Nanometer-scale Science and Technology
Room: 12 - Session NS+SP-MoA

Monday Afternoon, October 29, 2012

Nanopatterning and Nanolithography
Moderator: P.E. Sheehan, U.S. Naval Research Laboratory

2:20pm NS+SP-MoA1 Optical Properties of As-Prepared and Annealed Gold Nanostructures Fabricated by Interference Lithography A.B. Tsargorodska, University of Sheffield, UK; A.V. Nabok, Sheffield Hallam University, UK; A.J. Lee, University of Leeds, UK; G.J. Leggett, University of Sheffield, UK

Gold nanostructures were fabricated by interferometric lithography (IL) with a view to their application for optical bio-sensing based on localized surface plasmon resonance. This approach relies upon very modest instrumentation, high processing speed and capacity for fabrication of dense arrays of nanostructures over macroscopic areas. The dimensions and morphology of nanostructures obtained were characterized by AFM/SEEM. The arrays of 65-200nm wide and 125-400nm long gold nano-dots and nano-rods with spacing of 120-220nm were fabricated by IL.

UV-vis absorption spectra of gold nanostructures showed a characteristic peak at ca. 520nm associated with localized surface plasmons. A spectroscopic ellipsometry study of the gold nanostructures was carried out. Raman spectra of a thin film of phathlocyanine adsorbed on gold nanostructures showed an enhancement of Raman scattering of up to 200 times compared with the same film deposited on continuous gold layer.

Post-lithographic processing by annealing was found to improve the optical properties of the nanostructure arrays still further. Annealed samples appear higher and smaller compared to as-prepared samples. AFM images show that all the samples annealed at between 450-470°C for 50-120min retain a regular pattern. XRD analysis of annealed gold nanostructures evinces formation of crystal clusters on the surface.

In order to demonstrate the bioconjugating capabilities of annealed and as-prepared nanostructures, the LSPR response of nanostructures to changes in the local environment (RI) was investigated. The RI sensitivity of the annealed samples is found to be 145nm/RIU. Compared to the RI sensitivity of the as-prepared sample (52.5nm/RIU) this value is ~2.7 times higher due to smaller size of nanostructures and larger spacing between them.

A model bioconjugating of streptavidin-biotin binding and BSA were successfully performed on as-prepared and annealed gold nanostructures. Measurements were based on the LSPR shift induced by local RI change when proteins are immobilized on the nanostructure. Streptavidin concentrations were washed over the nanostructured surface in cumulative succession ranging from 0.1ug/ml to 0.1mg/ml, followed by an identical protocol with biotin. Characteristic increases and saturation are noted through shifts in the absorption of the LSPR peak at ca. 635nm. Annealed nanostructures demonstrated a higher detection limit (DL) compare to as-prepared nanostructures. BSA for BSA is 1.5mM and 50pM for annealed and as-prepared nanostructures, respectively. Our results demonstrate the potential of interference lithography for the application of plasmon-based bioconjugating devices.

2:20pm NS+SP-MoA2 Fast Turnaround 3D Nanolithography using Heated Probes – from Nanofabrication to Directed Assembly A.W. Knoll, P. Paul, IBM Research - Zurich, Switzerland, F. Holzner, IBM Research - Zurich, Switzerland, J.L. Hedrick, IBM Research - Almaden, M. Despont, IBM Research - Zurich, Switzerland, C. Kuenen, IBM Research - Zurich, ETH Zurich, Switzerland, H. Wolf, IBM Research - Zurich, Switzerland, N.D. Spencer, ETH Zurich, Switzerland, U. Duerig, IBM Research - Zurich, Switzerland

INVITED

A novel thermal scanning probe lithography (tSPL) method based on the local removal of organic resist materials has been developed at the IBM Research Laboratory in Zurich [1-3]. A polymeric poly(2-phenylaldehyde) resist [2-4] responds to the presence of a hot tip by local material decomposition and desorption. Thereby arbitrarily shaped patterns can be written in the organic films in the form of a topographic relief, constrained only by the shape of the tip. The combination of the fast 'direct development' patterning of a polymer resist and the in-situ metrology capability of the AFM setup allows to reduce the typical turnaround time for nano-lithography to minutes.

Patterning rates of 500 kHz have been achieved. For this, the mechanics and drive waveform of the scan stage were optimized, achieving high speed linear scanning with an overall position accuracy of ± 10 nm over scan-ranges and scan-speeds of up to 50 µm and 20 mm/s, respectively. A pre-tension-and-release strategy was used to actuate the cantilever above its resonance frequency of 150 kHz.

Fabrication of three dimensional patterns is done in a single patterning step by controlling the amount of material removal at each pixel position. The individual depths of the pixels are controlled by the force acting on the cantilever.

The structuring capability in the third dimension adds an entirely new feature to the lithography landscape and finds applications e.g. in multi-level configuration, nano/micro-optic components and directed positioning of nanoparticles. For the latter, shape-matching guiding structures for the assembly of nanorods of size 80nm × 25nm have been written by thermal scanning probe lithography [4]. The nanorods were assembled into the guiding structures by means of capillary interactions. Following particle assembly, the polymer was removed cleanly by thermal desorption of the reaction by-product. The pseudo-localized (~ 100nm spot), pulsed laser enables thermal desorption of the reaction by-product without the issues associated with bulk substrate heating, which shortens the precursor residence time and distort pattern fidelity due to thermal drift. Current results show a significant purification of platinum deposits (~35% Pt) with the addition of synchronized laser pulses as well as a significant reduction in deposit resistivity. Measured resistivity from structures fabricated by tSPL is 1.2×10^7 mW-cm whereas 3 orders of magnitude lower than standard EBID platinum structures (2.2×10^5 mW-cm) from the same precursor and are lower than the lowest reported EBID platinum resistivity with post-deposition annealing (1.4×10^4 mW-cm).

3:40pm NS+SP-MoA4 Parallel Stacking of Extended π-conjugated Molecules on Si(100)-H Surface M.Z. Hossain, Gumi University, Japan, H. Kato, Osaka University, Japan, M. Kawai, The University of Tokyo and RIKEN, Japan

Understanding and controlling of intermolecular interaction in highly organized π-conjugated molecules is crucial for the development of the high performance organic devices. Molecule-molecule interaction through frontier orbital states is assumed to be substantially varies with the packing pattern and intermolecular distance. H-terminated Si(100) surface has appeared as an ideal template for controlled stacking of π-conjugated molecules. We have reported controlled formation of some simple acene molecules such as 9-vinyl anthracene, 2-vinyl naphthalene and styrene on H-terminated Si(100) using scanning tunneling microscopy (STM) and ultraviolet photoelectron spectroscopy (UPS).

Controlled stacking of styrene molecules on H-terminated Si(100) surface through chain reaction mechanism is well known. Self-directed growth of molecular stacking is exclusively directed along the dimer row on the surface. With the phenyl rings in the same plane the highest packing density is obtained. Unlike styrene molecule, 9-vinyl anthracene forms both orderly stacking pattern along the dimer row and disorderly nanopattern on the surface. The relative appearances of these orderly and disorderly nanopatterns in STM...
images depend on the applied sample bias while scanning. At lower bias, the orderly pattern appears much brighter than that of the disorderly pattern. This contrast difference between two structures relates to the enhanced π-π interaction in the case of orderly stacked nanopattern. We also observe that the disorderly nanopattern of anthracence molecules undergoes reversed chain reaction very frequently compared to that of styrene molecule.

The 2-vinyl naphthalene molecules form relatively shorter assemblies along the dimer row. Some short disorderly structures are also seen in STM images. UPS measurements of different surfaces containing high-density nanoparticles of these acene molecules have been performed. The observed peaks in UPS spectra are reasonably in agreement with the theoretical calculations reported earlier. The details of the UPS spectra and the reaction mechanism of each molecule forming different nanopatterns will be discussed.

4:00pm
N5+SP-MoA7  Kinetics of disilane reaction on Si(100)-(2×1): Flux, Temperature, and H-coverage Dependence. D. Dick, J.-F. Veyan,
University of Texas at Dallas, P. Mathieu, Mc Gill University, Canada, J.N. Randall, Zyvex Laboratories, Y.J. Chabal, University of Texas at Dallas

Atomic Layer Epitaxy (ALE) is a fundamental process in the Atomically Precise Manufacturing (APM) of nanoscale devices. Si2H6 has been shown to be a good precursor molecule for patterned growth on Si(100) using Chemical Vapor Deposition (CVD). For ALE of silicon, three parameters have to be considered: the incident Si2H6 flux, the temperature, and partial H coverage of the surface. These parameters have been characterized experimentally using in-situ infrared absorption spectroscopy to characterize the nature and coverage of species on a Si (100)-(2×1) surface in a well-controlled environment (2x10-10 Torr base pressure).

The flux dependence was studied over two orders of magnitude and temperature dependence from 173 K to 473 K for varying fluxes. It was found that the nature of SiHx species formed at the surface was strongly dependent on the flux, with higher silanes (e.g. x>1) for higher fluxes. Furthermore, the time required for saturation also decreased with increasing flux and temperature.

The impact of partial H coverage was also investigated with H coverage from 0.01 to 1ML. Monolayer, achieved by partial desorption of a H saturated surface. On the substrate’s surface has been also investigated with FT-IR under UHV conditions. It has been found that even a very small amount of Hydrogen (~1% ML) substantially reduces the chemisorption of disilane on the clean parts of Si(100)-(2×1).

This complex dependence on flux and temperature arises from the complex and highly temperature dependent adsorption/dissociation behavior of disilane on Si(100). The dependence of partial H coverage highlights the spatial requirements of the dissociation products, as uncovered in our earlier work.1


4:20pm
N5+SP-MoA8 SPM Lithography on Silicon Reconstructed and Hydrogen-Passivated Surface. J. Fu, K. Li, N. Pradeep, L. Chen, R. Silver, National Institute of Standards and Technology

We are using Scanning Probe Microscope (SPM) to write patterns directly on a single crystal silicon surface that was reconstructed and hydrogen-passivated in the ultra high vacuum (UHV) chambers, and are followed by the process of reactive ion etching to transfer the patterns into the silicon bulk. The patterns were written either by an atomic force microscope (AFM) in the ambient or by a scanning tunneling microscope (STM) in UHV chambers and was immediately proceeded by an oxidation process in bulk. The patterns were written either by an atomic force microscope (AFM) in the ambient or by a scanning tunneling microscope (STM) in UHV chambers and was immediately proceeded by an oxidation process in bulk. The features patterned by STM, it was conjectured the height were 0.2-0.3nm prior to RIE. After the RIE process, the height was measured to be 0.7nm.

In terms of patterning, the AFM in the ambient environment and the STM in the UHV both can perform lithography; however, the mechanism are quite different: the AFM is adding species, OH, to the surface, and the STM is removing species, H, from the silicon surface.

The features patterned by the AFM prior to RIE process were 1-2nm in height, and were 15-20nm height after RIE and 6nm wide (FWHM) that were measured and confirmed by a critical dimension AFM (CD-AFM). For the features patterned by STM, it was conjectured the height were 0.2-0.3nm prior to RIE. After the RIE process, the height was measured to be 0.7nm.

4:40pm
N5+SP-MoA9 Ultra-High Frequency Surface Acoustic Wave Generation in Silicon Using Inverted Nanoimprint Lithography. S. Byunkkkö, University of Twente, Netherlands, B. Vratzov, NT&D Nanotechnology and Devices, Germany, D. Att, J. van der Veen, University of Twente, Netherlands, P.V. Santos, Paul-Drude-Institut für Festkörperlektronik, Germany, W.G. van der Wiel, University of Twente, Netherlands

Surface acoustic waves (SAWs) are widely used and very important phenomena in both research and industry. SAWs are mechanical vibrations propagating along the surface by confining the acoustic energy over a depth of typically one wavelength. In piezoelectric materials, they can be excited through the inverse piezoelectric effect by using interdigital transducers (IDTs): two interlocking comb-shaped metallic electrode pairs. A piezoelectric potential wave accompanies the mechanical wave. The unique SAW properties make them suitable for wide range of applications. Among the most exciting applications of SAWs are acousto-optical modulators as well as the control of excitons, electrons, and spins in semiconductors. For almost all SAW applications, there is a strong demand for higher frequencies, for example to enhance processing speed or to reach the quantum regime. There are two main factors determining the IDT resonant frequencies: the acoustic properties of the substrate, and the IDT period constrained by lithography resolution. In latter case, the resolution of standard photolithography (minimum finger width of approx. 0.5 um) limits the operation frequency to around a few GHz, even for high acoustic-velocity substrates.

In this study, an alternative lithography technique, “step-and-flash nanoimprint lithography” (SFIL), with a novel material system for lift-off was used to reach very high resolution and higher reproducibility. Hydrogen silsequioxane (HSQ) was used as a planarization layer and an excellent etch stop for removing species, H, from the silicon surface.

This complex dependence on flux and temperature arises from the complex and highly temperature dependent desorption/dissociation behavior of disilane on Si(100). The dependence of partial H coverage highlights the spatial requirements of the dissociation products, as uncovered in our earlier work.1

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Furthermore, the time required for saturation also decreased with increasing flux and temperature.

The impact of partial H coverage was also investigated with H coverage from 0.01 to 1ML. Monolayer, achieved by partial desorption of a H saturated surface. On the substrate’s surface has been also investigated with FT-IR under UHV conditions. It has been found that even a very small amount of Hydrogen (~1% ML) substantially reduces the chemisorption of disilane on the clean parts of Si(100)-(2×1).

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5:00pm
N5+SP-MoA10 Variance of Proximity Effect Correction Parameter Measured on Silicon. D.A. Czaplewski, L.E. Ocola, Argonne National Laboratory

We present data showing that the relative intensity of the backscattered electrons, h, to primary write electrons varies depending on the experimental setup. Our data is consistent with the wide range in values reported in the literature for h. The value of h was determined using a doughnut and cross method [1] where the range of the backscattered electrons was measured to an uncertainty of less than 1%. We find that using this method to determine h provides values that are dependent on the dose of the doughnut. Such a result raises questions on the effectiveness of standard proximity effect correction models (PEC) if they rely on a single h value. Standard PEC models attempt to load level the dose received by the desired pattern in the e-beam resist from all sources of electrons, including the forward and backward scattered electrons during an electron beam exposure.

The relative dose of the backscattered electrons was measured using a set of intersecting lines at the center of tori patterns in a doughnut and cross method [1], (Fig. 1). The backscattered electron dose was varied by changing the dose of the tori with respect to the dose of the intersecting lines as can be seen in Fig. 2. The expected behavior would be a consistent ratio of the backscattered contribution and the line dose; however, as the backscattered electron dose is decreased, the relative contribution to the pattern increases faster than the line dose contribution (Fig. 3). The causes of this phenomenon are not certain yet, but the implication is that it could change existing PEC models. Through further study of this phenomenon, it is possible that the models used in PEC could be updated to accommodate this data and therefore give more accurate dose assignments.

This paper presents the nanopatterning of the hydrogel-gold nanocomposite. We synthesized water based colloidal suspension of covalently bound gold nanoparticles (5-10nm) on the backbone of Poly(Allyl amine) by reduction of gold salt on the amine groups of polymeric chain. Poly(N-isopropylacrylamide) was dissolved in Poly(Allyl amine) based gold colloidal solution. This solution then spun-coated on Hexamethyldisilazane primed Si wafer for nanolithography. The hydrogel precursor was dried in air. Resulting thickness of the hydrogel precursor was approximately 1 mm. The samples were then exposed to 110-180 μC/cm² e-beam. The exposure dose was optimized for different thickness of precursor. Different characterization techniques such as UV-vis spectroscopy, differential scanning calorimetry, FTIR, scanning electron microscopy, and atomic force microscopy were employed for characterization. Thus, gold nanoparticles were incorporated into a three dimensional, cross-linked, polymeric hydrogel network by nanopatterning technique. With the ability of hydrogels to expand or contract with changes in pH and temperature, the spacing between nanoparticles can be controlled, allowing a single material being able to resonate at different frequencies. The ability to perform lithography to form a patterned hydrogel with homogeneous nanoparticles opens the door to development of nanoswitches, sensors, MEMS as well as drug delivery devices.
Graphene and Related Materials Focus Topic
Room: 13 - Session GR+AS+NS+SP+SS-TuA

Graphene Characterization Including Microscopy and Spectroscopy
Moderator: J.C. Hone, Columbia University

2:00 pm GR+AS+NS+SP+SS-TuA1 High Resolution Real and Reciprocal Space Photoelectron Emission Microscopy on Heterogeneous Graphene/SiC(000-1). K. Winkler, B. Kromker, 1Omicron NanoTechnology, Germany, N. Barrett, IRAMIS, Selaclay, France, E. Conrad, GeorgiaTech

We present energy filtered electron emission spectromicroscopy with high spatial and wave-vector resolution on few-layer epitaxial graphene on SiC(000-1) grown by furnace annealing.

Conventional electron spectroscopy methods are limited in providing simultaneous real and reciprocal or k-space information from small areas under laboratory conditions. Therefore, the characterization of materials with only micron scale sample homogeneity such as epitaxially grown graphene requires new instrumentation. Recent improvements in aberration compensated energy-filtered photoelectron emission microscopy (PEEM) can overcome the known limitations in both synchrotron and laboratory environments. Here we report 2D maps of the k-parallel π±σ band dispersion in micron-scale regions and correlate them with spatially resolved chemical information on the same regions. Only the combination of high lateral, high energy, high k-resolution and controlled switching between real space and k-space allows detailed understanding of micron size sample sites with 1–3 layers graphene. The experiments underline the importance of simultaneous lateral, wave vector and spectroscopic resolution on the scale of future electronic devices in order to precisely characterize the transport properties and band alignments.


As silicon-based electronics are reaching the nanosize limits of the semiconductor roadmap, carbon-based nanoelectronics has become a rapidly growing field, with great interest in tuning the properties of carbon-based materials. Chemical functionalization is a proposed route, but syntheses of graphene oxide (GO) produce disordered, nonstoichiometric materials with poor electronic properties. We report synthesis of an ordered, stoichiometric, solid-state carbon oxide that has never been observed in nature and coexists with graphene. Formation of this material, graphene monoxide (GMO), is achieved by annealing multilayered GO. A combination of transmission electron microscopy and infrared microspectroscopy have provided critical experimental evidence to identify the novel structure. These results indicate that the resulting thermally reduced G-O (TRG-O) consists of a two-dimensional nanocrystalline phase segregation: unoxidized graphicite regions are separated from highly oxidized regions of GMO. GMO has a quasi-hexagonal unit cell, an unusually high 1:1 O:C ratio, and a calculated direct band gap of approximately 0.9 eV.

This work was supported by the NSF (CMMI-0856753 and CMMI-0900509). This work is based upon experiments performed at the Synchrotron Radiation Center. The SRC is funded by the University of Wisconsin-Madison and the University of Wisconsin-Milwaukee. Work performed at the SRC IRENI beamline be done with support from an NSF Major Research Instrumentation grant (DMR-0619759). The authors thank Bruker Technologies for the Grazing Angle Objective used for this work.


2:40 pm GR+AS+NS+SP+SS-TuA3 Scanning Tunneling Spectroscopy of Epitaxial Graphene: Local Band Mapping and Wavefunction Engineering. P.N. First, Georgia Tech

Because the crystalline orientation is determined prior to growth, epitaxial graphene (EG) on silicon carbide is an excellent material to consider for 2D wavefunction engineering, where device properties are designed through wavefunction confinement and material strain. In pursuit of this goal, we use scanning tunneling microscopy (STM) and spectroscopy (STS) to characterize the local structural and electronic properties of EG and a simple EG nanostructure. With some care, STS can be used to measure the full energy-momentum dispersion of both filled and empty states, on length scales determined by the coherence of the graphene wavefunctions. Applying a magnetic field introduces a field-tunable comb of discrete Landau level energies that we use to obtain high momentum resolution, to characterize the tip-induced surface potential, and to detect subtle interlayer interactions in a multilayer graphene stack. * Work performed in collaboration with NIST Center for Nanoscale Science and Technology. ** Funded in part by NSF and by NRI-INDEX.

4:00 pm GR+AS+NS+SP+SS-TuA7 Intercalation of O2 and CO Controlled by the Mesoscopic Structure of Graphene. E. Grånäs, J. Knudsen, S. Göthlin, U. Schröder, C. R. B. Busse, Universität zu Köln, Germany, M.A. Arman, K. Schulte, J.N. Andersen, Lund University, Sweden, T.W. Michely, Universität zu Köln, Germany

Intercalation of gases between epitaxial graphene and its substrate has become a topic of interest for studies due to, for example, the unique opportunities to modify the graphene-substrate interaction and the possibilities to perform chemistry under the graphene layer. Further, a profound knowledge about the intercalation process is of great importance for graphene related devices. In this work, we investigate the CO oxidation and O2 intercalation to graphene by using a combination of X-ray photoemission spectroscopy (XPS) and scanning tunneling microscopy (STM) to observe the graphene edge at elevated temperatures and pressures is essential for, among other things, the correct interpretation of gas adsorption studies on graphene supported metal cluster arrays.

We have studied intercalation and etching of Ir(111) supported graphene upon gas exposure to common gasses such as O2 and CO in the entire pressure interval from 100 to 0.1 mbar. Comparing perfect graphene layers without holes with graphene films, that only covers a fraction of the Ir(111) surface, we reveal that the holes - or more specific the graphene edges - are essential for intercalation.

For oxygen exposed graphene we develop a coherent picture of temperature dependent oxygen etching and intercalation. Using X-ray photoemission spectroscopy (XPS) and scanning tunneling microscopy (STM) we show that a perfect graphene layer is stable against etching and intercalation up to 700 K, whereas at higher temperatures etching, but no intercalation, takes place. In contrast, a partial graphene coverage on Ir(111) enables dissociative oxygen adsorption on the bare Ir and subsequent intercalation underneath graphene flakes at 355 K and above. Intercalated oxygen remains stable up to a temperature of 600 K, above this temperature it desorbs in the form of CO or CO2. We have determined XPS and STM fingerprints for the intercalated oxygen structure and we unambiguously assign it to a p(2x1)-O structure similar to the one observed on clean Ir(111). The decoupling of the graphene-graphene interface in the metal substrate is directly visualized through the inability to form well-ordered Pt cluster arrays on the O-intercalated areas of graphene on Ir(111). Further, we have identified the rate limiting step for oxygen intercalation to be unlocking of the graphene edge and propose that this takes place through bond breaking between graphene edge bonds and the Ir substrate.

Using a combination of high pressure X-ray photoemission spectroscopy (HP-XPS) and STM we also show that CO intercalation takes place at room temperature and pressures up to 1 mbar range. 1 mbar range for intercalated CO is determined to be (3\(\sqrt{3}\times\sqrt{3})R\bar{3}0\) identical to the structure observed on clean Ir(111) upon high pressure CO exposure.

4:20 pm GR+AS+NS+SP+SS-TuA8 Long-range Atomic Ordering and Variable Interlayer Interactions in Two Overlapping Graphene Lattices with Stacking Misorientations. T. Ohta, T.E. Beecham, Sandia National Laboratories, J.T. Robinson, Naval Research Laboratory, G.L. Kellogg, Sandia National Laboratories

We report a method to examine the effect of stacking misorientation in bilayer graphene by transferring chemical vapor deposited (CVD) graphene onto monolithic graphene epitaxially grown on silicon carbide (SiC) (0001). The resulting hybrid bilayer graphene displays long-range Moiré diffraction patterns having various misorientations even as it exhibits electron reflectivity spectra nearly identical to epitaxial bilayer graphene grown directly on SiC. These varying twist angles affect the 2D (G')-band shape of the Raman spectrum indicating regions of both a monolayer-like single π state and Bernal-like split π states brought about by the differing interlayer interactions. This hybrid bilayer graphene fabricated via a transfer process therefore offers a means to systematically study the electronic properties of bilayer graphene films as a function of stacking misorientation angle.

The work at Sandia National Laboratories was supported by LDRD and by the US DOE Office of Basic Energy Sciences, Division of Materials Sciences and Engineering. Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-
5:00pm GR+AS+NS+SP+SS-TuA9 Formation of Graphene on SiC(0001) (E. He)

Northwestern Univ. B. Detslefs, J.M.P. Alaboson, Myers-Ward, C.R. Eddy, Jr., D.K. Gaskill, the formation of the so-called “buffer layer” at the interface, which is known to affect the electronic properties of the graphene. Despite numerous efforts to determine the nature of the buffer layer, debate persists concerning its atomic and chemical structure. Here, we use the X-ray Standing Wave (XSW) technique to create a precise chemically-sensitive description of the distributions of Si and C at the interface. This technique, which combines X-ray scattering and X-ray Photoelectron Spectroscopy (XPS), is capable of locating coherent distributions of chemically distinct species above a single crystal surface. This allows for a more detailed description of the interface than those afforded by scattering or XPS alone. Our analysis shows that the buffer layer, which is present in both UHV and furnace-grown EG/SiC(0001), contains no substantial non-bulk or oxide silicon component, and is thus purely carbon. We identify two chemically distinct carbon species within the interface layer, each with a distinct location above the Si-terminated surface, and report their positions and distributions with sub-angstrom precision. These results help to clarify long-standing uncertainties about the interfacial structure of graphene/SiC(0001). Further, we also highlight the potential for XSW with XPS as a valuable tool in the structural determination of complex interfaces, such as functionalized, doped, or intercalated epitaxial graphene.

5:00pm GR+AS+NS+SP+SS-TuA10 Formation of Graphene on SiC(0001) in Disilane and Neon Environments (M. J. Bedzyk, Carnegie Mellon University)

We have prepared graphene on the SiC(0001) surface (the so-called C-face of the (0001) surfaces), by heating the SiC in a Si-rich environment produced either by using disilane (=10^4 Torr) or cryogenically-purified neon (1 atm). With the Si-rich environments, we obtain considerably better uniformity in the thickness for thin, ~ML-thick graphene on the C-face compared to that observed in samples prepared in vacuum or in an argon environment. We also find that different interface structures occur in these environments. In particular, we find a graphene-like buffer layer forming at the interface, analogous to the well known behavior of the SiC(0001) surface (the Si-face).

Studies are performed using atomic force microscopy (AFM), low-energy electron diffraction (LEED), and low-energy electron microscopy (LEEM). For the heating preparation in vacuum, LEED patterns show a characteristic close-packed pattern together with graphene streaks. In contrast, for the graphene produced in either the disilane environment (=10^4 Torr) or 1 atm of neon, LEED patterns reveals a complex \( 4\sqrt{3} \times 4\sqrt{3} - R\sqrt{3} \) arrangement along with graphene spots. This structure is somewhat similar to the well known 6\( \times 6 \) \( 2\sqrt{3} \times 2\sqrt{3} - R\sqrt{3} \) “buffer layer” of the Si-face, with satellite spots surrounding the primary Si spots, and is interpreted as arising from a C-rich buffer layer on the SiC. Selected area diffraction on these surface areas reveals a wavevector magnitude precisely equal to that of graphene, thus proving that the buffer layer does indeed have structure very close to that of graphene (the pattern is interpreted as a distortion of the buffer-layer graphene due to bonding to the underlying SiC). Using LEEM, measurements from the buffer layer of the reflected intensity of the electrons as a function of their energy reveal a new characteristic reflectivity curve, not seen for vacuum-prepared graphene.

After oxidation of the samples, the \( 4\sqrt{3} \times 4\sqrt{3} - R\sqrt{3} \) and \( 3\sqrt{3} \times 3\sqrt{3} - R\sqrt{3} \) spots appear on the surface. This latter behavior is interpreted as oxidation of the SiC surface beneath the buffer layer. Selected area diffraction on portions of the surface that were previously identified as buffer layer still reveal a wavevector magnitude precisely equal to that of graphene. However, LEEM reflectivity curves on those areas reveal a complex new spectrum, indicative of the so-called “decoupling” of the buffer layer from the SiC. This decoupling is consistent with our interpretation of this new interface structure as being a graphene buffer layer on C-face SiC.

This work is supported by NSF.

Scanning Probe Microscopy Focus Topic Room: 16 - Session SP+AS+BI+ET+MI+NS-TuA

Advances in Scanning Probe Imaging

Moderator: S. Allen, The University of Nottingham, UK, Z. Gai, Oak Ridge National Laboratory

2:00pm SP+AS+BI+ET+MI+NS-TuA1 Molecules Investigated with Atomic Resolution using Scanning Probe Microscopy with Functionalized Tips (L. Gross, F. Mohn, N. Moll, G. Meyer, IBM Research - Zurich, Switzerland)

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Single organic molecules were investigated using scanning tunnelling microscopy (STM), noncontact atomic force microscopy (NC-AFM), and...
Kelvin probe force microscopy (KPFM). With all of these techniques submolecular resolution was obtained due to tip functionalization by atomic manipulation. The techniques yield complementary information regarding the molecular structural and electronic properties.

Using NC-AFM with CO terminated tips, atomic resolution on molecules has been demonstrated and the contrast mechanism was assigned to the Pauli repulsion [1]. On the other hand, by using STM the molecular frontier orbitals, i.e., the highest occupied and the lowest unoccupied molecular orbitals (HOMO and LUMO), were mapped [2]. Using a CO terminated tip for orbital imaging with the STM, the resolution can be increased and the image corresponds to the projection of the gradient of the molecular orbitals due to the p-wave character of the tip states [3]. Finally, KPFM reveals information about the distribution of charges within molecules by measuring the z-component of the electrostatic field above the molecule, as demonstrated on the hydrogen tautomerization switch naphthalocyanine [4].

References:

3:00pm SP+AS+BI+ET+MI+NS-TuA3 Functional Imaging of Jahn-Teller Dynamics at the Single-Molecule Scale. J. Lee, S.M. Perdue, A. Rodriguez Perez, P.Z. El-Khoury, V.A. Apkarian, University of California, Irvine

Taking advantage of both elastic and inelastic tunneling processes of a molecule isolated at the double-barrier tunneling junction of a scanning tunneling microscope, both static and dynamic parts of the Hamiltonian can be visualized with submolecular resolution. This is illustrated by imaging Jahn-Teller (JT) driven vibronic dynamics within Zn-etiroporphyrin (ZnEtio), in its various reduced forms, in what may be regarded as nature’s choice of a molecule as a controllable current switch. Unique interpretations are afforded through simultaneously recorded functional images, such as maps of: a) energy resolved differential current, b) spectrally resolved electroluminescence, c) conduction bistability, d) reduction oxidation potentials (maps of charging and discharging). We focus on the radical anion, ZnEtio‘, which is reduced by injecting an electron to a single ZnEtio molecule adsorbed on a thin aluminum oxide film grown on NiAl(110). In contrast with the neutral, the saddle-shaped radical anion lies flat on the surface of the oxide, the exchange magnetic interaction is directly mapped, and an excess electron is localized in the p orbital of the entire porphyrin macrocycle, as a result of the JT active rectangular (B1g) distortion of the molecule. The static JT potential leads to conduction bistability, with reversed switching polarity depending on whether tunneling electrons are injected in the occupied p orbital or the diamond (B1g) coordinate which serves as a transition state that connects the p and p orbitals at the two B1g minima. In addition to the JT switching, the dynamic JT states are directly imaged through electroluminescence spectra, induced by injection of a second electron in the anion. The spectra consist of a continuum due to radiative ionization of the dianion, and sharp Fano resonances of the vibronic progression of the JT active modes. A detailed analysis of the spectra yields the vibronic couplings and the wavefunctions. Vibronic structure is inherent in STM topographic images, and has hitherto not been fully recognized.

4:00pm SP+AS+BI+ET+MI+NS-TuA4 Atomic and Chemical Resolution of Heterogeneous 1-D Metallic Chains on Si(100) by Means of nc-AFM and DFT. M. Setvin, M. Ondracek, P. Mutombo, Z. Majzik, P. Jelinek, Institute of Physics of ASCR, Czech Republic

Scanning Probe techniques are widely used to image atomic and electronic structure of surfaces and nanostructures. However atomic and chemical resolution of complex nanostructures (e.g. molecules, nanoparticles or nanowires) is still a great challenge. Several methods (see e.g. [1]) have been already proposed to achieve the single-atom chemical resolution. In the work [3] it was showed that the single-atom chemical identification can be achieved via force-spectroscopy measurements using Frequency Modulation Atomic Force Microscopy (FM-AFM). The validity of the method was demonstrated on semiconductor surface alloy composed of isovalent species (Si3N4 and Pb). In this particular case, the valence electrons of surface atoms possess very similar electronic structure close to sp3 hybridization with characteristic dangling bond state. Hence the maximum short-range force is mainly driven by the position of the dangling bond state with respect to the Fermi level.

In this work, we investigated atomic and chemical structure of heterogeneous 1-D chains made of III and IV group metals grown on Si(100) surface [4] by means of room-temperature (RT) FM-AFM measurements combined with DFT simulations. Here 1D chains consist of heterogeneous buckled-dimer structures with unknown chemical ordering. What more, the presence of buckled dimmers composed by chemical species of different valence makes this system very challenging for true atomic and chemical resolution by means of SPM.

In this contribution, we will show first that FM-AFM technique even at RT is able to achieve atomic resolution of individual atoms forming dimmers, much superior to the contrast obtained by the traditional STM technique. Secondly, we will demonstrate that the single-atom chemical identification is still possible combining the force-spectroscopy at RT with DFT simulations even in such complex systems as the heterogeneous 1D metallic chains.

References:

4:00pm SP+AS+BI+ET+MI+NS-TuA7 Simple Routes to High Speed and Super Resolution AFM. J.K. Hobbs, University of Sheffield, UK

INVITED

Over the past two decades atomic force microscopy has developed to become the workhorse of molecular nanotechnology. However, despite this success, it has failed to deliver consistently in two areas where it arguably has most potential, namely sub-molecular resolution imaging and the following of processes in real time. Here our work to tackle these challenges will be discussed.

We have developed a new approach to reaching high resolution within a conventional AFM, based on torsionally driven T-shaped cantilevers, dubbed “torsional tapping AFM”. The use of torsional oscillations gives improved mechanics (high Q-factor, high frequency), without excessively increasing the spring constant. The small offset of the tip from the axis of rotation gives improved lever sensitivity. Combined, these result in an approximately 12 fold improvement in sensitivity when compared to the same AFM with a conventional tapping cantilever. This improved sensitivity allows ultra-sharp whisker tips to be used in a routine manner, giving true molecular resolution even on soft materials measured using tens of nanometres of topography. For example, individual polyethylene chains both in the crystalline phase, and at the interface with the amorphous phase, can be clearly imaged in a conventionally processed sample of plastic, with polymer chain-to-chain resolution down to 0.37 nm [1].

Data from semi-crystalline polymers to naturally occurring protein crystals will be presented.

High speed AFM requires methods for scanning rapidly, for maintaining tip-sample contact (“feedback”), and for constructing the topography image. We have shown that resonant scanners [2] give a robust method for rapid scanning. In a conventional AFM the feedback and the topographic image are intrinsically linked. However, this places a limit on scan speed as it demands that the tip has reached equilibrium at each point on the image if the height is going to be accurately obtained. We have adopted a different approach, in which the height of the tip is directly measured using an interferometric approach, freeing the feedback loop to minimise tip-sample forces. This allows topographic images with height traceable to the wavelength of the interferometric laser to be obtained at imaging rates greater than one frame a second. Coupled with resonant scanners, giving scan areas up to 40×40 μm an AFM platform capable of in-line industrial applications is obtained.

with similar molecular spacings and displayed an island growth morphology.

We have achieved an excellent resolution on an ambient running AFM. We have demonstrated that the combination of STM and AFM is essential, if not a must, to look at ultimate monolayers in the ambient conditions. Overall a facile green chemistry method for TMA monolayer fabrication from aqueous media on a HOPG surface has been established.

5:00pm SP+AS+BI+ET+MI+NS-TuA10 Understanding the Role of the Probe in SPM Imaging of Metal Oxides: New Opportunities for In-Depth Surface Analysis, H. Möntig, Univ. of Münster, Germany; M. Todorovic, Univ. Autónoma de Madrid, Spain; M.Z. Baykara, Yale Univ.; T.C. Schwindemann, Southern Connecticut State Univ., J. Götzen, Ö. Üverdi, E.I. Altman, Yale Univ.; R. Perez, Univ. Autónoma de Madrid, Spain; U.D. Schwarz, Yale Univ.

Metal oxide surfaces play an indispensable role in a number of catalytic processes of technological and scientific importance. A fundamental understanding of the role that metal oxide surfaces play in such applications requires an experimental technique that allows analyzing chemical and electronic surface properties down to the atomic scale. The powerful method of three-dimensional atomic force microscopy (3D-AFM) in combination with scanning tunneling microscopy (STM) can be used towards this goal with great success. However the interpretation of results is not straightforward, particularly because the structure and chemistry of the probe tip employed in the experiments influences the measured data.

In this talk, using a combination of experimental STM data and density functional theory (DFT) calculations, we will study the effect of changing the tip structure and chemistry, as well as imaging parameters such as tip-sample distance and bias voltage on STM images obtained on the model surface of Cu(100)-O, a surface oxide layer consisting of nearly co-planar copper (Cu) and oxygen (O) atoms. We observe that STM image contrasts and atomic species with highest tunneling probability vary greatly with changing tip properties and imaging parameters. Reasonable matches between calculated and experimentally recorded STM images are observed, allowing the determination of particular tip models used in the experiments. Additionally, the effect of rotating the model tip structures with respect to the sample surface results in asymmetric features in simulated STM images, reproducing certain peculiar patterns observed experimentally. To sum up, the results presented here underline the significant role that the tip plays in SPM measurements and describe potential routes to optimize the gathered information through deliberate manipulation of tip properties as well as imaging parameters.

5:20pm SP+AS+BI+ET+MI+NS-TuA11 Characterizing the Best Tips for NC-AFM Imaging on Metal Oxides with Force Spectroscopy and Theoretical Simulations, D. Fernandez-Torre, Universidad Autónoma de Madrid, Spain; A. Yurtsever, Osaka University, Japan; P. Fou, Universidad Autónoma de Madrid, Spain; Y. Sugimoto, M. Abe, S. Morita, Osaka University, Japan, R. Perez, Universidad Autónoma de Madrid, Spain.

Metal oxides play a key role in a wide range of technological applications. To optimize their performance, it is essential to understand their surface properties and chemistry in detail. Noncontact atomic force microscopy (nc-AFM) provides a natural tool for atomic-scale imaging of these insulating materials. Some of these materials, including ceria (CeO2), and particularly titania (TiO2), have been extensively studied with nc-AFM in the last few years. Experiments on the rutile TiO2(110) surface show, at variance with STM, that a variety of different contrasts can be obtained, and frequent changes among different imaging modes are observed during scanning. The two most common contrasts are the "protrusion" and the "hole" mode imaging modes, that correspond, to imaging bright the positive or the negative surface ions respectively, but other contrasts like the "neutral" mode and the "all-inclusive" mode—where all the different chemical species and defects are imaged simultaneously—have been also identified.

Understanding the image contrast mechanisms and characterizing the associated tip structures is crucial to extract quantitative information from nc-AFM measurements and to identify the nature of the observed defects. While in many cases the same nc-AFM image can be explained by different models, and even different underlying tip-sample interactions, we show here that the combination of force spectroscopy (FS) measurements and first-principles simulations can provide an unambiguous identification of the tip structure and the image contrast mechanism. In particular, we show that the best tips to explain the protrusion and hole mode forces are TiOx-based clusters differing in just one H atom at the tip apex, discarding previously proposed Ti-terminated tips that would lead to forces much larger than the ones observed in the experiments. The less frequent neutral and all-inclusive images are associated to Si tips where contamination is limited to just an O atom or OH group at the apex. These models provide a natural explanation for the observed contrast reversals by means of H transfer to/from the tip, an event that we indeed observe in our simulations.

As tip contamination by surface material is common while imaging oxides, we expect these tips and imaging mechanisms to be valid for other oxides. Our results for the imaging of CeO2 surfaces and of metal atoms (K, Pt) adsorbed on TiO2 support this conclusion.

5:40pm SP+AS+BI+ET+MI+NS-TuA12 Direct Probe of Interplay between Local Structure and Superconductivity in FeTe0.55Se0.45, M.H. Pan, W.Z. Lin, Q. Li, B.C. Sales, S. Jesse, A.S. Sefat, S.V. Kalinin, Oak Ridge National Laboratory.

A key challenge in high-temperature superconductivity is to determine the role of local crystallographic structure and chemical effects on the superconducting critical temperature, Tc. Iron chalcogenide superconductors (‘111’) are ideal model systems for deciphering the role of local effects on the superconductivity, primarily because they cleave leaving non-polar surfaces unlike other families of iron arsenide superconductors (‘1111’ or ‘122’) and cuprates. Here, we explore the interplay between local crystallographic structure, composition and local electronic and superconductive properties. Direct structural analysis of scanning tunneling microscopy (STM) data allows local lattice distortions and structural defects across a FeTe0.55Se0.45 surface to be explored on a single unit-cell level. Concurrent superconducting gap (SG) mapping reveals suppression of the SG at well-defined structural defects, identified as a local structural distortion (Gainer-Preston zone). The strong structural distortion is related to the vanishing of the superconducting state. This study provides insight into the origins of superconductivity in iron chalcogenides by providing an example of atomic-level studies of the structure-property relationship.
Scanning probe microscopy (STS) was used to collect barrier height data on the molecule Indolo[2,1-b]quinazoline-6,12-dione (tryptanthrin) at the solution-graphite interface. Tryptanthrin and its analogues have shown good promise as potential therapeutic agents for a variety of pathogenic organisms. In this experiment, apparent barrier height ‘$\Phi_{ap}$’ (work function) values were collected at sub-molecular resolution. The STM topography and barrier height data are collected simultaneously so that the barrier height data can be compared with the topography of the molecule. Since a positive sample bias was used throughout the experiment, the topography corresponds to the lowest unoccupied molecular orbital (LUMO) of the molecule. These topography and barrier height image data are compared with the density functional theory (DFT) computed molecular orbital of tryptanthrin to ascertain the positions of various lobes of the molecule. These data from barrier height images correspond to the amount of energy required by electrons to enter or leave different parts of the molecule, which might be indicative of the likely locations of redox events.
Nanoscale Catalysis and Surface Chemistry
Moderator: U.D. Schwarz, Yale University

8:20am NS+AS+SS+SP-WeM2 Preparation and Performance of Solid Rocket Propellant Containing In Situ-Synthesized Nanoparticle Catalysts, and Fabrication of Palladium Nanoparticles for Use in Rocket Propulsion Systems, R. Tenne, University of California, Berkeley, M. Feldman, University of California, Berkeley, and J. Zhao, University of California, Berkeley

9:00am NS+AS+SS+SP-WeM4 Synthesis and Catalytic Activity of WS2 Nanotube Supported Cobalt and Nickel Catalysts Towards Thiophene Hydrodesulfurization, M. Komarneni, Z. Yu, A. Chakradhar, U. Burghaus, North Dakota State University, Y. Tsverin, R. Popovitz-Biro, Y. Feldman, R. Tenne, Weizmann Institute of Science, Israel

Inorganic nanotubes (INT) including WS2: INT are promising materials for heterogeneous catalysis due to their intriguing properties like enhanced surface area, defects, and confinement effects. The promotion effects of Co and Ni combined with novel nanomaterials such as INT-WS2 may create the next generation hydrodesulfurization (HDS) catalysts. To verify this, M/INT-WS2 (M = Co or Ni) catalysts were synthesized and their catalytic activity towards HDS of thiophene was characterized by gas chromatography/ambient pressure catalytic tests and ultra-high vacuum (UHV) thermal desorption spectroscopy (TDS) experiments. Synthesis of M/INT-WS2 involved two steps: Surface activation of INT-WS2 by palladium seeding process and electroless plating method to coat nanoparticles of M. The deposited nanoparticles of M formed non-uniform layer on the INT surface. Nanoparticles of size 10-20 nm for Co (hcp structure) and 10-20 nm for Ni (fcc structure) were coated on INT-WS2. Next, the catalytic activity of M/INT-WS2 towards thiophene HDS was characterized using an atmospheric flow reactor. M/INT-WS2 catalysts exhibited enhanced HDS activity when compared to pristine INT-WS2, mainly due to the promotion effects of Co and Ni. Hydrogen sulfide and hydrocarbons such as 1,3-butadiene, butane, cis-2-butene, and trans-2-butene were formed as HDS products by both pristine and M/INT-WS2. Commercial HDS catalysts, CoMo and NiMo from Haldor Topsoe were found to show ~4 times higher activity than M/INT-WS2 synthesized in this study. These results are promising and show that further optimization of the nanofabrication process yields better HDS nanocatalysts. Furthermore, the adsorption kinetics of thiophene on M/INT-WS2 was studied by TDS at UHV conditions. Thiophene adsorbed on internal, external, and groove sites of the M/INT-WS2 bundles. Binding energies of thiophene on Ni/INT-WS2 are ~10 kJ/mol smaller than that of pristine INT-WS2. Thiophene also decomposed on M/WS2, NT at UHV conditions. In addition, catalytic screening of nanocatalysts such as Au and Co-Ni coated INT-WS2, pristine MoS2 nanoparticles with fullerene-like structure (IF), and Re-doped IF-MoS2 for HDS was also performed. Overall, M/INT-WS2 catalysts were the best HDS catalysts among the new nanocatalysts studied. The results also reflected the promotion effects of Co and Ni on the M/INT-WS2 catalysts for higher thiophene conversion rates.

11:20am NS+AS+SS+SP-WeM11 Charge Exchange and Molecule/Metal Coupling in Fulvalene Surface Chemistry, G. Rosas, B.G. Sumpter, Oak Ridge National Laboratory, J.A. Schlueter, Argonne National Laboratory, P. Maksymovych, Oak Ridge National Laboratory

Understanding the epitaxy of organic semiconductors on the surface, and the ensuing processes of charge transfer and band-alignment is crucial for the deterministic design of energy harvesting and light-emitting devices based on molecular heterojunctions. While most of the attention so far has been directed to pi-conjugated aromatic compounds, little is known about the properties of the fulvalene family in contact with metal surface. Here we will present a spectroscopic study of bis(ethylenedithio)tetrafulvalene (ET) on Ag(111) in the sub-monolayer to monolayer coverage. Varying coverages of ET adsorption show the molecules dimerize in parallel, bonding to the Ag surface along the long-axis of the molecule. The dimers remain mobile after adsorption, resulting in the formation of a two-phase surface material: unidimensional loosely stacked nanoclusters and finely packed, two-dimensional domains of interlocked molecules. These structures are an intermediate kinetic state, as the molecules further chemically react with the underlying Ag surface following annealing to temperatures as low as 40 C. It is thought based on these data that the dimers form chemical bonds with a single, shared Ag adatom upon adsorption, as observed for other pi orbital dominated aromatic molecules such as PTCDI. Formation of a reactive layer has significant implications for the orbital alignment at the interface. We have therefore probed the properties of the 2D ordered layer and the reacted layers using a combination of current-distance and image-potential state spectroscopy. The interpretation of these results will be presented in conjunction with the first-principles calculations of the respective structures, and correlated with the induced density of interface states (IDIS) model for orbital alignment at metal-molecule interface.

This research was conducted at the Center for Nanophase Materials Sciences, sponsored at the Oak Ridge National Laboratory by the Division of User Facilities, U.S. Department of Energy.

11:40am NS+AS+SS+SP-WeM12 Subpicosecond-pulse Photoinduced Chemistry on Nanoscale Palladium Model Catalyst Surfaces, A. Bhattacharya, Brookhaven National Laboratory, R. Palermo, J.C. Lofaro, Stony Brook University, H. Park, M.G. White, N. Camillone, Brookhaven National Laboratory

To date, time-resolved investigations of surface chemical reaction dynamics have almost exclusively been conducted on metal single crystals. However, current and proposed catalysts and photocatalysts generally consist of nanometer-scale metal particles supported on metal oxides. To conduct time-resolved investigations of the surface chemical dynamics of such systems we have synthesized and characterized arrays of palladium nanoparticles (approximately 4 to 10 nm in diameter) supported on clean rutile TiO2(110). We will present our synthetic approach and the results of chemical and morphological characterization and thermal chemistry experiments on these arrays. We will also discuss the results of subpicosecond-pulse photoinduced desorption of molecular oxygen and carbon monoxide, as well as the photoinduced bimolecular reaction between adsorbed atomic oxygen and carbon monoxide. Comparisons to the same reactions on single crystal Ptd(111) surfaces will be made, and the unique features of the chemistry and dynamics at the nanoscale highlighted.

Scanning Probe Microscopy Focus Topic
Room: 16 - Session SP+AS+BI+ET+MI+NM+SS+TF-WeM

Probe-Sample Interactions, Nano-Manipulation and Fabrication
Moderator: S. Allen, The University of Nottingham, UK, A.-P. Li, Oak Ridge National Laboratory

8:00am SP+AS+BI+ET+MI+NM+SS+TF-WeM Controlled Coupling of Silicon Atomic Quantum Dots at Room Temperature: A Basis for Ultracompact Electronic Devices, R.A. Holowka, University of Alberta and The National Institute for Nanotechnology, Canada, J. Pitters, The National Institute for Nanotechnology, Canada, G. DiLabio, M. Taucer, P. Piva, L. Livadaru, University of Alberta and The National Institute for Nanotechnology, Canada

INVITED Quantum dots are small entities, typically consisting of just a few thousands atoms, that in some ways act like a single atom. The constituent atoms in a dot coalesce their electronic properties to exhibit fairly simple and potentially very useful properties. It turns out that collectives of dots exhibit joint electronic properties of yet more interest. Unfortunately, though extremely small, the finite size of typical quantum dots puts a limit on how close multiple dots can be placed, and that in turn limits how strong the coupling between dots can be. Because inter-dot coupling is weak, properties of interest are only manifest at very low temperatures (milliKelvin). In this work the ultimate small quantum dot is described – we replace an “artificial atom” with a true atom - with great benefit.
It is demonstrated that the zero-dimensional character of the silicon atom dangling bond (DB) state allows controlled formation and occupation of a new form of quantum dot ensembles - at room temperature. Coulomb repulsion causes DBs separated by less than ~2 nm to experience reduced localized charge. The unoccupied states so created allow a previously unobserved electron tunnel-coupling of DBs, evidenced by a pronounced change in the time-averaged view recorded by scanning tunneling microscopy. It is shown that fabrication geometry determines net electron occupation, and tunnel-coupling strength within multi-DB ensembles and moreover that electrostatic separation of degenerate states allows controlled electron occupation within an ensemble.

Some speculation on the viability of a new “atomic electronics” based upon these results will be offered.

9:20am

**SP+AS+BI+ET+MI+NM+NS+SS+TF-WeM4** Atomic Forces and Energy Dissipation of a Bi-Stable Molecular Junction, C. Lotze*, L. Grosset al., Freie Universität Berlin, Germany.

Tuning Fork based dynamic STM/AFM is a well established method combining the advantages of scanning tunneling and dynamic force microscopy. Using tuning forks with high stiffness, stable measurements with small amplitudes, below 1 Å are performed. In this way, conductance and frequency shift measurements of molecular junction can be obtained simultaneously [1] with intramolecular resolution [2].

One of the most intriguing aspects of molecular junctions relates to the effect of structural bi-stabilities to the properties of the junction. These lead, for example, to conductance fluctuations, telegraph noise and the possibility to switch the electrical transport through the junction.

In this presentation, we characterize a model bi-stable molecular system using dynamic force spectroscopy. The effect of current-induced stochastic fluctuations of conductance are correlated with fluctuations in force. In our experiment we identified the last from both, frequency shifts and energy dissipation measurements, picturing a regime in which electrical transport and mechanical motion are coupled.


9:30am

**SP+AS+BI+ET+MI+NM+NS+SS+TF-WeM5** Acetylene on Cu(111): Imaging a Molecular Pattern with a Constantly Rearranging Tip, Y. Zhu*, J. D. Parkin, et al., University of California Riverside.

In-situ scanning tunneling microscopy. It is shown that fabrication geometry determines net electron occupation, and tunnel-coupling strength within multi-DB ensembles and moreover that electrostatic separation of degenerate states allows controlled electron occupation within an ensemble.

Abstract: Using variable temperature STM and DFT simulation, we identify the phases of acetylene adsorbed on the Cu(111) surface. Depending on the coverage, a diffusion-derived surface pattern of acetylene on Cu(111) is validated by STM. The modification of the STM image transfer function through the adsorption of an acetylene molecule onto the tip apex is taken into account. In this case, the images of acetylene patterns on Cu(111) also include direct evidence of the rotational orientation and dynamics of the acetylene species attached to the tip apex. DFT modeling of acetylene/Cu(111) reveals that the molecular orientation and separation is governed by a balance of repulsive interactions associated with stress induced in the top surface layer and attractive interactions mediated by the electronic structure of the substrate. Computationally modeling of the substrate with 3 layers obtains the periodicity of the intermolecular interaction that provides a theoretical underpinning for the experimentally observed molecular arrangement.

9:40am

**SP+AS+BI+ET+MI+NM+NS+SS+TF-WeM6** Atomic Scale Imaging and Electronic Structure of Trimethylaluminum Deposition on III-V Semiconductor (110) Surfaces, T. J. Kent*, M. Edmonds, E. Chaggarov, A. C. Kummel, University of California San Diego

Silicon based metal oxide semiconductor field effect transistors (Si-MOSFETs) are quickly approaching their theoretical performance limits [1]. As a result many semiconductors are being explored as an alternative channel material for use in MOSFETs. III-V semiconductor are an appealing alternative to Si because of their higher electron mobilities. The limiting factor in III-V based MOSFET performance is defect states which prevent effective modulation of the Fermi level. The InGaAs (001) As-rich (2x4) surface contains two types of unit cells: ideal unit cells with double As-dimers and defect unit cells with single As-Dimers. The missing As-dimer unit cells, which comprise ~50% of the surface, are believed to cause electronic defect states at the semiconductor-oxide interface, specifically at the conduction band edge of the semiconductor. In-situ scanning tunneling microscopy and spectroscopy (STM/STS) and density functional theory (DFT) modeling show that TMA readily passivates the As-As dimers in the ideal unit cell but the missing InGaAs(001)-2x4 may not be fully passivated by TMA. To improve the electronic structure of the interface, the sidewalls of the finFETs on InGaAs(001) can be fabricated along the (110) direction. The (110) surface contains only buckled III-V heteroatoms in which the lower group III atom is sp2 hybridized with an empty dangling bond and the upper group V atom is sp3 hybridized with a full dangling bond. This results in an electrically unpassined surface.

To investigate the benefit of using a (110) surface as a channel material, the atomic and electronic structure of the ALD precursor trimethylaluminum (TMA) monolayer deposited on III-V (110) surfaces has been studied using in-situ STM and STS. Both GaAs and InGaAs samples were studied. GaAs wafers were obtained from Wafertech with a Si doping concentration of 4x1018/cm3. The (001) samples were cleaved in-situ to expose the (110) surface. Samples were transferred to the STM chamber (base pressure 1x10-11 torr) where the atomic bonding structure of the precursor monolayer unit cell was determined. STS, which probes the local density of states (LDOS), was used to determine Fermi level pinning. A model of TMA chemisorption was developed in which TMA chemisorbs between adjacent As atoms on the surface, giving a highly ordered monolayer with a high nucleation density which could allow for aggressive effective oxide thickness (EOT) scaling.

10:40am

**SP+AS+BI+ET+MI+NM+NS+SS+TF-WeM9** A New Experimental Method to Determine the Torsional Spring Constants of Microcantilevers, G. Hähner*, J. Parkin, D. Pascual, M. Edmonds, J. F. Portoles, Newcastle University, UK

Cantilever based technologies have seen an ever increasing level of interest since the atomic force microscope (AFM) was introduced more than two decades ago. Recent developments employ microcantilevers as stand-alone sensors by exploiting the dependence of their oscillating properties on external parameters such as adsorbed mass [1], or the density and the viscosity of the liquid environment [2](see references). They are also a key part in many microelectromechanical systems (MEMS) [3]. In order to quantify measurements performed with microcantilevers their stiffness or spring constants have to be known. Following calibration of the spring constants a change in oscillation behavior can be quantitatively related to physical parameters that are probed. The torsional modes of oscillation have attracted significant attention due to their high sensitivity towards lateral and normal forces, and recent developments in torsional-tapping AFM technology [5]. However, the methods available to determine the torsional spring constants experimentally are in general not simple, not very reliable, or risk damage to the cantilever [6].

We demonstrate a new method to determine the spring constants of the torsional modes of microcantilevers experimentally with high accuracy and precision. The method is fast, non-destructive and non-invasive. It is based on measuring the change in the resonance frequencies of the torsional modes in function of the height flow escaping from the free cantilever ends for rectangular cantilevers will be presented and compared to results obtained with other methods [7].

approach requires that a calibrated reference device is available, but it has been shown to be effective in providing measurement traceability[2].

The main remaining difficulty of this approach for typical users is the positional uncertainty of the tip on the reference device, which can introduce calibration uncertainties of up to around 6%. Here we present a new reference device based on a torsional spring of relatively large dimensions compared to the typical AFM cantilever and demonstrate how it is calibrated. This method has the potential to calibrate the reference device traceably[3] to the SI with a 1% accuracy by applying techniques typically used for the characterisation of micromechanical devices. The large dimensions of the device reduce the positional uncertainty below 1% and simultaneously allow the use of the device as an effective reference array with different reference stiffnesses at different positions ranging from 0.090 N/m to 4.5 N/m


11:20am SP+AS+BI+ET+MI+NM+NS+SS+TF-WeM11 Nanoscale Surface Assembly by Single-Molecule Cut-and-Paste, H.E. Gaub, Ludwig-Maximilians Universität, Germany

INVITED

Bottom up assembly of functional molecular ensembles with novel properties emerging from composition and arrangement of its constituents is a prime goal of nanotechnology. With the development of Single-Molecule Cut-and-Paste (SMC&P) we provided a platform technology for the assembly of biomolecules at surfaces. It combines the Å-positioning precision of the AFM with the selectivity of DNA hybridization to pick individual molecules from a depot chip and allows to arrange them on a construction site one by one. An overview on different applications of this technology will be given in this talk. One recent example demonstrates the functional of receptors for small molecules. By SMC&P we assembled binding sites for malachite green in a molecule-by-molecule assembly process from the two halves of a split aptamer. We show that only a perfectly joined binding site immobilizes the fluorophore and enhances the fluorescence quantum yield by several orders of magnitude. To corroborate the robustness of this approach we produced a micron-sized structure consisting of more than 500 reconstituted binding sites. To the best of our knowledge this is the first demonstration of a one by one bottom up functional bio-molecular assembly. Figure included in supplemental document. S. Kufer, Puchner E. M.,Gumpp H., Liedel T. & H. E. Gaub Science (2008), Vol 319, p 594-S. Kufer, Strackharn, M., Stahl S.W., Gumpp H., Puchner E. M. & H. E. Gaub Nature Nanotechnology (2009), Vol 4, p 45-M. Erdmann, R. David. A.N. Fornof, and H. E. Gaub, Nature Chemistry (2010), Vol 2, p 755-M. Strackharn, S. Stahl, E. Puchner & H.E. Gaub, Nanoletters (2012) in press
Scanning Probe Microscopy Focus Topic
Room: 16 - Session SP+AS+BI+ET+MI+TF-WeA

Emerging Instrument Formats
Moderator: A. Belu, Medtronic, Inc.

2:00pm
SP+AS+BI+ET+MI+TF-WeA1

Invited

Electrochemical reactions in solids underpin multiple applications ranging from electroresistive non-volatile memory and neuromorphic logic devices to chemical sensors and electrochemical gas pumps, to energy storage and conversion systems including metal-air batteries and fuel cells. Understanding the functionality in these systems requires probing reversible (oxygen reduction/evolution reaction) and irreversible (cathode degradation and activation, formation of conductive filaments) electrochemical processes. Traditionally, these effects are studied only on the macroscopically averaged level. In this talk, I summarize recent advances in probing and controlling these transformations locally on nanometer level using scanning probe microscopy. The localized tip concentrates an electric field in a nanometer scale volume of material, inducing local ion transport. Measured simultaneously, the electromechanical response (piezoresponse) or current (conductive AFM) provides the information on bias-induced changes in a material. Here, I illustrate how these methods can be extended to study local electrochemical transformations, including vacancy dynamics in oxides such as titanates, \( \text{LaSr}_1-x\text{Co}_3, \text{BiFeO}_3, \) and \( \text{Y}_2\text{Zr}_1-x\text{O}_2. \) The formation of electrochemical hysteresis loops indistinguishable from those in ferroelectric materials illustrate the role ionic dynamics can play in piezoresponsive force microscopy and similar measurements. In materials such as lanthanum-strontium carbonate, mapping both reversible vacancy motion and vacancy ordering and static deformation is possible, and can be corroborated by post mortem STEM/EELS studies. The possible strategies for elucidation ionic motion at the electroactive interfaces in oxides using high-resolution electron microscopy and combined ex-situ and in-situ STEM/DFM studies will be discussed. Finally, the future possibilities for probing electrochemical phenomena on in-situ grown surfaces with atomic resolution are discussed. This research was conducted at the Center for Nanophase Materials Sciences, which is sponsored at Oak Ridge National Laboratory by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

2:40pm
SP+AS+BI+ET+MI+TF-WeA3
Probing Electrochemical Phenomena in Reactive Environments at High Temperature: In Situ Characterization of Interfaces in Fuel Cells. S.S. Nonnenmann, B. Kungas, J.M. Vohs, D.A. Bonnell, University of Pennsylvania

Many strategies for advances in energy related processes involve high temperatures and reactive environments. Fuel cell operation, chemical catalysis, and certain approaches to energy harvesting are examples. Scanning probe microscopy provides a large toolbox of local and atomic resolution measurements of phenomena at a scale that enables understanding of complex processes involved in many systems. Inherent challenges exist, however, in applying these techniques to the realistic conditions under which these processes operate. To overcome some of these challenges, we have designed a system that allows SPM at temperatures to \( 850^\circ \text{C} \) in reactive gas environments. This is demonstrated with the characterization of an operating fuel cell. Solid oxide fuel cells (SOFCs) offer the highest conversion efficiencies with operating temperatures ranging from \( 400^\circ \text{C} \) to \( 1000^\circ \text{C} \); and operate under variable gaseous fuel environments – \( \text{H}_2\)-based environments (anode side) and \( \text{O}_2\)-based environments (cathode side). Topography and the temperature dependence of surface potential are compared to impedance. While not (yet) at atomic levels of spatial resolution, these probes are at the scale to examine local interface properties.

3:00pm
SP+AS+BI+ET+MI+TF-WeA4
High-Resolution Scanning Local Capacitance Measurements. M. Brukman, University of Pennsylvania, S. Nanayakkara, National Renewable Energy Laboratory, D.A. Bonnell, University of Pennsylvania

Spatial variation of dielectric properties often dictates the behavior of devices ranging from field effect transistors to memory devices to organic electronics, yet dielectric properties are rarely characterized locally. We present methods of analyzing 2nd harmonic-based local capacitance measurements achieved through non-contact atomic force microscopy. Unlike contact-based methods, this technique preserves tip shape and allows the same probe to realize high-resolution topographic imaging and scanning surface potential imaging. We present an improved analysis of the electrical fields between tip and sample, yielding high sensitivity to the capacitance-induced frequency shift.

The techniques are applied to thin-film ceramics (SrTiO2 and HfO2), metals (Pt and Ti), and mixed-phase self-assembled monolayers to illustrate application over all orders of dielectric constant. Conversion from frequency shift signal to dielectric constant \( \kappa \) is demonstrated, with sub-5 nm spatial resolution and dielectric constant resolution between 0.25 and 1.

4:00pm
SP+AS+BI+ET+MI+TF-WeA7
Experimental Calibration of the Higher Flexural Modes of Microcantilever Sensors. J.D. Parkin, G. Hähner, University of St Andrews, UK

Microcantilevers are widely employed as probes not only in atomic force microscopy [1], but also as sensors for mass [2], surface stress [3], chemical identification [3], or in measuring viscoelastic properties of cells [4]. Use of the higher flexural modes of microcantilever sensors is an area of current interest due to their higher Q-factors and greater sensitivity to some of the properties probed [2]. A pre-requisite for their exploitation, however, is knowledge of the spring constants [5]. None of the existing cantilever calibration techniques can calibrate the higher flexural modes easily.

We present a method that allows for the determination of the spring constants of all flexural modes. A flow of gas from a microchannel interacts with the microcantilever causing a measurable shift in the resonance frequencies of all flexural modes [6]. The method is non-invasive and does not risk damage to the microcantilever. From the magnitude of the frequency shifts the spring constants can be determined with high accuracy and precision. Experimental data for the response of the first four flexural modes of microcantilever beams used in AFM with spring constants in the range of \( ~0.03-90 \text{ N/m} \) will be presented.

The spring constants of the first mode determined using our method are compared to those obtained with the Sader method [7]. Finite element analysis computational fluid dynamics (CFD) simulations of the experimental setup are used to provide an insight into the interaction of the flow with the microcantilever.

References

4:20pm
SP+AS+BI+ET+MI+TF-WeA8

As its name implies, Atomic Force Microscopy (AFM) has long been used to acquire images at the atomic scale. However these images usually only show the lattice of atoms in the crystal and do not show individual atomic defects. In order to achieve atomic resolution, researchers have typically had to design their systems for the ultimate in noise performance, sacrificing ease of use, flexibility, and scan size. Recently we have demonstrated that, by using Peak Force Tapping, our large sample platforms (Dimension Icon, Dimension FastScan) are capable of obtaining atomic resolution imaging along with maps of the tip-sample interaction. Unlike standard TappingMode, or FM-AFM, Peak Force Tapping uses instantaneous force control, allowing the system to be insensitive to long range forces while maintaining piconewton level control of the force at the point in the tapping cycle that provides the highest resolution – the peak force. Since the modulation frequency is far from resonance, the technique is less sensitive to the cantilever thermal noise (Brownian motion). In addition to topography, this technique can provide maps of the interaction between the tip and the sample. This is possible since Peak Force Tapping has access to the instantaneous force between tip and sample at any point in the modulation cycle. To study the details of a tip-sample interaction, Atomic Peak Force Capture can acquire the entire force distance curve used to create the interaction maps. These curves can be exported for easy analysis with models of tip-sample interaction. In this talk we will discuss the latest atomic resolution results using Peak Force Tapping and the
implications of this with regard to studies of dissolution, crystallization, ordered liquids, and corrosion.

4:40pm SP+AS+BI+ET+MI+TF-WeA9 Nanoscale Chemical Composition Mapping with AFM-based Infrared Spectroscopy, C.B. Prater, M. Lo, Q. Hu, Anasys Instruments, C. Marcott, Light Light Solutions, B. Chase, University of Delaware, R. Shetty, K. Kjoller, E. Dillon, Anasys Instruments. INVITED

The ability to identify material under an AFM tip has been identified as one of the "Holy Grails" of probe microscopy. While AFM can measure mechanical, electrical, magnetic and thermal properties of materials, until recently it has lacked the robust ability to chemically characterize unknown materials. Infrared spectroscopy can characterize and identify materials via vibrational resonances of chemical bonds and is a very widely used analytical technique. We have successfully integrated AFM with IR spectroscopy (AFM-IR) to obtain high quality infrared absorption spectra at arbitrary points in an AFM image, thus providing nanoscale chemical characterization on the sub-100 nm length scale. Employing the AFM-IR technique, we have mapped nanoscale chemical, structural and mechanical variations in multilayer thin films, nanocomposites, polymer blends, organic photovoltaics, and biological materials including hair, skin, and bacterial and mammalian cells. Light from a pulsed infrared laser is directed at a sample, causing rapid thermal expansion of the sample surface at absorbing wavelengths. The rapid thermal expansion creates an impulse force at the tip, resulting in resonant oscillations of the AFM cantilever. The amplitude of the cantilever oscillation is directly related to the infrared absorption properties of the samples, enabling measurements of IR absorption spectra far below the conventional diffraction limit. AFM-IR can be used both to obtain point spectra at arbitrary points and to spatially map IR absorption at selected wavelengths. Simultaneous measurement of the cantilever's contact resonance frequency as excited by the IR absorption provides a complimentary measurement of relative mechanical properties. We have used these techniques to chemically identify individual chemical components in polymer nanocomposites and multilayer films and performed subcellular spectroscopy and chemical imaging on biological cells. Using self-heating probes we have been able to locally modify the state of a semicrystalline polymer and observe the resulting change in absorption spectra on the nanoscale. Using polarization sensitive AFM-IR, we have mapped spatial variations in molecular orientation in electrospun fibers.

5:40pm SP+AS+BI+ET+MI+TF-WeA11 Quantifying Nanomechanical Properties with Simultaneous AM-FM and tanδ Imaging, T. Mehr, A. Moshar, R. Proksch, I. Revenko, N. Geisse, S. Hohlbauch, D. Walters, J. Cleveland, J. Bemis, C. Callahan, D. Beck, Asylum Research

Frequency-Modulated (FM) is a powerful, quantitative technique for mapping interaction forces between an oscillating tip and sample. Since FM-AFM typically requires the use of three feedback loops, one ongoing challenge has been stable and cross-talk free operation. Amplitude-modulated Atomic Force Microscopy (AM-AFM), also known as tapping mode, is a proven, reliable and gentle imaging method with widespread applications. Recently, the phase signal of the first resonant mode has been recast in terms of the tip-sample loss tangent.[1] This allows quantitative imaging of a response term that includes both the dissipated and stored energy of the tip sample interaction. Combining AM and FM imaging allows reaping the benefits of both techniques.[2] Because the feedback loops are decoupled, operation is more robust and simple than conventional FM imaging. In this mode, the topographic feedback is based on the AM signal of the first cantilever resonance while the second resonance drive is frequency modulated. The FM image returns a quantitative value of the frequency shift that in turn depends on the sample stiffness and can be applied to a variety of physical models. We will present results on a wide variety of materials as well as discussing quantitative separation of the elastic and dissipative components of the tip-sample interactions.[3]

References


5:20pm SP+AS+BI+ET+MI+TF-WeA12 Simultaneous Scanning Tunneling and Atomic Force Microscopy with Subatomic Spatial Resolution, F.J. Giessibl, University of Regensburg, Germany

Frequency-modulation AFM can be combined with scanning tunneling microscopy, yielding a simultaneous data set for current and average force gradient. Ternes et al. [1] have shown that for some metallic contacts, force and current are proportional. The interaction of a tungsten tip with a CO molecule adsorbed on Cu(111), however, yields a much different symmetry and distance dependence of tunneling current and force [2]. The tunneling current yields a gaussian dip over the CO molecule, while the forces show a strong angular dependence with force fields that vary strongly by distance and angle within the extent of the single front atom, displaying subatomic variations. While the simultaneous acquisition of current and force can reveal new information about the atomic and electronic structure of matter, the tunneling current can modify the atomic forces. This “phantom force” [3,4], a modification of the electrostatic attraction between tip and sample, originates in an alteration of the effective potential difference between tip and sample caused by strongly localized voltage drop induced by the tunneling current. The talk discusses the potential of combined STM/AFM as well as the challenges, in particular with respect to tip preparation and characterization.


Thursday Morning, November 1, 2012

Magnetic Interfaces and Nanostructures
Room: 6 - Session MI+SP+AS-ThM

Emerging Probes in Magnetic Imaging, Reflectometry and Characterization
Moderator: Z. Gai, Oak Ridge National Laboratory, V. Lauter, Oak Ridge National Laboratory

8:00am MI+SP+AS-ThM1 Toward Microscopy with Direct Chemical and Magnetic Contrast at the Atomic Level. V. Rose, Argonne National Laboratory

In this talk we will discuss the development of a novel high-resolution microscopy technique for imaging of nanoscale materials with chemical, electronic, and magnetic contrast. It will combine the sub-nanometer spatial resolution of scanning tunneling microscopy (STM) with the chemical, electronic, and magnetic sensitivity of synchrotron radiation. [1] Drawing upon experience from a prototype that has been developed to demonstrate general feasibility, current work has the goal to drastically increase the spatial resolution of existing state-of-the-art x-ray microscopy from only tens of nanometers down to atomic resolution. The technique will enable fundamentally new methods of characterization, which will be applied to the study of energy materials and nanoscale magnetic systems. A better understanding of these phenomena at the nanoscale has great potential to improve the conversion efficiency of quantum energy devices and lead to advances in future data storage applications. The combination of the high spatial resolution of STM with the energy selectivity afforded by x-ray absorption spectroscopy provides a powerful analytical tool.

Work at the Advanced Photon Source, the Center for Nanoscale Materials, and the Electron Microscopy Center was supported by the U. S. Department of Energy, Office of Science, Office of Basic Energy Sciences, under contract DE-AC02-06CH11357.


8:40am MI+SP+AS-ThM3 Effect of Sub-Micrometer Scale Magnetic Inhomogeneity on the Magnetoelectric Coupling in Manganites. A. Biswas, University of Florida

The conventional magnetoelectric (ME) coupling in multiferroics is defined as the effect of a magnetic/electric field on the electric-polarization/magnetization. However, the strength of the ME coupling is usually small. Our recent results have revealed methods for significantly increasing the ME coupling in perovskite manganites and can be summarized in two broad categories: (1) in the phase separated manganite (La1-xPrx)1-xCaxMnO3 (LPCMO), we have discovered that anisotropic strain which increases towards the FePt/FePt3 interface is found. Below the Néel temperature, antiferromagnetic FePt3 layer and nuclear resonant scattering of synchrotron x-rays (making use of the Mössbauer effect) to probe the antiferromagnetic FePt3 layer. Below the Néel temperature, antiferromagnetic order appears in the FePt layer with a spin wavevector pointing along the [100] axis. A net magnetization of the FePt, which increases towards the FePt/FePt3 interface is found.

This work was supported by the Fund for Scientific Research-Flanders (FWO), the KU Leuven Concerted Research Action program (GOA/09/006), the Belgian Interuniversity Attraction Poles research programs (IAP P6/42), and the KU Leuven BOF (CREA/07/005) program.

9:20am MI+SP+AS-ThM5 Impact of Interfacial Magnetism on Magnetocaloric Properties of Thin Film Heterostructures. C. W. Miller, University of South Florida

In an effort to understand the impact of nanostructuring on the magnetocaloric effect, we have grown and studied gadolinium in W(5nm)/Gd(30nm)/W(5nm)n heterostructures. The entropy change associated with the second-order magnetic phase transition was determined from the isothermal magnetization for numerous temperatures and the appropriate Maxwell relation. The entropy change peaks at a temperature of 284 K with a value of approximately 3.4 J/K g for 30 Koe field change; the full width at half max of the entropy change peak is about 70 K, significantly wider than that of bulk Gd under similar conditions. The relative cooling power of this nanoscale system is about 240 J/kg, somewhat lower than that of bulk Gd (410 J/kg). Polarized neutron reflectometry was used to determine the depth profile of the magnetic moment per Gd atom, mGd. Despite sharp interfaces observed by transmission electron microscopy, mGd is systematically suppressed near the Gd-W interfaces. Because the peak magnetic entropy change is proportional to mGd, the maximum achievable magnetocaloric effect in Gd-W heterostructures is reduced. By extension, our results suggest that creating materials with Gd-ferromagnet interfaces may increase the mGd relative to the bulk, leading to enhanced magnetocaloric properties. Together, these observations suggest that nanostructuring may be a promising route to tailoring the magnetocaloric response of materials.

Supported by AFOSR and NSF.

10:40am MI+SP+AS-ThM9 Polarized Neutron Reflectometry on Exchange Biased Thin Films. K. Temst, KU Leuven, Belgium

Polarized neutron reflectivity has established itself as an important tool in the study of magnetic thin film systems. It provides a high-resolution magnetic depth profile and it offers vectorial probing of the magnetization. In recent years polarized neutron reflectivity has played an influential role in elucidating the magnetic structure of exchange bias systems, i.e. structures in which a ferromagnetic layer is coupled to an antiferromagnetic layer. Exchange bias leads to a net magnetization shift of the hysteresis loop, an increase in coercivity, and often a pronounced asymmetry of the hysteresis loop shape as well as a complex magnetic history. With this contribution we will take a closer look at two such exchange bias systems and highlight the role of polarized neutron reflectivity.

As a first model system, the archetypal exchange bias system Co/CoO will be highlighted. The antiferromagnetic CoO layer is prepared by oxidizing the surface of a Co thin film, by exposing it to a reduced oxygen atmosphere. We will review the properties of exchange bias in surface-oxidized Co thin films, with the emphasis on the asymmetry of the magnetization reversal mechanism and the training effect. We will also discuss how the training effect can be (partially) restored by applying a magnetic field perpendicular to the initial cooling field direction. Recently we explored an alternative way to establish exchange bias between Co and CoO. Instead of creating an antiferromagnetic CoO layer by oxidizing a metallic Co layer, the antiferromagnetic CoO is produced by implantation of oxygen ions into a Co layer. Polarized neutron reflectivity (PNR) is used to determine the magnetic depth profile and to probe the magnetization reversal mechanism. Simultaneously with the PNR measurements, in situ anisotropic magnetoresistance measurements were carried out.

The second example is a ferromagnet/antiferromagnet FePt/FePt3 bilayer in which complementary use is made of polarized neutron reflectivity (for studying the magnetic depth profile in the ferromagnetic layer) and nuclear resonant scattering of synchrotron x-rays (making use of the Mössbauer effect) to probe the antiferromagnetic FePt3 layer. Below the Néel temperature, antiferromagnetic order appears in the FePt layer with a spin wavevector pointing along the [100] axis. A net magnetization of the FePt, which increases towards the FePt/FePt3 interface is found.
We will present studies of magnetic vortex structures, where we found a stochastic character in the nucleation process, which can be described within a symmetry breaking DM interaction [3]. We will also present time resolved studies of dipolar coupled magnetic vortices, where we find an efficient energy transfer mechanism, which can be used for novel magnetic logic elements [4].

This work was supported by the Director, Office of Science, Office of Basic Energy Sciences, Materials Sciences and Engineering Division, of the U.S. Department of Energy under Contract No. DE-AC02-05-CH11231.


We have developed an imaging technique based on x-ray excited luminescence microscopy (XELM), that will enable elemental and magnetic specific imaging of a wide range of materials such as those used in solar cells, magnetic materials, spintronic devices, ferroelectrics, and solid-state lighting. This new scientific tool utilizes the benefits of pulsed, polarized, tunable synchrotron radiation excitation with microscopic detection of the resulting optical emission. A unique offshoot of the microscope is the ability to perform element specific magnetic microscopy of micron-sized features or domains in magnetic fields. X-rays transmitted through thin films are attenuated and the resultant absorption spectrum can be determined by changes in the substrate luminescence. Since many substrates, such as SrTiO3 and GaAs, used in thin film growth have intense optical emission, this tool should impact many materials where photoelectron emission microscopy (PEEM) cannot be performed since it is not useable on insulating materials or if magnetic or electric fields are required. This approach will be especially useful at low temperatures where luminescence yields are highest, and PEEM has difficulties. In this presentation we will present some initial results from the microscope on some prototype solar cell materials and lithographically patterned Permalloy/GaAs and Permalloy/Cu/Co/GaAs samples. The results demonstrate the potential of XELM for elemental and magnetic specific imaging.

This work was performed at the Center for Nanoscale Materials and the Advanced Photon Source. It was supported by the U.S. Department of Energy, Office of Science and Office of Basic Energy Sciences under the contract number DE-AC02-06CH11357 and by the Department of Energy, Energy Efficiency and Renewable Energy, Solid State Lighting Program.
Electron Transport at the Nanoscale: Molecules and Defects

Moderator: S. Allen, The University of Nottingham, UK, J. Wendelken, Oak Ridge National Laboratory

2:00pm ET+SS+GR+SP-ThA1 Molecule Substrate Interactions Probed by Scanning Tunneling Microscopy, H.-J. Gao, Chinese Academy of Sciences INVITED

Molecule substrate interactions play an important role in constructing nanostructures of functionalities and controlling of the physical properties and thus have stimulated a great interest in the past decades. One of the challenges in this top is to make a single molecule or small quantum systems stably connected to a single metal atom on metal surfaces, and further to make the nanostructure create electric energy and optical/electro radiation. In the talk, I will present that by using Au adatoms of the Au(111) surface we have successfully formed a single molecule rotor array, isolated tetraphenyl zinc(ZnTPh)5 molecules sticking to the Au adatoms of the Au(111) and rotating on the surface at 78 K. This kind of single molecular rotor was also found to be controlled by the different sites of the Au(111) surface using low temperature scanning tunneling microscopy (STM). Furthermore, by changing the molecular structure the same molecular family of the Fe, ZnFe and FePc can also modulate the rotation of the molecular rotors. Guest and the lateral structure of a molecule/substrate interface can be modified by the attachment of ligands, our results will be helpful for opening up the possibility to tailor physical properties of a single molecule or complex aggregates to the desired specifications.


Scanning tunneling microscopy (STM) is used to study two dinuclear organometallic molecules, meta-Fe2 and para-Fe2. These molecules share identical molecular formulas but differ in their positions of connectivity to the central phenyl ring. STM images of neutral meta-Fe2 and para-Fe2 show symmetrical distribution of electron density across the two metal centers. Chemical oxidation of these molecules leads to mixed-valence species. STM images of mixed-valence meta-Fe2 show an asymmetric distribution of the electron density between the two metal centers. Despite the greater distance between the two metal centers, the electron density remains symmetric in mixed-valence para-Fe2. Comparison with constrained density functional (CFT) calculations lead to the conclusion that through-bond coupling of the two metal centers is more prevalent than through-space coupling. Further investigation of mixed-valence dinuclear organometallic molecules with asymmetric electron state density may open up the possibility for their use in molecularly based electronic devices.

3:00pm ET+SS+GR+SP-ThA4 Rectification by a Single Molecular Diode, L. Adamska, University of South Florida, M. Kozhushner, Institute of Chemical Physics, Russian Academy of Sciences, I.I. Oleyniik, University of South Florida

Single-molecule rectification has been recently observed using the STM modulated break-junction technique in non-symmetric diblock dipyrimitidinyl-diphenyl molecule covalently bound to metallic electrodes. To provide an insight to the nature of the rectification, first-principles calculations of atomic, electronic and transport properties of gold/molecule/gold junctions have been performed for both symmetrical tetraphenyl and asymmetrical dipyrimitidinyl-diphenyl diblock molecules. The charge transport was found to occur by hole resonant tunneling mechanism via positively-charged hole states of the molecule, which are very susceptible to the external electric field and dispersive interactions with the metallic electrodes. It was found that the rectification effect in chemically-assyemtrical dipyrimitidinyl-diphenyl molecule is due to strong localization of the hole wave function at one end of the diblock under applied electric field. Such behavior is contrasted by symmetric I-V curves exhibited by symmetric tetraphenyl molecule.


Graphene, due to its unique electronic structures, has quickly become one of the most notable “super-materials” poised to transform the electronics and nanotechnology landscape. The symmetry of the graphene honeycomb lattice is a key element for determining many of graphene’s unique electronic properties, such as the linear energy-momentum dispersion and the reduced backscattering (i.e., high carrier mobility). However, topological lattice defects, such as grain boundaries and step edges, break the sublattice symmetry and can affect the electronic properties, especially in transport of graphene in unexpected ways. To utilize the full potential of graphene a complete understanding of the physical and electronic properties of defects in this system is needed. By using a scanning tunneling potentiometry method with a low temperature four-probe scanning tunneling microscope, two-dimensional maps of electrochemical potentials have been measured across individual grain boundaries on the graphene films grown on copper foil and transferred to SiO2. An Atomic Force Microscope (AFM) is implemented to image the grain boundary that forms between individual graphene flakes that grow on the surface. The AFM imaging along with scanning tunneling potentiometry characterize the grain boundaries formed between coalesced grains on the SiO2 surface. Results of the study of the grain boundaries of the electronic transport across this potentially revolutionary new electronic system will be presented. This research was conducted at the Center for Nanophase Materials Sciences, which is sponsored at Oak Ridge National Laboratory by the Office of Basic Energy Sciences, U.S. Department of Energy.

4:00pm ET+SS+GR+SP-ThA7 Static and Dynamic Conductance in Topological Defects in Ferroelectrics, S.V. Kalinin, Oak Ridge National Laboratory INVITED

Topological defects in ferroic materials are attracting much attention both as a playground of unique transport, magnetic, and ferroic phenomena and due potential applications in reconfigurable electronic devices. In this presentation, I will summarize recent studies of transport phenomena in 2D (domain walls) and 1D (vortices and antivortices) in ferroelectric materials by combination of piezoresistive field microscopy, phase-field modelling, and density functional theory. In particular, the observations, such as memory effects and hysteresis in domain wall conductance, metallic conductivity of ferroelectric walls, and conductivity in vortex cores will be discussed. For domain walls, these observations are consistent with carrier accumulation due to presence of charged wall segments. We further demonstrate that a continuum of non-volatile metallic states across decades of conductance can be encoded in the size of ferroelectric nanodomains using electric field. For vortices, modelling predicts that the core structure can undergo a reversible transformation into a metastable twist structure, extending charged domain walls segments through the film thickness. The vortex core is therefore a dynamic conductor controlled by the coupled response of polarization and electron/mobile vacancy subsystems with external bias. This controlled creation of conductive 1D channels opens a pathway for design and fabrication of topological electronic devices and systems based on domain patterning. Finally, recent insight on domain walls structure from combination of aberration corrected scanning transmission electron microscopy and phase-field theory will be discussed. Research supported (SVK) by the U.S. Department of Energy, Basic Energy Sciences, Materials Sciences and Engineering Division and partially performed at the Center for Nanophase Materials Sciences, a DOE-BES user facility.

4:40pm ET+SS+GR+SP-ThA9 Transport and Mechanical Properties of Molecular Junctions formed by Acetophenon Deposited on Si (100) Surface, M. Setvin, Z. Majzik, O. Krcjci, P. Hapala, P. Jelinek, Institute of Physics of ASCR, Czech Republic

One of the main challenges of Molecular electronics is to understand and control charge transfer through a reproducible single molecule contact between electrodes. Most investigations of electron transport through molecules have been performed in “blind” junction experiments, where the molecular conformation and contact geometry cannot be probed. Therefore large gaps in our knowledge remains since in molecular electronics the atomic-scale structure of the entire junction including the leads is important for its conductance properties. Our goal is to study electrical transport through well-defined molecular junction on semiconductor surfaces. Formation of molecular junctions using organic molecules on semiconductor surfaces might lead to interesting phenomena. For example, the presence of the band gap in electrodes can
lead to the negative differential resistance observed in transport through molecules bonded to dangling-bond sites[1].

In this contribution, we investigate formation of molecular junction consisting of a single acetophenone molecule deposited on Si(100) surface in upright position by means of simultaneous AFM/STM measurements and DFT calculations. We used a modified UHV VT STM/AFM Omicron machine allowing simultaneous acquisition of the current and forces with atomic resolution using a tuning fork sensor[2]. The simultaneous acquisition of the tunneling current and force during tip approach allows precise control of contact formation and its consequences on the charge transport through molecular junction[3]. On other hand, DFT simulations provide more insight into interaction mechanism between probe and molecule. It also helps to understand induced structural and electronic relaxations during tip approach.

References

5:00pm ET+SS+GR+SP-ThA10 Experimental Determination of the Charge Neutrality Level (CNL) of Conjugated Polymers, W. Wang, R. Schlaf, University of South Florida
The charge neutrality levels (CNL) of poly-(3-hexylthiophene) (P3HT) and poly-[2-methoxy-5-(2'-ethylhexyloxy)-p-phenylene vinylene] (MEH-PPV) were determined. This was achieved by investigating a number of interfaces between these conjugated polymers and metals with varying work function. The interfaces were fabricated using the electrospray deposition technique, which allows the direct deposition of polymers from solution into vacuum environment without interference of significant surface contamination. This enabled the measurement of the charge injection barriers at clean polymer interfaces without contamination interlayer with photoemission spectroscopy. The results of these measurements enabled the establishment of the correlation between barrier heights and substrate. The results indicate that conjugated polymer interfaces form in a similar fashion as small molecular materials interfaces to metals as described by the ‘induced density of states’ (IDIS) model. In contrast, the present results also suggest that the ‘integer charge transfer ’ (ICT) model, which was developed for polymer/metal interfaces, only describes interfaces prepared under atmosphere or inert environment, where the presence of contamination weakens the interaction between the materials in contact.

5:20pm ET+SS+GR+SP-ThA11 Investigation on the Electronic Structure of Arylthio (ArS)-functionized CdSe Nanoparticle Hybrid Materials, Z. Li, University of South Florida, K. Mazzio, University of Washington, S. Gatmann, University of South Florida, C.K. Luscombe, University of Washington, R. Schlaf, University of South Florida
CdSe nanoparticles are a promising material for 3D heterojunction solar cells due to their tunable electronic and optical properties through size control. By covalently bonding these nanoparticles to organic ligands, a novel light absorption, charge separation and transportation system can be formed. This presentation will discuss efforts to measure the hybrid-internal orbital line-up at the nanoparticle/ligand junction. Photoemission spectroscopy was used to characterize a prototypical arylthio (ArS)-functionized CdSe molecule nanoparticle/ligand hybrid. Generally, in such hybrid molecules, the highest occupied molecular orbital (HOMO) emission is a superposition of the emissions of the nanoparticles and that of the organic ligands. This superposition makes the direct study of the orbital line-up through PES measurements impossible. Hence, a new technique needed to be developed to delineate the orbital line-up at such inner-molecular contacts. This was achieved through the additional investigation of the isolated components, which yielded detailed information of their electronic structure. Combination of this data with measurements on the assembled hybrid molecules enabled the determination of the orbital line-up at the interface. The influence of the covalent bond on the orbital line-up was investigated by comparing the hybrid line-up with the electronic structure of a physisorbed interface formed between the hybrid components. This interface was prepared through in-vacuum electrospray deposition of a thin film sandwich of CdSe and ArSH films, while measuring the transition from the CdSe to the ArSH electronic structure.

8:20am ET+SS+GR+SP-FrM1 What is Missing in the Space Charge Limited Current Theory?, X.-G. Zhang, Oak Ridge National Laboratory, S.T. Panedides, Vanderbilt University

INVITED
Space-charge-limited currents are important in energy devices such as solar cells and light-emitting diodes, but the available theory from the 1950s finds it necessary to postulate defect states that are distributed in energy in order to match data. This has prevented the theory to be used in extracting reliable defect information such as energy level and trap density from measurements. Here we revisit the theory and show that this postulate is not warranted. Instead, we demonstrate that dopants and the concomitant Frenkel effect, which have been neglected, control the shape of measured current-voltage characteristics. For highly disordered material, there is a significant inter-trap tunnelling current in the Ohmic regime, which accounts for the observed peak in the noise power. The new theory can anchor efforts to develop experimental techniques to measure deep-trap levels.

This research was conducted at the Center for Nanophase Materials Sciences, sponsored by ORNL by the Division of Scientific User Facilities (XGZ) and by Division of Material Science and Engineering, Basic Energy Sciences, U.S. Department of Energy (STP), and the McMinn Endowment at Vanderbilt University (STP).

8:30am ET+SS+GR+SP-FrM3 Mapping Solar Cell Internal Fields and Band Offsets, H. Cohen, Y. Izhakhi, G. Hodes, Weizmann Institute of Science, Israel

The internal fields and band offsets across device interfaces are key features in various applications and, yet, this information is generally inaccessible by standard electrical tools. A systematic approach addressing this problem is demonstrated here, based on chemically resolved electrical measurements (CREM). Studying nanoporous photovoltaic cells, we resolve the internal details layer-by-layer and, thus, extract a realistic band diagram for the multi-interfacial structure. We show the spontaneous evolution of two p-n-like junctions and quantify the associated band bending at corresponding domains. An account for the ‘real’ working conditions of the device is attempted by exposing the cell to optical and electrical stimuli, revealing the charge trapping at each specific layer and showing how certain sample treatments affect the trapping mechanisms. Our methodology overcomes a critical missing link in device characterization and in fundamental studies of nanoscale solid-state devices.

9:00am ET+SS+GR+SP-FrM4 Quantum Degeneracy Revealed by the Relation between the Tunnelling Current and the Chemical Force, J. Pielmeick, M. Ondraczek, Institute of Physics of ASCR, Czech Republic, F. Flores, Universidad Autonoma de Madrid, Spain

Recent progress has allowed merging AFM and STM into a new experimental setup where tunnelling current and atomic forces are recorded simultaneously. The possibility to collect both quantities simultaneously opens new horizons not only in advanced characterization at the atomic scale but also in understanding fundamental relations between the electron transfer and formation of the chemical bond between two bodies.

Actually, there is a long-standing debate in the scientific community about the relation between the chemical force and the tunneling current (see e.g. [1]) on the atomic scale. Both the tunneling current and the short-range component of the force, induced by the formation of the chemical bond, exhibit in atomic contacts an exponential decay with increasing distance in the range of several angstroms. As the quantities depend directly on the wave-function overlap between outermost atoms of tip and surface, the corresponding exponential functions should have similar characteristic decay length. In particular, the relation between the chemical force F and the tunneling current I follows the law F=I, where n is an integer number. Over the last 10 years, several different scaling factors n, varying from 1 to 4, have been proposed by different groups based on both theoretical analysis and experimental measurements (see reference in [2]), still there is no consensus on the relation between the chemical force and the tunneling current.

In this contribution, we explain the relation between the tunneling current and the interaction force at the atomic scale using a simple analytical model

[2]. The model unveils the existence of two characteristic scaling regimes, where the tunneling current is either proportional to the chemical force I-F or to the square of the chemical force, i.e. I-F². We show that the existence of a given regime is basically controlled by two parameters: (i) the electron spin degeneracy and (ii) the hopping between electronic levels involved in the interaction process. Finally, we will collate our theoretical prediction with experimental AFM/STM measurements of single-atom point contacts and complex DFT simulations [3] to confirm the existence of these two characteristic regimes.


9:40am ET+SS+GR+SP-FrM5 Understanding the Influence of the Tunnelling Current and the Chemical Force on the Contrast Formation in KPFM, Z. Majzik, M. Onrubiec, M. Siec, J. Berger, P. Jelinek, Institute of Physics of ASCR, Czech Republic

The Kelvin Probe Force Microscopy (KPFM) [1] senses the variation in the electrostatic force. The electrostatic force is Felec=−dC/dz(βbias−Vcpd), where Vcpd denotes the local contact potential difference (LCPD). Atomic scale resolution was achieved by KPFM on the prototypical Si(111)−7×7 surface [2]. It was shown that the formation of a chemical bond between the closest tip-surface atoms induces significant variation in the LCPD [2]. Lately it was observed that the tunneling current leads to the raise of additional electrostatic component of the force [3]. Consequently, the total electrostatic force must be the combination of several components where the contribution of each component is defined by the tip-sample separation.

Recent progress in Scanning Probe Microscopy opens the possibility of simultaneous acquisition of the tunneling current, atomic forces and local potential difference with atomic resolution [4]. The aim of this contribution is to discuss the origin of the electrostatic force contribution at different tip-sample separations. In particular, we performed simultaneous site-specific AFM/STM measurements on Si(111)−7×7 using a modified Omicron qPlus (tuning fork based) system [5]. We found that along the tip approach three characteristic regimes can be well distinguished. At large tip-sample separations, the capacitance is a function of tip geometry and the tip-sample distance. Approaching the tip closer towards the surface, quantum effects become to play important role. The overlap between the tip and sample wave functions produces electron tunneling, which induces additional electrostatic force. Formation of the chemical interaction between the tip apex atom and the adatoms of the 7×7 surface induces changes in the electron charge distribution reflected in variation of the LCPD [2] and the permissivity in the tunneling gap. Hence the capacitance is modified accordingly. In order to have better understanding of the impact of the chemical interaction, atomic hydrogen was deposited to saturate the dangling bonds of adatoms. Over the hydrogenated adatoms, nor strong shift in the LCPD or sudden change in the capacitance was observed. Further to gain more insight into ongoing processes we carried out DFT calculations for tip-sample interaction to understand affect of the formation of covalent bond between tip apex and surface adatoms on the Si 7×7 surface.

References
isolated contact traces are lithographically patterned \textit{ex situ} onto the silicon substrate. Then a hard mask is \textit{grown} and lithographically patterned and connected to the BEEM sample plate which is then inserted into the ultra-high vacuum chamber. The metal is then deposited on top of the hard mask and then mounted \textit{in situ} onto the STM for BEEM measurements. BEEM measurements comparing both \textit{in situ} and \textit{ex situ} deposited films will be presented.

10:20am \ ET+SS+GR+SP-FrM7 \ Electronic Transport on the Nanoscale, R. Moeller, University of Duisburg-Essen, Germany \textbf{INVITED}

To study the transport through objects at the nanoscale a scanning tunneling microscope with several tips is used. Two different configurations will be discussed. The lateral transport of electrons may be studied by using two tips to drive a current parallel to the surface. A third tip enables to map the corresponding electrochemical potential. Measurements for a 2D conducting layer will be discussed. To analyze the transport perpendicular to the surface, a thin metallic layer is placed on a semiconducting surface. At the interface a Schottky barrier is formed, which can only be overcome by electrons of sufficient energy. This may be used to split the current of electrons coming from the tip of the microscope into two parts, the ballistic electrons and the electrons which have been scattered. This technique has been applied to study the ballistic transport of electrons through individual molecules. On the other hand inelastic processes may be revealed by analyzing the fluctuations in the tunneling current observed at different positions of the tunneling tip above an adsorbed molecule.
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