of the N-well structure was found to be thinner by about 0.4um for the failed device. Third, a GaAs device with Zn thermally diffused through the Si3N4 mask was studied to determine the lateral diffusion length of Zn. Applications in other cases will be presented at the conference.

11:40am **EM+NS-WeM11 Capacitance Measurements on Gold Nanowires**, ***A. Wlasenko,*** McGill University, Canada

There are several assumptions made about classical capacitors (C=Q/V): the density of states(DOS) of the plates is infinite, the potential drop occurs entirely across the plates, electrons don't interact, and there is no tunnelling. In mesoscopic capacitors, the voltage drop doesn't occur entirely across the plates, and the finite DOS plays an important role. In the experimental setup presented, the voltage-dependence of C is measured for gold nanowires allowing the DOS to be deduced according to theory [H. Guo et al., APL 74, 2887-2889 (1999)]. A piezotube is used to retract a gold sample in contact with a gold tip to form a nanowire. Along with measurements of C, simultaneous measurements of conductance(G) are made with a current preamplifier to monitor the transmission properties of the contact. Changes in the C of this nanowire are measured by a modified RCA Video Disc sensor: a resonant circuit is connected to a 915 MHz oscillator, and the amplitude of the signal is measured. A change in C leads to a shift in the resonance peak which is detected by the sensor. The sensor is calibrated using a ball bearing and metal plane geometry in comparison with classical calculations. The sensor is sensitive to C changes as low as 10@super -17@ F. When the tip and sample are in contact, the sensor measures a convolution of C and G. Where a variation in C leads to a shift in the resonance peak, a variation in G leads to a change of the FWHM. Measurements on either side of the resonance peak can be made in order to seperate C and G.

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