

3rd International Workshop

Nanoscale Imaging for Energy Applications

September 11-13, 2012 The Center for Nanophase Materials Sciences Oak Ridge National Laboratory, TN

Organizers Olga S. Ovchinnikova, An-Ping Li, Sergei V. Kalinin, and Arthur P. Baddorf



Nanoscale Imaging for Energy Applications GENERAL INFORMATION

Internet Access. There will complimentary wi-fi access available on the ORNL visitor network. **Meals at ORNL.** For attendees of the workshop breakfast, lunch is provided as part of the registration fee.

Transportation. For attendees staying in the Comfort Inn hotel in downtown Oak Ridge, there will be a shuttle provided to transport participants from the hotel to ORNL and back each day. **Poster Presenters.** Please mount your poster display on Wednesday before 1:00 pm. Boards are numbered. See poster list on pages 40 - 41 to learn your assigned board space. Instructions for preparing posters:

- Tabletop poster boards and adhesive Velcro tabs will be provided for mounting posters.
- You may bring your poster to the meeting in any convenient format such as a rolled-up sheet, pre-mounted on a rigid board, or as a set of smaller sheets.
- Posters must fit within an area that is 42 inches wide and 42 inches high.
- Posters should be printed mechanically using ink-jet, laser, or similar type printer and have a "professional" appearance, meeting the customary standards for display at international scientific conferences

Speakers. Please arrive in the session room, SNS 156, 15 minutes prior to the start of the session to set up your laptop computer.

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WORKSHOP SPONSORS

Center for Nanophase Materials Sciences, ORNL FIRST Center, ORNL American Vacuum Society (AVS) Chemical Sciences Division, ORNL



Oak Ridge National Laboratory, Center for Nanophase Materials Sciences, September 11-13, 2012



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Olga S. Ovchinnikova, An-Ping Li, Sergei V. Kalinin, and Arthur P. Baddorf Scope

Energy generation, storage, and conversion systems are an integral component of emerging green technologies, including solar power, automotive, and storage components of solar and wind energy economics. Despite the rapidly expanding manufacturing capabilities and wealth of phenomenological information on the macroscopic behaviors of energy storage and conversion systems, the microscopic mechanisms underpinning solar cell, battery and fuel cell operations in the nanometer to micron range are not well understood. This series of keynote and invited talks will cover the recent advances in characterization of energy relevant materials systems using Scanning Probe Microscopy (SPM) techniques, as well as the state of the art in energy dissipation and transformation measurements by SPM. Topics include mapping of carrier dynamics and photo-induced behavior of photovoltaic materials, ionic and electronic transport in fuel cells and Li-ion batteries, energy harvesting, and energy dissipation imaging by multiple resonant and band excitation SPMs, as well material characterization using mass spectrometry combined with SPM and optical spectroscopies for multimodal imaging. A number of contributed talks will be included. Ultimately, our goal is to build a network of materials scientists centered on the applications of SPM for energy problems and to promote rapid dissemination of theoretical knowledge, experimental protocols, and novel technique development in this rapidly growing area.

The 3rd International Workshop on Nanoscale Imaging for Energy Applications will be hosted in conjunction with the Annual Symposium of the Tennessee Valley Chapter (TVC) of AVS. The TVC-AVS focus session is an interdisciplinary meeting to discuss advances in SPM and applications of nanostructure analysis. Contributions from all areas of nanoscience and vacuum science are encouraged. **Invited Speakers**

- Richard Caprioli, Vanderbilt University Multi-modal imaging: Combining MALDI-MS imaging with MRI and microscopy
- Phillip First, Georgia Institute of Technology Measuring the effects of scalar and vector potentials in graphene
- M. Hersam, Northwestern University Scanning probe microscopy of energy materials
- Lincoln Lauhon, Northwestern University Correlated functional imaging of energy interconversion in hybrid Nanostructures
- M. Pan, Oak Ridge National Laboratory Oxide surfaces on atomic scale: STM quest
- V. Sethuraman, Brown University Strain dynamics in energy materials
- Z.L. Wang, Georgia Institute of Technology *Nanopiezotronics and nanowire based energy harvesting*
- Nick Winograd, Pennsylvania State University Nanoscale chemical imaging of biomaterials with mass spectrometry
- Vassilia Zorba, Lawrence Berkeley National Laboratory Optical near and far-field femtosecond laser ablation for nanoscale chemical imaging

Call for Abstracts—Talks and Posters

Both oral and poster presentations are welcome. A limited number of graduate student fellowships will be awarded to reimburse the registration fee and lodging at the conference hotel. **Abstracts must be received by August 13**. Instructions and on-line submission will be provided at the CNMS web site, <u>www.cnms.ornl.gov</u>. Contact An-Ping Li (<u>apli@ornl.gov</u>) for additional information on abstract submission, Olga Ovchinnikova (<u>ovchinnikovo@ornl.gov</u>) for student fellowship.





Oak Ridge National Laboratory, Center for Nanophase Materials Sciences, September 11-13, 2012

Tuesday, September 11, 2012	
Room SNS 156 1:00-1:15 Room SNS 156 1:15-1:35	Welcome and Introduction Sean Smith, Oak Ridge National Laboratory Multimodal Chemical Imaging Gary Van Berkel, Oak Ridge National Laboratory "Molecular Cartography: Combining mass spectrometry with atomic force microscopy"
1:35-2:05	Richard Caprioli, Vanderbilt University "Multimodal imaging: Combining MALDI-MS imaging with MRI and microscopy"
2:05-2:35	Nicholas Winograd, Pennsylvania State University "Nanoscale chemical imaging of biomaterials with mass spectrometry."
2:35-2:55	Kevin Kjoller, Anasys Instruments "Nanoscale Chemical, Mechanical and Thermal Analysis using Atomic Force Microscopy."
2:55-3:25	Vassilia Zorba, Lawrence Berkeley National Laboratory "Optical near and far-field femtosecond laser ablation for nanoscale chemical imaging."
3:25-3:35	Break
3:35-4:05	Maryia Nudnova, ETH-Zurich "Active sample capillary ion source for ambient mass spectrometry coupled with optical near-field microscopy."
4:05-4:25	David Lewis, Nanonics "Nanometric optical analysis and spectroscopy for solid state devices."
4:25-4:45	Robert Shaw, Oak Ridge National Laboratory "Using near field SHG to watch ZnO nanowires react with CO2"
4:45-5:05	Wei Liu, WiTec "Characterization of Nanomaterial with Confocal Raman-AFM."
5:05-5:25	David Faddis, Nanosurf "FluidFM Technology: Combining AFM with Nanofluidics for Applications in Life Science and Beyond"
5:25-7:00	Vendor Interaction and Demos
7:00	Return to Hotel



V

Nanoscale Imaging for Energy Applications

Vednesday, Sept	tember 12, 2012
Room SNS 156	Keynote Speaker
	Zhong L. Wang, Georgia Institute of Technology
8:30-9:30	"Nanopiezotronics and nanowire based energy harvesting."
Room SNS 156	SPM for Energy Applications
9:30-10:00	Vijay Sethuraman, Brown University
	"Strain Dynamics in Energy Materials"
10:00-10:20	Break
10:20-10:50	Mark Hersam, Northwestern University "Scanning probe microscopy of energy materials."
10:50-11:10	Keith Jones, Asylum Research "In-situ Cantilever Shape Profiling – Implications for Quantitative Nanomechanical and Functional Measurements."
11:10-11:30	Amit Kumar, Oak Ridge National Laboratory "Scanning probe ESM measurements of fuel cell materials."
11:30-12:00	Tom Arruda, Oak Ridge National Laboratory "Probing irreversible reactions in Li-air batteries."
12:00-1:00	Lunch
1:00-3:00	Poster Session and Vendor Interaction
3:00-3:20	Stephen Minne, Bruker AXS <i>"Atomic Imaging and Electrical Characterization with Peak Force</i> <i>Tapping."</i>
3:20-3:40	Laurene Tetard, Oak Ridge National Laboratory "Mode Synthesizing Atomic Force Microscopy – a multifrequency platform for nanomechanical and nanochemical characterization of soft materials."
3:40:-4:00	Andrew Stack, Oak Ridge National Laboratory "In situ mineral reaction rates from AFM."
4:00-4:20	Enrico Colla, École Polytechnique Fédérale de Lausanne "Direct Observation and modeling of local stress release by ferroelastic domain switching in piezoelectric materials."
4:20-4:30	Break
4:30-4:50	Adam Kollin, RHK "Advanced SPM Instrumentation."
4:50-5:10	Thomas Schulmeyer, SPECS "New milestones in scanning probe microscopy."
5:10-7:00	Vendor Interaction and Demos
7:00	Return to Hotel



hursday, Septer	nber 13, 2012
Room SNS 156	AVS Sponsored Session
8:30-9:00	Philip First, Georgia Institute of Technology "Measuring the effect of scalar and vector potentials in graphene."
9:00-9:20	Kendal Clark, Oak Ridge National Laboratory "Mapping electrical conductivity across grain boundary in epitaxial graphene"
9:20-9:40	Zhigang Jiang, Georgia Institute of Technology "Point-contact Spectroscopy Study of Topological Insulators and Superconductors"
9:40-10:00	Michael D. Williams, Clark Atlanta University "Electronic structure of electronic field effects in epitaxial graphene"
10:00-10:20	Break
10:20-10:50	Minghu Pan, Oak Ridge National Laboratory "Oxide surfaces on atomic scale: STM quest."
10:50-11:20	Ying-Hao Chu, National Chiao Tung University "Complex oxide interfaces and nanostructures."
11:20-11:40	Bridget Rogers, Vanderbilt University "Synthesis and Characterization of Ceramic Oxide Phosphors"
11:40-12:00	Steve Overbury, Oak Ridge National Laboratory "Searching for active sites on carbon catalysts."
12:00-1:00	Lunch
1:00-1:30	Lincoln Lauhon, Northwestern University "Correlated functional imaging of energy interconversion in hybrid nanostructures."
1:30-1:50	Subhadra Gupta, University of Alabama "Optimization of Fully Perpendicular Magnetic Tunnel Junctions by Rapid Thermal Annealing"
1:50-2:10	T. Zac Ward, Oak Ridge National Laboratory "Controlling electronic phase competition in confined manganites"
2:10-2:40	Peter Maksymovych, Oak Ridge National Laboratory "Tunneling microscopy of molecular and thermoelectric surfaces."
2:40-4:00	Vendor Interaction and Demos
6:30-8:30	Dinner & Business Meeting, AVS TN Valley Chapter



Nanoscale Molecular and Elemental Cartography: Combining Atomic Force Microscopy with Mass Spectrometry

Gary J. Van Berkel, Ph.D. Distinguished Research Staff Oak Ridge National Laboratory Oak Ridge, TN 37831-6131

Currently available analytical technologies for nanoscale interrogation face a trade-off between spatial resolution and the specificity of the chemical information they can obtain. We believe that divide can be bridged by combining atomic force microscopy, AFM (and its multiple variants of physical and chemical characterization), with the definitive elemental and molecular characterization capabilities of mass spectrometry (MS). The resulting multimodal imaging capability of this combination (aka, molecular or elemental cartography) has the potential to provide unprecedented physical and chemical characterization capabilities at the nanometer scale. This presentation will discuss how material of interest from AFM mapped surfaces might be sampled for mass spectral interrogation using AFM enabled nanoscale thermal desorption, liquid extraction or laser ablation. The ability to physically and chemically image systems under ambient conditions (whether in air or in liquid) that this combination provides is a growing field of interest because preparation of samples for nanoscale analysis in vacuum (such as would be the case for SIMS, secondary ionization mass spectrometry) is often difficult, time-consuming, and even at times not possible.

This work was supported by the Division of Chemical Sciences, Geosciences, and Biosciences, Office of Basic Energy Sciences, United States Department of Energy. ORNL is managed by UT-Battelle, LLC for the U.S. Department of Energy under contract DE-AC05-000R22725.



Multi-modal Imaging: Combining MALDI MS Imaging with Microscopy and MRI

Richard M. Caprioli Vanderbilt University, Nashville, TN, U.S.A.

Imaging MALDI MS (IMS) produces molecular images of peptides, proteins, lipids and metabolites present in intact tissue sections. It employs desorption of molecules by direct laser irradiation to map the location of specific molecules from fresh frozen and formalin fixed tissue sections without the need of target specific reagents such as antibodies. Molecular maps can be directly correlated to known histological regions within the tissue. Images are produced in specific m/z (mass-to-charge) values, or ranges of values, typically covering the MW range 1000-100,000. Individual m/z values derived from each pixel can then be assembled to produce selected molecular images. Recent technological advances both in sample preparation and instrumental performance has allowed IMS to achieve a high spatial resolution (<10 microns) and at high speeds so that a typical sample tissue (e.g., a whole mouse brain section) can be imaged in less than 10 min. Both fresh frozen and formalin fixed tissues can be analyzed.

Correlation of IMS with other imaging modalities such as microscopy and magnetic resonance spectroscopy enables more detailed spatial information to be obtained. For example, we have developed a protocol termed histology-directed molecular analysis whereby only selected areas of cells in the tissue are of interest are ablated and analyzed based on studies performed by microscopy and other histology protocols. We have also correlated MRI images with IMS. In these studies, sections of tissue from an animal previously analyzed by MRI of the intact animal are scanned and the registered with sections from a rendered volume of tissue from MRI. 3D images can be produced in this manner when serial sections are employed throughout the animal specimen. A new initiative will address direct combination of data from other imaging technologies in an image fusion process to sharpen MS images. Finally, the presentation will briefly discuss strengths and weaknesses of the technology.



Nanoscale Chemical Imaging of Biomaterials with Cluster SIMS

Nicholas Winograd State University, University Park, PA 16802, USA.

Bombardment of molecular solids with polyatomic projectiles allows interrogation of the sample with reduced chemical damage accumulation. Hence, it is now possible to perform depth profiling experiments with a depth resolution of less than 10 nm. In our hands, the projectile of choice is C_{60} due to the fact that the ion beam can be focused to a 250 nm spot size, and erosion of the sample can be performed with minimal chemical damage, especially at low temperature. With this combination of properties, it is feasible to think about creating 3-dimensional molecule-specific images.

A basic impediment to accomplishing this goal involves the fact that the SIMS images provide only chemical information and no direct depth information. The measureable quantity is the incident ion beam fluence, which can indirectly be related to depth, but independent measurements are required. The formation of topography and differential sputtering effects across the sample surface can also degrade the quality of the 3-D rendering when 2-D images are stacked. We have employed AFM in combination with SIMS imaging to develop protocols for correcting for these phenomena. Here, examples are shown using a patterned trehalose thin film and an Irganox delta layer reference material provided by NPL in the U.K. The idea is to provide chemical information with SIMS, and the depth information, acquired at each pixel in the image, using AFM. In addition to examining eroded craters directly, we have also developed a wedge-beveling technique that allows sputtering yield and topography to be determined with a single SIMS measurement and a single AFM measurement.

The long term aim of developing these protocols is to be able to acquire high resolution chemical images of single biological cells. So far, it appears that differential sputtering effects are not too serious for these samples. The combined SIMS/AFM strategy developed here will be important for verifying these initial observations. Finally, there is an emerging interest in gas cluster ion sources, namely Ar_{2500} , since even less chemical damage than C_{60} is observed, and the depth resolution during erosion appears to be less than 5 nm. It is anticipated that the methods described here will be useful in helping to characterize the behavior of these and other projectiles as they become incorporated into more laboratories. In general, we show that the AFM/SIMS combination is a powerful tool for 3-dimensional chemical imaging.



Nanoscale Chemical, Mechanical and Thermal Analysis using Atomic Force Microscopy

Kevin Kjoller Anasys Instruments

Atomic force microscopy (AFM) is a powerful tool to characterize the nanoscale topography of materials. Using the AFM probe to perform other analytical techniques couples the high spatial resolution capabilities of the AFM with the unique characterization inherent in that technique. This presentation will describe how thermal analysis and IR spectroscopy can be performed by making use of the AFM probe as the signal detector.

Nanoscale thermal analysis (nano-TA) uses a microfabricated thermal probe to locally heat a region on a sample above its transition temperature. This allows monitoring spatial variations in crystallinity, molecular weight or cross linking. These unique probes can also be used for a newly developed technique called Lorentz contact resonance. This technique combines a directed magnetic field with an AC current in the thermal probe enabling wide bandwidth force excitation, this can be used to excite multiple flexural and torsional resonances of the probe. These resonances are modified by the flexural and lateral stiffness and damping properties of the sample allowing characterization of these properties at the nanoscale. Further, by controlling the tip temperature the technique can also rapidly probe the temperature dependence of the nanomechanical properties of the sample surface.

Nanoscale IR spectroscopy (AFM-IR) couples a pulsed tunable IR source with an AFM. The AFM is used to measure the local rapid thermal expansion of a sample caused by infrared absorption. AFM-IR can be used both to obtain absorption spectra covering a range from 900 to 3600 cm⁻¹ at arbitrary points and to spatially map IR absorption at selected wavelengths. In addition, the polarization of the incident light can be rotated allowing characterization of oriented materials. By analyzing the various absorption bands of a sample, unknown materials can be identified with high spatial resolution and information complementary to nano-TA can be obtained.



Optical Near and Far-field Femtosecond Laser Ablation for Nanoscale Chemical Imaging

Vassilia Zorba Lawrence Berkeley National Laboratory, Berkeley, CA

Ultrafast laser-based schemes are used to improve the limits of detection in laser plasma spectroscopy at small spatial and depth material sampling length scales. Configurations used include single and double-laser pulse geometries in the optical far-filed as well as near-field laser ablation. Sub-micron spatial resolution was systematically achieved in the optical far-field yielding attogram-scale limits of detection in model material systems. In addition, nm-scale depth resolution was achieved using fs-laser irradiation for the study of interfacial layers in electrochemical energy storage systems. Finally, we demonstrate the use of double-laser pulse configurations as secondary excitation sources that offer significant spectral emission intensity and sensitivity enhancements, and assist towards further minimizing the requirements for ablated mass in laser-ablation based chemical analysis as they relate to nanoscale energy applications.



Active sample capillary ion source for ambient mass spectrometry coupled with optical near-field microscopy

<u>Maryia Nudnova</u>, Renato Zenobi ETH Zurich, Zurich, SWITZERLAND

Mass spectrometry can potentially be used as an analytical technique for acquiring highly localized chemical information. It has been shown that scanning probe microscopy (SNOM) combined with MS allows acquiring both µm-scale topographic and chemical imaging at ambient pressures. In the SNOM-MS combination, the laser beam, which is allowed through a SNOM tip, performs an ablation thus providing the analyte for further MS analysis. Higher spatial resolution means a smaller diameter of the ablation spot and, consequently, smaller craters from which the ablation takes place. Thus, efficient sampling and subsequent ionization of ablated material are of key importance for effective nanoscale chemical imaging at atmospheric pressure. Plasma based ambient ionization sources can provide both efficient sampling and ionization. In this work, we developed a plasma-based active capillary. The constructed active capillary was optimized for performing surface analysis of solid samples with high spatial resolution.

Imaging mass spectrometry with high spatial resolution requires ionization of very small amounts of substances preferably at atmospheric pressure. A special ionization source capable of achieving this goal was constructed in our laboratory. This source is based on a dielectric barrier discharge (DBD).

The «tube etched» SNOM tips were used as for topographical data acquisition as for near-field laser ablation. A Nd:YLF laser (Triton, Spectra Physics, Mountain View, CA) was used for near field ablation. The laser beam was focused onto the optical fiber blunt end, whose etched end was glued onto the tuning fork of the SNOM. In the experiments, the shear-force scan head was moved in a point-to-point mode and was stopped at each target point for 1.5 sec. The laser operation was synchronized with the SPMLab software by pulse generator (model 555, Berkley Nucleonics Corp., San Rafael, CA). The typical ablation pattern is presented on Fig. 1.



Fig. 1 A 50×50 µm area of sinapic acid pellet surface scanned after 25 near-field laser ablation events with 85 nJ per pulse laser. A scanning optical near-field microscopy (Topometrix Lumina, Veeco Metrology Inc., Santa Barbara, CA, USA) was used for data acquiring.

Two DBD sources with different electrode configuration were constructed for coupling mass spectrometer with SNOM. The sources are capable of ionizing gaseous and evaporated liquid and solid samples during their transport into the vacuum region of a mass spectrometer. The geometry of the electrode was shown to be a key determinant of ionization efficiency. The taper cap-tape active capillary was found to be ten times more sensitive that the wire-tape capillary. It is possible to analyze solid samples that can easily evaporate, using a mass spectrometer equipped with the active capillary presented here. In this case, no spatial resolution is provided. However, we believe that this kind of application will also be interesting for surface analysis. For example, a contamination of a solid surface with volatile organic molecules can be analyzed and monitored by the technique developed. In the case of solid samples, neutrals for ionization can be generated both by laser ablation and by evaporation from a solid surface.

The sensitivity of the constructed active capillary is currently at the level of 100 fmol for the evaporated samples. The combination with far-field laser ablation renders the active capillary with the current sensitivity suitable for surface analysis with high spatial resolution, although the sensitivity of the constructed active capillary is still not sufficient for it to be combined with near-field laser ablation. The applicability of the constructed plasma source to near-field laser ablation needs further improvements in sensitivity.



Nanometric optical analysis and spectroscopy for solid state devices

Steven M. Barnett,^{1,2} Aaron Lewis,¹ David Lewis,¹ Hesham Taha,¹ and Rimma Dechter¹ ¹Nanonics Imaging, Har Hotzvim Hi Tech Park, 19 Hartum Street, BYNET Building, Jerusalem 97775 Israel ²Barnett Technical Services, PO Box 582922, Elk Grove, CA 95758

Advanced SPM measurements on energy-related materials will be discussed. The study of graphene will be presented using AFM-Raman and Tip Enhanced Raman Spectroscopy (TERS) through the use of SPM tips composed of tapered glass fibers with gold balls positioned at the tip for Raman enhancement. The need for tip scanning independent of sample scanning in order to achieve large enhancements will be discussed. Other applications to be presented will include the incorporation of multiple SPM probes in the near-field for advanced near-field scanning optical microscopy (NSOM) and electrical characterization of energy materials.



Using near field SHG to watch ZnO nanowires react with CO₂*

<u>Robert W. Shaw</u>* and Katherine L. Cimatu Chemical Sciences Division Oak Ridge National Laboratory

Zinc oxide nanowires (NWs) are not stable in the atmosphere, a limitation for their utility for electro-optical applications such as solar energy cells and light emitting diodes. An ability to monitor material degradation on a nanometer scale is a powerful tool for a fundamental understanding of surface chemical processes. Nanoscale chemical images of *individual* bare and alumina-coated zinc oxide nanowires were recorded using tip-enhanced second harmonic generation (SHG) spectroscopy before and after exposure to carbon dioxide and water vapor. Images were collected for the *same* bare nanowire after each successive two-day exposure period. Conversion of the bare ZnO NW to SHG-inactive zinc carbonate was evident from farfield and near-field SHG images, as well as simultaneously recorded atomic force microscopy (AFM) data. The near-field SHG signal provided a profile of the evolving NW with a spatial resolution approaching 100 nm. Exposed 3-nm ALD alumina-coated ZnO NWs showed reduced, but still observable, degradation. Further nanometer-scale imaging should lead to the discovery of protective layers to prevent or delay ZnO degradation.

*Research sponsored by the Division of Chemical Sciences, Geosciences, and Biosciences, Office of Basic Energy Sciences, U.S. Department of Energy.





Characterization of Nanomaterial with Confocal Raman-AFM

Wei Liu WITec Instruments Corp.

Characterization of nanoscale material has been growing fast and impacting more and more key applications in material science. One particular approach is the combination of both physical and chemical tools together to study the material at nanometer scale. AFM has been successfully used to investigate sample surface physical properties for years, while recently developed Raman imaging capability provides potential chemical insights of the sample. WITec Alpha 300 microscope is such a tool to study the nanomaterial with both physical and chemical imaging capabilities, a few applications will be shown.



Carbon Nanowire characterization: Confocal Raman image on left and AFM topo image on right.



FluidFM: Combining Atomic Force Microscopy and Nanofluidics in a Universal Liquid Delivery/Extraction and Manipulation System

David Faddis Nanosurf, Inc 999 Broadway, Suite 205, Saugus, MA 01906 781-549-7361, faddis@nanosurf.com

The FluidFM combines an Atomic Force Microscope (AFM) with micro-channeled cantilevers for local liquid dispensing and extraction, and manipulation of single living cells under physiological conditions. A continuous fluidic circuit is achieved that extends from an external liquid reservoir, through the probeholder and the hollow cantilever to the tip aperture. Both overpressure and an under-pressure can be applied to the liquid reservoir and hence to the built-in fluidic circuit. A variety of single cell experiments are made possible by utilizing pressure control through this novel cantilever. We will discuss some of these potential applications that include:

- Utilizing the nano-fluidic channel in the cantilever to dispense soluble molecules with submicrometer precision. For example, selective, local perforation of the cell membrane allows for introduction of dyes into a living cell.
- Conversely, the pressure may be reversed to utilize the cantilevers for selective extraction of materials from a single cell.
- Combined with precise AFM positioning capabilities, the FluidFM may be used to displace living organisms with micrometric precision in a non-destructive manner. Thus, a technique may be devised for selectively identifying and sorting individual cells based on measurable properties.
- AFM force spectroscopy capabilities may be utilized to measure the adhesion strength of individual cells as they are displaced from the substrate.

The universality and versatility of the FluidFM will stimulate original experiments at the submicrometer scale not only in biology but also in physics, chemistry, and material science.





Piezotronics and Piezo-phototronics

Zhong Lin (Z.L.) Wang School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta USA E-mail: zlwang@gatech.edu

Piezoelectricity, a phenomenon known for centuries, is an effect that is about the production of electrical potential in a substance as the pressure on it changes. The most well known material that has piezoelectric effect is the provskite structured Pb(Zr, Ti)O₃ (PZT), which has found huge applications in electromechanical sensors, actuators and energy generators. But PZT is an electric insulator and it is less useful for building electronic devices. Wurtzite structures, such as ZnO, GaN, InN and ZnS, also have piezoelectric properties but they are not extensively used as much as PZT in piezoelectric sensors and actuators due to their small piezoelectric coefficients. In fact, due to the polarization of ions in a crystal that has non-central symmetry, a piezoelectric potential (piezopotential) is created in the crystal by applying a stress. For materials such as ZnO, GaN, InN in the wurtzite structure family, the effect of piezopotential to the transport behavior of charge carriers is significant due to their multiple functionalities of piezoelectricity, semiconductor and photon excitation. By utilizing the advantages offered by these properties, a few new fields have been created. Electronics fabricated by using inner-crystal piezopotential as a "gate" voltage to tune/control the charge transport behavior is named *piezotronics*, with applications in strain/force/pressure triggered/controlled electronic devices, sensors and logic units. *Piezo-phototronic effect* is a result of three-way coupling among piezoelectricity, photonic excitation and semiconductor transport, which allows tuning and controlling of charge generation, separation, transport and/or recombinatioin in electro-optical processes by strain induced piezopotential. Piezotronics is likely to have important application in sensors, humansilicon technology interfacing, MEMS, nanorobotics and active flexible electronics. The role played by piezotronics in interfacing human-CMOS technology is similar to the mechanosensation in physiology. We anticipate the near future applications of piezotronics and piezo-phototronics in sensor network, bioscience, human-machine interfacing and integration, and energy sciences (LED, solar cell).

- [1] Z.L. Wang and J.H. Song 'Science, 312 (2006) 242.
- [2] Z.L. Wang, Nano Today 5 (2010) 540.
- [3] W.Z. Wu, Y.G. Wei and Z.L. Wang, Adv. Materials, 22 (2010) 4711.
- [4] W.Z. Wu, Y.G. Wei and Z.L. Wang, Nano Letters, 11 (2011) 2779
- [5] Y.F. Hu, Y.L. Chang, P. Fei, R.L. Snyder and Z.L. Wang, ACS Nano, 4 (2010) 1234.

[6] Q. Yang, X. Guo, W.H. Wang, Y. Zhang, S. Xu, D.H. Lien, Z.L. Wang, *ACS Nano*, 4 (2010) 6285.

[7] Q. Yang, W.H. Wang, S. Xu, Z.L. Wang, Nano Letters, 11 (2011) 4012.

[8] Z.L. Wang, Adv. Mater., DOI: 10.1002/adma.201104365.

[9] Personal website: <u>www.nanoscience.gatech.edu</u>



STRAIN DYNAMICS IN ENERGY MATERIALS *In situ* Electrochemical and Mechanical Measurements in Lithium-battery Electrodes

Vijay A. Sethuraman Brown University

Rechargeable lithium-ion batteries, widely used to power consumer electronics, are now being considered for use in electric vehicles (EVs). Five major obstacles remain in their mainstream adoption: energy and power density (range and acceleration of the EV, respectively), durability, safety and cost. Replacing graphite, the most commonly used negative electrode, with silicon can increase the specific energy density of a lithium-ion cell by *ca.* 30%. However, Si exhibits large volume expansion and contraction during lithiation and delithiation (charging and discharging), and consequently suffers from mechanical decrepitation and thus, capacity fade with poor cyclability. Since mechanical damage in an electrode is driven by stress and strain fields that are induced during repeated charge/discharge cycles, it is essential to characterize stress and strain evolution in the electrode. Furthermore, for accurate modeling of battery performance, cycle life and reliability, there is a need to accurately measure the mechanical properties of lithiated silicon (an amorphous alloy of silicon and lithium). In this talk, I will present *in situ* stress measurements on both thin-film and composite silicon electrodes¹ during the electrochemical lithiation and delithiation processes as well as an experimental determination of biaxial modulus of lithiated silicon.^{2,3}

Further, I will present an analysis of the dependence of electric potential on the state of stress of a lithiated-silicon electrode. Based on Larché and Cahn chemical potential for a solid solution, a thermodynamic argument is made for the existence of the stress-potential coupling in lithiated-silicon and, based on the known properties of the material, the magnitude of the coupling is estimated to be ca. 60 mV/GPa in thin-film electrode geometry. An experimental investigation has been carried out in which the stress was varied incrementally while measuring the electrode potential simultaneously; the relation between stress change and electric potential change is measured to be 100 - 120 mV/GPa, which is within the same order of magnitude as the prediction. I will discuss the importance of this coupling in the context of interpreting the hysteresis loops observed in potential *vs.* state-of-charge plots.⁴

Finally, I will explore challenging yet rewarding research opportunities that exist in the diagnostics realm of the electrochemical energy-storage field using the mechanicalcharacterization techniques developed at Brown University as well as the Center for Nanophase Materials Sciences at Oak Ridge National Laboratory.

Relevant publications:

- 1. V.A. Sethuraman, M.J. Chon, M. Shimshak, V. Srinivasan, P.R. Guduru. *In Situ* Measurements of Stress Evolution in Silicon Thin Films during Electrochemical Lithiation and Delithiation. *Journal of Power Sources*, 195, 5062-5066, 2010.
- V.A. Sethuraman, M.J. Chon, M. Shimshak, N.P. Van Winkle, P.R. Guduru. *In situ* Measurements of Biaxial Modulus of Si Anode for Li-ion Batteries. *Electrochemistry Communications*, 12 (11), 1614-1617, 2010.
- 3. V.A. Sethuraman, V. Srinivasan, A.F. Bower, P.R. Guduru. *In situ* Measurements of Stress-Potential Coupling in Lithiated Silicon. *Journal of the Electrochemical Society*, 157, A1253-A1261, 2010.
- 4. V.A. Sethuraman, N. Van Winkle, D.P. Abraham, A.F. Bower, P.R. Guduru. Real-time Stress Measurements in Lithium-ion Battery Negative-electrodes. *Journal of Power Sources*, 206, 334-342, 2012.



Scanning probe microscopy of energy materials

Mark C. Hersam Northwestern University http://www.hersam-group.northwestern.edu/

Probing energy materials at the nanoscale has the potential to provide insight into the operation, performance, and reliability of energy conversion and storage devices such as photovoltaics, batteries, and catalysts. Towards this end, our research group develops and employs multiple scaning probe techniques that enable concurrent characterization of the physical structure, chemistry, and electronic properties of energy materials and devices in a diverse range of environments. For atomic resolution characterization, ultra-high vacuum (UHV) scanning tunneling microscopy (STM) is well-suited for conductive substrates such as chemically modified graphene [1-5]. On the other hand, ambient conductive atomic force microscopy (cAFM) allows nanoscale variations in electrical conductivity to probed on partially insulating substrates [6-8]. Furthemore, by integrating custom optics with cAFM, spatial distributions in electroluminescence [9] and photocurrent [10] can be probed in fully fabricated organic light-emitting diodes [11] and organic photovoltaic devices [12], respectively. Finally, ion conductance microscopy (SICM) enables nanoscale electrochemical scanning characterization of Li-ion battery electrodes in their native electrolyte environment [13,14]. The relative merits and limitations of each of these techniques will be discussed in the context of energy materials and devices.

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In-situ Cantilever Shape Profiling – Implications for Quantitative Functional and Nanomechanical Measurements

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Ouantitative mechanical and functional measurements using the Atomic Force Microscope (AFM) are inextricably linked to quantifying the response of the cantilever. The cantilever is an extended mechanical object with normal modes that depend on the boundary conditions both of the cantilever structure itself and on the boundary conditions imposed by the tip-sample interactions. By using the encoded and motorized optical beam controls on a commercial microscope (schematically indicated in Figure 1), I have been able to directly measure the extended shape of the cantilever as it interacts with various samples and in a variety of measurement modes. Integration of the laser scanning mechanism directly in the AFM has enabled these shape measurements during normal AFM operation in a variety of imaging modes ranging from force curves and contact modes to AC modes. This presentation will focus on contact and force curves measurement modes. Piezoresponse Force Microscopy (PFM) and Electrochemical Strain Microscopy (ESM) have led to significant progress in our understanding of the nano-electromechanical origins of function in a large variety of materials. In many cases of interest, the response of a cantilever does not behave as a simple, single-mode system. To elucidate the effects of local and long-ranged drive mechanisms on the multi-modal cantilever probe, the response of the cantilever as a function of both frequency and optical beam spot position has been measured in-situ while the cantilever is directly probing a functional material, either with PFM or ESM. A typical resulting spectrogram is shown in Figure 2. Two interesting results are (i) the crossover between the dc and first contact mode is measurable at surprisingly low frequencies, and (ii) contrary to initial expectations, operating on resonance actually provides more stable and reliable quantification of the cantilever drive. This is explained in terms of the high Q-factor of the resonance. There has been significant interest in quantifying the nanomechanical response of samples using force curves. As has been pointed out, the response of the cantilever during force curves has significant harmonic terms that contain most of the information on the tip-sample interactions. Previously, the detection sensitivity, stiffness and phase of the cantilever response at these harmonic frequencies have been assumed to be constant. I have examined this assumption while modulating the tip-sample interaction forces as a function of load. Surprisingly, there is often a low frequency crossover similar to that observed for the functional imaging the 0th and first contact resonance. Among other considerations, one significant factor in the crossover frequency is the presence of long-range forces. This may have far-reaching implications in quantifying nanomechanical properties based on conventional force curve measurements - especially force curves performed at higher frequencies.



Figure 1 – A motorized stage with optical encoders integrated into our AFM can repeatedly position the spot on the cantilever to within 200nm. This allows accurate measurements of the cantilever shape as it interacts with the sample. Figure 2 – (a) amplitude and (b) phase as a function of drive frequency and spot position for a cantilever in contact with a ferroelectric lithium niobate sample. The first contact resonance is at ~300kHz and the second is at ~930kHz. Note the presence of a measured phase change for a spot position near the tip end of the cantilever. This phase change occurs for frequencies well below the first contact resonance.



Scanning Probe ESM measurements of fuel cell materials

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Electrochemical energy conversion systems based on gas-solid interactions such as solid oxide fuel cells (SOFC) are one of integral components of current and future energy technologies. The energy conversion in these systems is underpinned by a series of complex mechanisms like ion and vacancy diffusion, electronic transport and solid-gas and solid-liquid reactions at surfaces and triple phase junctions. One of the critical steps in the SOFC and Li-air battery operation leading to large overpotentials and charge-discharge hysteresis is the kinetics of the oxygen oxidation reaction (ORR). It is important thus to explore the mechanisms behind this enhancement which remain elusive, largely due to the lack of experimental techniques capable of probing ORR on the nanoscale. Oxygen vacancies play a significant role in determining the functionality of electro-resistive devices, non-volatile memories based on resistive switching and solid oxide fuel cells. Traditionally, the study of the role of oxygen vacancies in these processes is limited by high activation temperature and macroscopic measurement techniques. Here, we demonstrate spatially resolved local probing of the thermodynamics and kinetics involving the generation and diffusion of oxygen vacancies by utilizing chemical expansivity of these oxides upon application of concentrated electric fields. Using Band excitation Electrochemical strain microscopy (ESM), a strongly confined electric field at tip is used to drive the oxygen vacancies in these oxide materials and the resulting localized electrochemical strain is detected. A high frequency periodic bias is applied on the oxide material and the PFM tip acts as a probe of the local displacement arising due to migration of oxygen vacancies. Local strain hysteresis loops driven by vacancy diffusion have slow dynamics and thus open up. Mapping the loop opening as a function of the final bias allows establishment of the onset and kinetics of the diffusion process. Signal relaxation measurements enable us to locally characterize the diffusion dynamics of the vacancies. In mixed ionic-electronic oxide systems, we also utilize current-voltage measurements to probe the electronic transport. Correlated mapping of the local oxygen vacancy movement and diffusivity has been achieved with a resolution of ~ 30 nm. The mapping of vacancies is shown on purely ionic oxides (Yttrium stabilized zirconia and Samarium doped Ceria). Systematic mapping of ORR/OER activity on bare and Pt-functionalized yttria-stabilized zirconia (YSZ) surfaces is demonstrated. This approach allows directly visualization of ORR\OER activation process at the triple-phase boundary. The electrochemical activity studies around alloyed Au-Pt nanoparticles has been studied. The electrical field-dependence of ionic mobility is explored to determine the critical bias required for the onset of electrochemical transformation, potentially allowing to deconvolute reaction and diffusion processes in the fuel cell system on a local scale. The results of ESM performed at elevated temperatures and controlled oxygen environments on solid electrolytes will also be discussed.



Probing Irreversible Reactions in Li-air Batteries

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Currently, there are enormous efforts underway to develop Li-air batteries,¹ primarily for the application of electric automotive power as theoretical energy densities are close to that of gasoline.² Unfortunately, many hurdles remain in the path between the current understanding of Li-air battery materials, operation and properties and their widespread deployment. The idealistic approach employs the following two electrochemical reactions for the anode and cathode respectively:

$$Li(s) \rightarrow Li + 1e -$$
(1)

$$O_2(g) + 4 e_- + 4Li_+ \rightarrow 2 Li_2O$$
 (2)

Note that each equation represents the most difficult challenges in both battery and fuel cell research. That is, the case of employing Li metal as a secondary battery (eq.1) and the well known sluggish oxygen reduction reaction kinetics (eq. 2, shown here for the reaction with Li). On top of these two challenges remains the choice of a proper high voltage electrolyte. Each of the three aforementioned components exhibit a number of irreversible or partially reversible processes (i.e. poorly soluble Li2O byproducts, irreversible electrolyte decomposition etc.). While many endeavors are underway to investigate the processes in eqs. 1&2, less emphasis is placed on electrolyte research, with the consensus being to implement systems that work well for Li-ion batteries (solvent/salt solutions). Though these work well in some cases, the poor solubility of LiO_x moieties and O_2 as well as solvent decomposition remain problematic. One promising class of solid state Li-ion conductor electrolytes is of the LISICON variety (lithium superionic conductor). These are solid state glass ceramic materials that exhibit reasonably high Li ion conductivity $(10^{-4} - 10^{-3} \text{ S cm}^{-1})$, good thermal and chemical stability and is impervious to water and O2 crossover. Despite the above, the stability of LISICON in alkaline conditions and in direct contact with Li metal is suspect and hence warrants further investigation.¹ At the CNMS we use Electrochemical Strain Microscopy (ESM)⁴ based methods to study ion dynamics and irreversible processes in Lithium Ion Conducting Glass Ceramics (LICGC).⁵ ESM is an atomic force microscopy (AFM) based technique that employs a band excitation (BE) signal on the tip to measure ion dynamics with nanoscale resolution. In the reversible mode areas of contrast denote regions of low and high ion mobility. In the irreversible mode, electrochemical reactions can be induced on the surface of solid electrolytes which allows for the study of irreversible processes with higher resolution than traditional electrochemical methods. This talk will summarize our most recent findings on LICGC.⁶⁻⁷ Reversible ESM contrast has be observed (low bias) illustrating the high Li ion conductivity in LICGC. However, if a critical bias is achieved (ca. -3.5V and higher), solid Li metal nanoparticles are formed on the surface of LICGC under the tip. Particle formation thermodynamics and kinetics have been studied and will be presented. In addition, the partial re-oxidation (reversibility) of Li nanoparticles has been observed and will be discussed.

Figure 1. (a) single point ESM strain spectroscopy measurement on LICGC surface showing the change in tip height (blue curve) during cyclic voltammetry, red line plotting the measured current (versus time). The sketch above illustrates the strain spectroscopy measurement of reversibility.

Acknowledgement

Research was sponsored by the Laboratory Directed Research and Development Program and conducted at the Center for Nanophase Material 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.

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Atomic Imaging and Electrical Characterization with





PeakForce Tapping

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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, we can achieve atomic resolution imaging along with maps of the tip-sample interaction on general and unrestrictive large sample systems. An atomic resolution image, taken in air, resolving the aromatic ring of Polydiacetylene (PDA) is shown as an example of this capability in Figure 1 below. Recently, we have been able to couple the resolving and mechanical mapping capabilities of PeakForce Tapping with some of the traditional SPM electrical measurement techniques, Tunneling AFM (TUNA) & Kelvin Probe Force Microscopy (KPFM), while operating in a ppm controlled environment equipped with 300 sun backside sample illumination. This has enabled us to correlat high resolution images showing height, adhesion, and photocurrent of the well-studied OPV bulk heterojunction system consisting of poly (3- hexylthiophene) (P3HT) donor and [6,6]-phenyl-C61 butyric acid methyl ester (PCBM) acceptor.

The PeakForce QNM adhesion image unambiguously reveals a nanoscale fibrillar structure, where fibrils surround more compact domains. P3HT is known to form fibrils. The photocurrent carrier can then be pinpointed in the PeakForce TUNA current maps, which resolve the photocurrent at the same 10nm feature size level. This resolution in the current map is enabled the elimination of lateral forces, and avoids the artifacts and loss of resolution plaguing conventional, contact mode based conductive AFM imaging. Control of oxygen and water partial pressures at the 1ppm level ensures the measurement is not affected by chemical degradation. Additional data, taken with PeakForce Tapping combined with KPFM will be presented, where through instrumentation and probe design, measurement scatter has been reduced to ~10 mV standard deviation.





Figure 1: Atomic resolution image of PDA using PeakForce Tapping. Figure 2: P3HT:PCBM (Courtesy of N. Sebaihi and P. Leclere, CIRMAP – UMONS, Belgium) In Situ Mineral Reaction Rates from AFM



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Accurate predictions of the rates of growth and dissolution of minerals are important for, among other things, evaluating disposal strategies for spent nuclear fuel, the remediation of toxic substances produced via energy production and inhibition of scale formation in oil, natural gas, and geothermal energy production. Historical methods of measuring these properties have difficulty in distinguishing the relative contributions of individual surface structures, such as monomolecular steps, to the overall rate. Thus, flow through atomic force microscopy (AFM) has been utilized over the past 15 years to measure growth and dissolution rates of minerals in situ. New techniques and instruments are being designed to examine these properties under extreme environments such as under super-critical CO2 or high temperature. New computational techniques are giving us the ability to relate these measurements to intrinsic reactivities. In this talk, we will review some of the recent work in this field and discuss some of the challenges that working under condensed aqueous (or other) solvents presents for AFM. Particular attention will be paid to the reactions of sparinglysoluble salts (calcium carbonate, barium sulfate) and the effects of impurities (e.g., strontium).



Direct observation and modeling of local stress release by ferroelastic domain switching in piezoelectric materials

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Piezoelectric ceramics used in applications for harvesting energy from environment sources are often subjected to extreme and not always predictable mechanical conditions (for example wind speeds or water waves). In order to minimize the limitations imposed by the typical brittleness of these materials, different approaches to increase their toughness were recently explored and successfully applied.

In addition to intrusive approaches, like the inclusion of nanoparticles of metastable tetragonal zirconia, stress induced ferroelastic switching can provide an efficacious toughening mechanism (so called ferroelastic toughening) by releasing internal stresses through the reorientation of non-180 degree domains and preserving the piezoelectric properties of the materials. The optimization of this mechanism requires solid knowledge and understanding of the interaction between the tip of a progressing crack and the configuration of the involved ferroelestic domains.

Piezoelectric Force Microscopy (PFM) obviously represents an ideal tool to observe and characterize the ferroelectric domain reorientations in thin piezoceramic bars. In this contribution we will present an experimental approach allowing to control the crack formation and its growth and to estimate in advance the path of the fracture, in order to map the initial configuration of ferroelectric domains, to be compared with the configuration after the fracture. Results on PZT-53/47 ceramics used in multilayer actuators will be presented and discussed.

Phase-field modeling of ferroelastic toughening based on Landau-Ginzburg-Devonshire theory will be presented and the primary qualitative factors that control the magnitude of ferroelastic toughening identified and discussed.

This work was supported by the EU project CP-IP 212394 Hiper-Act.



Fascinating ferroelectric domain patterns in PZT ceramics, before and after fracturing



Advanced SPM Measurements

Adam Kollin RHK Instruments

New discoveries and advances in Nanotechnology depend on continuous innovation in research instrumentation. This is especially true for the crucial nanotech tool, the SPM. As a specialist and leader in UHV SPM, RHK provides ever greater innovative capabilities in each of its SPM product lines, many of which are truly unique, one-of-a-kind ground-breaking instruments. This talk will highlight RHK's latest generation of research instruments and the results that customers have obtained with them.



New Milestones in Scanning Probe Microscopy: Graphene on Rh(111) studied by DFT, STM and NC-AFM using KolibriSensorTM and TytoTM

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Graphene, a single layer of carbon atoms ordered in a "chicken-wire" lattice [1], is proposed to be used in many technological applications. One of the promising systems on the basis of graphene is its interface with metallic substrates [2]. Here graphene can be used as a protection layer for the underlying substrate, as a spin-filtering material separating two layers of a ferromagnetic material, or, in case of its growth on a lattice

mismatched surfaces [for example, Ir(111), Rh(111), or Ru(0001)], as a template for the preparation of ordered arrays of clusters.

For graphene on Rh(111) [Fig.1(a-c)] several regions of different arrangements of carbon atoms above a Rh(111) substrate can be found: ATOP [A; carbon atoms are above Rh(S-1) and Rh(S-2) atoms], HCP [H; carbon atoms are above Rh(S) and Rh(S-2) atoms], FCC [F; carbon atoms are above Rh(S) and Rh(S-1) atoms], and BRIDGE [B; Rh(S) atoms bridge the carbon atoms]. These places are marked in Fig.1(a) by circle, down-triangle, square, and stars, respectively. Among them, the BRIDGE positions are expected to be the most energetically favorable for the nucleation of deposited atoms on top of a graphene layer.

In this contribution we present the combined study of the graphene/Rh(111) system via application of the state-of-the-art DFT calculations, STM, and NC-AFM. The calculated imaging contrast for STM between all high-symmetry positions for graphene/Rh(111) is in very good agreement with experimental results and this contrast does not depend on the sign of the bias voltage applied between a tip and the sample. As opposed to the latter observation, the imaging contrast in atomically-resolved AFM measurements depends on the frequency shift of the oscillating tip that can be understood on the basis of measured force-spectroscopy curves.

Fig.1. (a) STM image, (b) crystallographic structure, (c) calculated STM image, and (d) NC-AFM image of the graphene layer on Rh(111).

For this the KolibriSensorTM, a new quartz sensor that excels in its performance reliability is used either for RT to HT studies in an SPM Aarhus 150 or for LT studies in the new Tyto scan head mounted into a JT-SPM.

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Measuring the effects of scalar and vector potentials in graphene

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Through scanning tunneling microscopy (STM) and spectroscopy (STS), characterization of graphene's local electronic properties is accomplished by the application of scalar potentials from quasistatic electric fields and vector potentials from magnetic fields. Conversely, the electronic structure of strained graphene can be described as the influence of effective vector and scalar potentials. We use STM and STS to investigate the local electronic structure of "twisted" graphene in high magnetic fields and to probe graphene nanostructures strained through the constraint of lattice matching to a silicon carbide substrate. In twisted graphene (atomic layers rotated with respect to one another), we find interlayer interactions on the scale of 10 meV, whereas in strained graphene, the electronic structure is reshaped on an energy scale approaching 1eV. The potential for such tailoring of graphene's electronic structure may have application to low-power ballistic devices or interconnects.

* Some work in collaboration with the NIST Center for Nanoscale Science and Technology

[†] Funded in part by NSF and NRI-INDEX.



Left: Epitaxial graphene growing over the top corner of a 15-nm-high step in the SiC substrate. *Middle:* STS at the color-coded points labeled at left (left to right positions correspond to spectra bottom to top). Thin black lines show electron-hole symmetry of spectra. *Middle (inset):* Scaling of strain-induced "pseudo–Landau level" energies with index *N. Right:* Atomic imaging near the position of the labels in the left image. Atom heights in both images have been magnified to enhance visibility on the curved edge; actual corrugation ~2 pm.



Mapping Electrical Conductivity across Grain Boundaries in Epitaxial Graphene

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Single layers of graphene formed on SiC look to be a promising system for the realization of graphene electronics. To utilize the full potential of graphene on SiC a complete understanding of the physical and electronic properties of this system is needed. Scanning Tunneling Microscope (STM) images along with scanning tunneling spectroscopy is used to characterize the graphene sample surface. STM images clearly show the distinction between 1 monolayer (ML) and 2ML regions and this transition is further confirmed by point spectroscopy and spectroscopic mapping across the boundary. Defects, grain boundaries, step edges and other potential scattering centers are thought to play a major role in the electronic properties, especially in transport, along the graphene sheets. Using a low temperature four-probe scanning tunneling microscope, potentiometry measurements are performed on the epitaxial graphene grown on 4H-SiC. Potentiometry maps spanning the transition from 1ML to 2ML graphene show a contrast change indicating a potential change at this interface. Results of the transport along this potentially revolutionary new electronic system will be presented. This research was conducted at the Center for Nanophase Materials Sciences, U.S. Department of Energy.



Point-contact Spectroscopy Study of Topological Insulators and

Superconductors

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When topological insulators are in contact with a superconductor, novel proximity effect occurs. Theory predicts that the proximity induced superconducting state is spinless and *p*-wave like, and Majorana bound states may appear at the edges. In topological superconductors, on the other hand, spin-orbit coupling is expected to induce a triplet component on the order parameter and gapless Andreev bound states may form on the surface. Here we present our point-contact spectroscopy studies of topological insulators and superconductors. Specifically, we use a superconducting Nb tip approaching the surface of topological insulator Bi2Se3 or use a normalmetal Au tip on the surface of topological superconductor CuxBi2Se3, to measure the interface conductance as a function of bias voltage, temperature and magnetic field. We find that a superconducting state can be indeed induced at the Nb/Bi2Se3 interface, evidenced by a zerobias conductance peak in the point-contact spectra. A zero-bias conductance peak is also observed for the Au/CuxBi2Se3 point-contact, while the spectra near the gap and the underlying physics are quite different.



Electronic structure of electronic field effects in epitaxial graphene

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Surface sensitive ultraviolet photoemission spectroscopy is used to study the modification of the electronic structure of multilayer epitaxial graphene grown on the carbon face of silicon carbide with applied electric fields. The fields are applied normal to the surface plane of the material. First principle density functional theory with interlayer Van der Waals interactions for multilayer epitaxial graphene and graphite structures correlate the field effects to the tuning of the interlayer coupling of rotationally stacked epitaxial graphene. The ability to control the electronic properties should extend the range of distinctive physical phenomena and applications for this material system.

Nanoscale, 2012,4, 2962-2967 DOI: 10.1039/C2NR11991A

Oxide Surfaces on Atomic Scale: STM Quest



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Oxide surfaces, especially transition metals oxides surface, offer a rich variety of structures and localized physical phenomena such as superconductivity, metal-insulator transition and chemical catalytic activity. Appling the technique of scanning tunneling microscopy (STM) into oxide surface science, has yielded considerable insight on atomic structure, properties and processes at surfaces. Here, we discuss the understanding of the model systems TiO_2 and $Sr_3Ru_2O_7$ in detail, in order to demonstrate the role of nonstoichiometry in surface structure evolution and the approach to interpreting structure-properties at the atomic level. A combination of STM, local crystallography analysis (LCA), sliding Fast Fourier Transform (SFFT) and theory is applied and the impact of these studies on our understanding of the behavior of oxides is summarized.

Ferroelectric Control of Multiferroic Oxide Interfaces



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Interfaces have emerged as key focal points of current condensed matter science. In complex, correlated oxides, heterointerfaces provide a powerful route to create and manipulate the charge, spin, orbital, and lattice degrees of freedom. The interaction of degrees of freedom at the interface has resulted in a number of exciting discoveries including the observation of a 2-D electron gas-like behavior at LaAlO₃-SrTiO₃ interfaces; the emergence of the ferromagnetism in a superconducting material at a YBa₂Cu₃O_{7-x}-La_{0.7}Ca_{0.3}MnO₃ interface and a ferromagnetic state induced in a BiFeO₃ layer at a heterointerface with La_{0.7}Sr_{0.3}MnO₃. These complex oxide interfaces create a huge playground to discover new emergent phenomena. The key question I would like to address in this presentation: can we use ferroelectricity to modulate the functionalities at complex oxide interfaces? In the beginning, I will take LaAlO₃-SrTiO₃ interface as a model system to illustrate the concept of ferroelectric control by using a combination of cross-sectional scanning tunneling microscopy, X-ray photoelectron spectroscopy, and transport measurements. Then, I will take BiFeO₃/La_{0.7}Sr_{0.3}MnO₃ as an example to demonstrate the design concept of the interfacial multiferroicity based on magnetic measurements and X-ray absorption spectroscopy. The last part of my talk will be focused on addressing the ferroelectric control of interfacial multiferroicity in BiFeO₃/La_{0.7}Sr_{0.3}MnO₃ system. The ferroelectric modulation paves a way to non-volatile control of the fascinating functionalities created at oxide interfaces.



Synthesis and Characterization of Oxide-based Ceramic Phosphors

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Oxide-based ceramic phosphors have been used in TV's, displays, LED's, and as laser sources. Recent work at Vanderbilt by our group has suggested that these materials may also be used as passive radiation exposure indicators. This talk will discuss our work to synthesize several oxide-based phosphors and characterize the materials' chemical, physical, and optical properties.

We use combustion synthesis to produce the oxide-based ceramic phosphors. Combustion synthesis involves heating a mixture of metal nitrates and a fuel until the mixture ignites. If the proper conditions are used the energy released by the combustion is sufficient to form polycrystalline material. Combustion synthesis, in general, is a fairly established technique. However, we have found that the properties of the synthesized materials are strongly dependent on several process variables and conditions. Mixing of the fuel and oxidizers during heating is key to achieving ignition and a producing uniform batch of material. The rate at which the mixture is heated also was found to be critical in achieving ignition, as fuels can begin to decompose before the ignition temperature is reached. Even the size of the crucible used to preform the synthesis can affect the resulting material. I will present results of our studies to understand the affects of process variables on the chemical, physical, and optical properties of combustion synthesized oxide-based ceramics. We used XRD to determine crystallinity and grain size of the oxides. XPS and RBS was used to determine the chemical composition and bonding within the oxides. Photoluminescent spectral intensities and peak positions was used to determine the optical properties of the oxides.

This work is funded by the Defense Threat Reduction Agency under contract number HDTRA 1-10-1-0112.



Searching for the active sites on carbon catalysts

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Although carbon is not well known as a catalyst, activated carbons and "carbon blacks" have been used as catalysts and research has focused on attempts to correlate catalytic activity with porosity, surface area, functional groups and even ash content. The scientific challenge is to achieve a detailed mechanistic understanding of the structural or chemical sites of the carbon that are catalytically active and explore how catalytic reactions proceed on carbon surfaces. Over the past decade, a variety of allotropic forms of carbons have been discovered including fullerenes, nanotubes, nanohorns, mesoporous carbon, carbon nanofibers, onion-like carbon, and nano diamonds. Use of such carbons provides new opportunities for correlating catalytic activity with structure, in carbons with controlled structure and/or functional groups. We have used such synthetic carbons as catalysts for oxidative dehydrogenations (ODH) in an effort to understand which structural sites control activity and selectivity in this reaction. ODH is more typically catalyzed by oxides and involve participation of surface adsorbed oxygen, so work has focused on oxygen functional groups. Starting with graphite oxide a series of catalysts containing variable amounts and types of oxygen were prepared and a series of structural and catalytic characterization were performed. Interestingly, there is poor correlation between oxygen content and catalytic performance. In another approach, a glassy carbon with enclosed fullerene-like cages of 2-3 nm was synthesized through a soft-template approach that created open mesopores of 7 nm. The open mesopores provided access to the fullerene-like cages, which were opened and closed through heat treatments. These graphitized mesoporous carbons (GMC) showed obvious catalytic activities in the ODH reactions when the fullerene-like cavities were open, regardless of the existence of the surface-oxygenated functionalities. The GMC catalysts were deactivated after the fullerene-like cavities were closed. It is concluded that the catalytic activity is related to the openness of the fullerene-like cavities, with open cavities providing access to graphitic planes with open edges. These results seem to suggest that a reexamination is needed of the generally accepted hypothesis that ODH occurs at oxygenated sites.

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.



Correlated functional imaging of energy interconversion in hybrid nanostructures

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Most advanced photovoltaic materials and devices consist of light harvesting units with one or more dimensions smaller than but comparable to the wavelength of visible light, including solar cells based on thin films, dye sensitized nanostructures, and quantum dots. As a consequence, the photonic structure of these materials becomes relevant to understanding their efficiency in harvesting light. In addition, materials that are produced en masse by inexpensive processes tend to have defects that influence their electronic structure, and by extension both the absorption of light and the efficiency with which the absorbed light is converted into electrical energy. We are interested in understanding the influence of geometry, defect structure, and doping on the absorption and utilization of light in nanostructured semiconductors and hybrid materials, including metal nanostructures that absorb light by plasmonic excitations. We have focused on semiconductor nanowires as model systems for measuring, modeling, and understanding lightmatter interactions at this length scale. By engineering the dimensions of the nanowires and nanowire devices, we enable spectrally resolved scanning photocurrent microscopy (SPCM) measurements to investigate energy interconversion in single hybrid nanostructures. Nanoscale resolution is achieved in correlated measurements of physical and chemical structure by transmission electron microscopy and atom probe tomography, respectively. This talk will discuss our recent investigations of plasmonically enhanced photocurrents in silicon nanowire devices and the influence of defect structure and local surface plasmon excitation on absorption and Raman scattering in nanowires.



Optimization of Fully Perpendicular Magnetic Tunnel Junctions by Rapid Thermal Annealing

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We have shown for the first time that magnetometry on the free layer of fully perpendicular magnetic tunnel junctions can be used to optimize the annealing conditions, and therefore the transport properties of these devices. Fully perpendicular Co-based multilayer (ML) CoFeB/MgO/CoFeB magnetic tunnel junctions (MTJ's)(1,2) have been reported by us previously. However, until recently, these types of perpendicular MTJ's have rarely exhibited TMR ratios greater than about 10%. This had been attributed to the inability to pull a sufficiently thick layer of CoFeB perpendicular on top of MgO, as well as the incomplete bcc templating of CoFeB from MgO owing to the adjacent fcc Co/Pd ML's. Recent results(3) on interfacial perpendicular anisotropy have generated great interest i9n thin, Ta-seeded and capped CoFeB/MgO/CoFeB MTJ's. Also of interest are MTJ's with pinned perpendicular synthetic antiferromagnets (SAF), of the form AP1/Ru/AP2 where AP1 and AP2 are Co-based multilayers , for instance, Co/Ni or Co/Pd. We report on fully perpendicular MTJ's with a thin Ta-seeded CoFeB free layer and a Co/Pd ML-based SAF pinned layer(4-7). A thin, amorphous Ta transition layer was utilized to transition from bcc CoFeB to fcc Co/Pd for the top pinned layer(7). Both blanket and patterned devices were rapid thermally annealed(8)at various times and temperatures. We achieved 50% TMR at 300 K (59% at 4.2K) at a RTA temperature of 500 0C for 8 minutes. Longer annealing times were seen to degrade the perpendicular magnetic anisotropy of the free layer, with a corresponding degradation in the TMR of the devices.

This research was supported bya U. S. Department of Defense DARPA-MTO STT-RAM Universal Memory contract through Grandis Inc., Milpitas, CA. Dr. Jane Howe and Dr. Harry Meyer III of ORNL are acknowledged for the TEM and XPS studies of the devices through a CNMS grant and the ORNL SHaRE program. Dr. Robert Shull Of NIST, Gaithersburg, is acknowledged for CIPT measurements.

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Controlling electronic phase competition in confined manganites

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We will discuss recent work on tuning the complex interactions that govern correlated electronic systems. Specifically, we will highlight our efforts to isolate single order parameters in the spin-charge-orbital-lattice hierarchy in order to further understand how these complex interactions govern mesoscopic phase competition. We have developed new experimental methods that allow for local spin, strain and charge engineering to be tested on these materials; which has allowed us a glimpse at the interplay driving emergent phenomena. This talk will focus on results that demonstrate the possibility of exerting a measure of control over the electronic phase competition leading to colossal magnetoresistance and the metal-insulator transition in manganites. These studies offer new means to quantitatively investigate the balanced energetics that drive complex materials and promise an ability to tune critical temperatures and desired electronic/magnetic properties.



Tunneling microscopy of molecular and thermoelectric surfaces

Petro Maksymovych Center for Nanophase Materials Sciences, Oak Ridge National Laboratory

The overwhelming majority of all experimental approaches to molecule-on-surface selfassembly have so far relied on thermal fluctuations to drive the tethering reactions of the precursor molecule and subsequent self-assembly of the adsorbed molecules. The most notable example is the attachment of alkanethiol molecules to a gold surface, which has become the quintessential workhorse of nanoscale science and the basis of numerous studies of molecular self-assembly, molecular electronics, and organic monolayers tethered to the surface. Yet thermally driven reactions do not allow for a rigorous control of the self-assembly process, and preclude simple approaches to patterning and nanostructuring. Moreover, a large number of potentially interesting anchor groups may not possess a thermally-activated pathway to selfassembly, particularly for the formation of the all-important three-dimensional monolayer. One notorious example is the alkyne triple bond, which over the years has proven to be next to impossible to immobilize on gold. We will discuss our recent discovery of an all-electron control over the tethering, self-assembly and dis-assembly reactions which is uniquely enabled by the ability to inject hot carriers in scanning tunneling microscopy [1]. This approach allows for facile self-assembly, defect manipulation, and disassembly of densely packed upright molecules anchored via an alkyne derivative at temperatures as low as 90 K. From a comprehensive theoretical analysis we have found that the anchor group exhibits an enticing narrowing of the HOMO-LUMO gap upon adsorption on gold, which partially explains the propensity of phenylvinylidene toward hot-hole disassembly reaction, and poses intriguing perspectives for subsequent studies of electron transport through vinylidene-functionalized molecules.

At the same time, molecular materials, in the form of polymers and single molecules, attain a growing interest from a thermoelectric community. Yet, the intrinsic heterogeneity of organic matter and the fragility of the lattice poses great challenges to conventional thermoelectric characterization. Scanning probes offer several potential solutions, and we will discuss our recent effort in the development of non-contact techniques for thermopower measurements. By contrasting thermoelectronic response of silver [2] and bismuth telluride surfaces, we will outline the possible pathways for self-consistency in such measurements and the possibility to bridge the gap between local observables and thermoelectronic response in the bulk.

This research was carried out at the Center for Nanophase Materials Sciences, sponsored by the Division of User Facilities at the Oak Ridge National Laboratory. Research on thermoelectric properties was sponsored by Laboratory Directed Research and Development Program of Oak Ridge National Laboratory, managed by UT-Battelle, LLC, for the U. S. Department of Energy.

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POSTER PRESENTATIONS

Poster

- 1. R. Sachan, *ORNL/University of Tennessee* Surface Plasmon response of self-organized bi-metallic plasmonic nanoparticles
- 2. Rebecca Agapov, University of Akron Protected TERS Probes for the Study of Polymer Surfaces
- **3. Mario Caruso,** *University of Rome* Directed selfassemblage of soft matter nanostrucres on inorganic surfaces
- **4.** Aubrie Pfirman, *Clemson University* Stabilization of 2D arrays of Silver Nanoparticles via PMMA
- **5.** Michael Wleklinski, *Purdue University In Situ* Analysis of Surfaces Prepared by Ion Soft Landing
- **6.** Robbyn Trappen, *West Virginia University* Characterization of Surfaces and Interfaces Utilizing Nonlinear Optics and Synchrotron Radiation
- 7. Rylan Bledsoe, *University of Tennessee* Analysis of Nanostructured Bismuth Telluride using TEM/STEM
- 8. Jiangchao Chen, University of South Dakota Experimental and Computational Photodissociation Pathway of Lanthanide Cyclopentadienyl-type Precursors for Laser Assisted Metal-organic Chemical Vapor Deposition
- **9. Heayoung Yoon,** *NIST/University of Maryland* High-resolution photocurrent imaging in Cadmium Telluride solar cells using electron beams and optical excitations
- **10. Ye-Che Hsiao**, *ORNL/University of Tennessee* Dielectric Film Effects on Photovoltaic Processes in Organic Solar Cells
- **11. Tanya David**, *Norfolk State University* End Functional Conjugated Polymers for Potential Self Assembly and Optoelectronic Applications
- **12. Jing Li**, *Wayne State University* Seperation and Characterization of Peptides and Synthetic Polymers by Matrix Assisted Ionization Vacuum (MAIV)-Ion Mobility Spectrometry (IMS)- Mass Spectrometry (MS)
- **13. Laine Radell**, *George Washington University* Real-Time Analysis of Metabolic Response in Electrically Stimulated Cells and Cell Assemblies by LAESI Mass Spectrometry
- **14. Yonathan Merid**, *Louisiana State University* Multi-layer Sublimation Coating for MALDI Imaging
- **15.** Sylwia Stopka, *George Washington University* Subcellular Analysis of Cell Organelles with Micro-dissection on Nanophotonic Desorption Ionization Platform
- **16. Yuanming Liu**, *University of Washington* Biological Ferroelectricity and Energy Conversion in Aortic Walls
- **17. Soonil Lee**, *Pennsylvania State University* Interrelationship of Dipole-Electronic Conduction-Defect Heterogeneity in Conductive Ferroelectric-Thermoelectrics



18. Omur Dageviren, Yale University Effects of Tip Structure on Three-Dimensional Force Fields Recorded by Scanning Probe Microscopy **19. Roel Flores,** University of Tennessee Probing the Intrinsic Various Single Walled Carbon Nanotube Structures 20. Armando Perez, University of New Orleans Polar Ordering inIndividual BaTiO₃ Colloidal Nanocrystals and Its Control via Colloidal Processing 21. Sandra Doria, University of Rome Electrochemical nanowriting on CeO2 thin films 22. Nataly Chen, University of Washington Delineated Local Electromigration for Nanoscale Probing in Lithium Ion Intercalation by Electrochemical Strain Microscopy 23. Luis Zaldivar, University of New Orleans Probing the Local Strain-Mediated Megnetoelectric Coupling Multiferroic Nanocomposites by Magnetic Field-Assisted Contact Electostatic Force Microscopy 24. Jackie Bracco, Wright State University Probing mineral growth kinetics using in situ AFM: the effect of impurities on growth rates and surface morphology 25. Medhi Yazdanpanah, NaugaNeedles Specialized Probes for AFM-SECM and Conductive AFM Under Liquid **26. Jasmine Hargrove,** Clark Atlanta University Band Gap Opening in Methane Intercalated Graphene **27. Billy Clark,** University of Alabama Optimization of Perpendicular Magnetic Tunnel Junctions with Various Annealing Conditions 28. Nicola Pellicciotta, NAST Center

Ab-initio calculations and molecular dynamics simulations of peptide adsorption onto metal oxide surfaces

29. Alejandro Lopez Bezanilla, *Oak Ridge National Laboratory*





Nanoscale Imaging for Energy Applications VENDOR LOCATIONS

Asylum InstrumentsRoom L182
Bruker NanoRoom LG82
Bruker DaltonicsRoom LG82
NanosurfRoomLG82
NanonicsRoomL182
Thermo ScientificRoom L182
SPECS Room L282
RHKRoom L282

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