Materials Electron Microscopy At the Limit


Wednesday, August 26th 2009
9.00 AM – 4.30 PM at the UIC Forum.
Program:

9.00 – 9.15  Welcome and Introduction
9.15 – 9.45  I. Arslan (UC Davis - MSA Touring Speaker)
             High Resolution 3-D Characterization of Nanomaterials using Tilt Tomography in the Scanning Transmission Electron Microscope
9.45 – 10.15 N. Zaluzec (Argonne)
            Pushing the Limits of AEM: Where are we now and where are we going?
10.15 – 10.45 W. Walkosz (University of Illinois at Chicago)
            Atomic Scale Characterization and First-Principles Study of alpha and beta phases of Si$_3$N$_4$

10.45 – 11.00 Coffee Break

11.00 – 11.30 E.A. Stach (Purdue University)
              Using in-situ TEM to understand growth termination of water-assisted single-walled carbon nanotube arrays
11.30 – 12.00 Y. Ito (Northern Illinois University & Argonne)
              Electron Magnetic Dichroism

12.00 – 1.30  Outside Lunch (see last page for options)

1.30 – 2.00  P. Voyles (University of Wisconsin Madison)
             Fluctuation Microscopy: Nanoscale Order in Amorphous Materials from Electron Nanodiffraction
2.00 – 2.30  J.M. Zuo (University of Illinois – Urbana Champaign)
             Atomic Resolution Characterization of Nanoscale Interfaces and Surfaces
2.30 – 3.00  M. Tanase (University of Illinois at Chicago)
             Magnetization Behavior In Patterned Thin Film Heterostructures

3.00 – 3.15  Coffee

3.15 – 3.45  A. Petford-Long (Argonne)
             Structure property relationships in nanoscale magnetic heterostructures
3.45 – 4.15  R.F. Klie (University of Illinois at Chicago)
             Aberration-corrected STEM analysis at UIC

4.15 – 4.30  Concluding remarks / Tour RRC-East
Nanotechnology has become a key component in the field of materials science. Rather than analyzing and determining the properties of bulk single or poly-crystals where the third dimension is assumed to be uniform, we must now analyze materials that have a finite size and shape in three dimensions, and not necessarily uniform in any of the directions. This new demand on materials characterization has led to the development of electron tomography for inorganic materials using Z-contrast imaging in the scanning transmission electron microscope (STEM). This technique involves taking a series of images of the sample at different tilt angles, normally ranging between -70° to +70° every 1 to 2 degrees, and using these two dimensional images to reconstruct a three dimensional volume of the sample. This tilt range may increase depending on the sample geometry and the holder used, but we are constantly battling against an artifact in the reconstruction called “the missing wedge.” This effect may be reduced greatly by performing dual axis tomography, or overcome completely using new holder technologies, but each technique has its pros and cons. These benefits and limitations will be discussed through examples of different inorganic materials.
Pushing the Limits of AEM: Where are we now and where are we going?

Nestor J. Zaluzec
Electron Microscopy Center, Argonne National Laboratory

The limits of Analytical Electron Microscopy are frequently discussed in terms of minimum detectable mass or mass fraction. While these are reasonable parameters to compare techniques it does not always convey the limitations or capabilities of a technique. In this presentation we will consider these traditional measures of sensitivity and then consider novel exploitations of electron column based x-ray and electron spectroscopy to extend the applications of microanalysis to the regime of problems which are now accessible with the new generation of aberration corrected microscopes.
Atomic Scale Characterization and First-Principles Study of alpha and beta phases of Si$_3$N$_4$

Weronika Walkosz
Nanoscale Physics Group, Department of Physics, University of Illinois at Chicago

Understanding the effects of dopants in multifunctional materials has received a great attention in the search for improved control over their properties and behavior in various applications. In silicon nitride (Si$_3$N$_4$) ceramics, additives of metallic compounds used to promote densification of the powder compacts during sintering, play an important role in the microstructural evolution, phase transformations, and the mechanical properties of the ceramic, such as its toughness. Studies aimed at achieving a microscopic description of the atomic arrangement and local bonding of the additives in Si$_3$N$_4$ are, therefore, of both fundamental and technological interests.

In this talk, I will present the results from a combined atomic-resolution Z-contrast imaging, electron energy-loss spectroscopy (EELS), and first principles studies of $\alpha$ and $\beta$-Si$_3$N$_4$ in the presence of (i) CeO$_{2-x}$ and (ii) SiO$_2$ compounds forming intergranular films (IGFs) in the ceramic. In particular, I will discuss the role of interstitial oxygen in stabilizing bulk $\alpha$-Si$_3$N$_4$ and its presence at the grain boundaries of the $\beta$-polymorph. In the case of $\beta$-Si$_3$N$_4$/CeO$_{2-x}$ sample, I will show that the arrangement of Ce atoms at the interface with Si$_3$N$_4$ is semi-crystalline, the details of which depend on the sintering techniques and additives used, as well as the thickness of the IGF. Using aberration-corrected Z-contrast imaging of the $\beta$-Si$_3$N$_4$/SiO$_2$ interface, I will argue the presence of atomic columns completing Si$_3$N$_4$ open rings, which have not been observed experimentally at Si$_3$N$_4$/REO interfaces but have been predicted theoretically on bare Si$_3$N$_4$ surfaces. Lastly, I will show the results of my EELS study at the $\beta$-Si$_3$N$_4$/SiO$_2$ indicating that O could be replacing N at the interface between the two materials in agreement with predictions from my first principles calculations. The observed structural and electronic variations in the atomic bonding of the dopant elements in Si$_3$N$_4$ will be briefly discussed along with the experimental and theoretical methods chosen to study this ceramic material.
Using in-situ TEM to understand growth termination of water-assisted single-walled carbon nanotube arrays

Eric Stach
Department of Materials Engineering, Purdue University

Following Hata’s pioneering studies of water-assisted “supergrowth”, vertically aligned single-walled carbon nanotube (SWNT) carpets have received enormous attention, as they have a large number of potential applications. However, a lack of understanding of the mechanisms of growth termination remains a limiting factor in optimizing these structures for widespread usage. In order to further understand the growth termination phenomena, we have investigated the evolution of catalyst morphology using both standard and in-situ transmission electron microscopy techniques. A series of tightly controlled annealing and growth studies were performed in a dedicated chemical vapor deposition using three different gas ambients (hydrogen, hydrogen and water, and hydrogen, water, and atomic hydrogen). Quantitative characterization of the catalyst sizes as a function of annealing time leads to an explanation for growth termination in terms of not only Ostwald ripening, but also in terms of catalyst diffusion into the substrate. These results are corroborated by real time observations of nanotube growth and growth termination in a dedicated environmental cell transmission electron microscope, where both ripening and substrate diffusion are directly observed. Strategies and preliminary results concerning the creation of an ‘immortal’ catalyst / support system for aligned CNT growth will be presented.
Electron Magnetic Dichroism

Yasuo Ito$^{1,2}$, Nestor J. Zaluzec$^2$

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Although interfacial and thin film magnetism as well as size effects have been already studied by means of microscopic and macroscopic analysis methods, to date, no technique is available to characterize individual magnetic moments on the nanometer scale or less. In this talk, two magnetic dichroic measurement techniques, Electron Magnetic Linear Dichroism (EMLD), and Electron Magnetic Circular Dichroism (EMCD), both of which are achieve by precise and detailed measurements of inelastic scattering from localized volumes in the materials obtained by employing high-energy resolution electron energy loss spectroscopy (EELS) will be reviewed, including a study of the temperature and angular dependence of EMLD on hematite with a superior angular resolution and consistency obtained by the high angular resolution electron channeling electron spectroscopy (HARECES) technique, and a recent demonstration of quantitative determination of the ratio of magnetic orbital to spin-moment using EMCD.
Electron nanodiffraction in a STEM is a powerful tool for nanometer-scale structural characterization of materials, from phase identification of small regions to solving the 3D structural of nanoparticles in atomic detail. I will discuss the application of systematic electron nanodiffraction to amorphous materials in a technique called fluctuation electron microscopy (FEM). We have used FEM to show that nanoscale order controls crystallization in high Al-content alloys and to begin to connect the structure of Zr-based bulk amorphous metals to their plastic deformation as measured by nanoindentation creep. I will discuss a new way to analyze FEM data using reverse Monte Carlo simulations, and I will close with a brief discussion of the first results from our new aberration-corrected Titan STEM. This instrument has the potential to create coherent electron probes for nanodiffraction including FEM covering three orders of magnitude in probe diameter, from 0.1 to 100 nm.
Atomic Resolution Characterization of Nanoscale Interfaces and Surfaces

Jian-Min Zuo
Department of Materials Science and Engineering, University of Illinois Urbana-Champaign

Nanoscience and nanotechnology is about controlling, manipulating and interfacing different structures of few to hundreds nanometers in size. The properties of these nanostructures are heavily influenced by their interfaces, including their surfaces. Characterization of nanoscale interfaces is still a challenge despite tremendous progress that has been made on nanoscale characterization techniques. We use a combination of aberration-corrected scanning transmission electron microscopy (STEM) and nanoarea electron diffraction (NED) for structure determination of individual nanostructures and EELS for electronic structure characterization. In STEM, atomic columns are imaged directly for heavy atoms. For electron diffraction, a nanometer-sized, coherent, parallel electron beam is used to record diffraction patterns. This talk will report what we have learnt from electron diffraction and imaging about the surface and interface of Au nanocrystals and interfaces in oxide superlattices. In each case, we will show how the electron probe is used to extract interfacial properties. The work is supported by DOE BES and NSF DMR.
Magnetization Behavior In Patterned Thin Film Heterostructures

Mihaela Tanase
Nanoscale Physics Group, Department of Physics, University of Illinois at Chicago

Magnetization processes in patterned magnetic heterostructures are of fundamental scientific interest and have important applications in information storage such as in permanent magnetic random access memories (MRAM). Continuous miniaturization causes material defects to play an increasingly important role in the switching behavior of these devices. Transmission electron microscopy (TEM) is the technique of choice for high spatial resolution characterization of non-ideal magnetic behavior and its relationship with nanoscale structure. In combination with novel phase retrieval techniques, Lorentz TEM has the potential of becoming an in situ quantitative technique for mapping magnetization reversal processes.

We have used a combination of Lorentz TEM, magneto-optical Kerr magnetometry and micromagnetic simulations to characterize the behavior of micron-size exchange biased magnetic nanostructures exhibiting vortex magnetization and imprinted with circular exchange bias. Circular exchange bias promotes a reversible vortex behavior and it controls the chirality of the vortex during reversal. It also stabilizes the vortex structure as a low energy state, acting against magnetocrystalline anisotropy which favors the formation of domain walls. Exchange bias suppresses stochastic processes due to thermal activation and cause the magnetization reversal to be reproducible over time, an important feature in applications.

Phase imaging based on the Transport–of-Intensity Equation (TIE) is emerging as a novel method for mapping magnetization phenomena in situ in the TEM. The phase shift of the electrons containing the magnetic information is obtained from the intensity of the wave and its derivative along the optical axis alone, and does not require a reference beam as conventional interferometry techniques do. Examples of application of TIE to patterned magnetic heterostructures will be given and requirements for becoming a quantitative technique as well as its limitations will be described. TIE-based phase retrieval has potential applications in novel systems such as multilayered magnetic and multiferroic heterostructures for data storage and logic applications. Furthermore, TIE is not limited to magnetic systems, as it offers opportunities in mapping electric fields and charge transport processes at the nanoscale.
Structure property relationships in nanoscale magnetic heterostructures

Amanda Petford-Long
Materials Science Division, Argonne National Laboratory

The properties of nanoscale magnetic materials depend critically on their microstructure and composition, with variations on the atomic scale leading to variations in properties. Of particular interest for technological applications in information storage systems are magnetic structures composed of thin layers, such as spin tunnel junctions. In such devices the microstructure and chemical profile across the layers are critical in determining the magnetic and transport properties, and therefore need to be critically controlled. However, these data are really only of interest in so far as they enable us to understand the origins of the magnetic and transport properties, and we have been using in-situ TEM to investigate these properties. We have used a combination of Lorentz TEM and in-situ magnetizing experiments to study the micromagnetic behavior at the sub-micron scale of magnetic nanostructures such as patterned exchange-biased magnetic disks. Quantitative analysis of the Lorentz TEM data has been carried out using the transport of intensity equation (TIE) approach. We have also developed in situ TEM capabilities that enable us to correlate the local tunneling properties of magnetic tunnel junctions with microstructure, and results of these studies will also be presented.
Atomic-Scale Studies of Complex Oxide Interfaces Using Aberration-Corrected Z-contrast Imaging and EELS

Robert F. Klie
Nanoscale Physics Group, Department of Physics, University of Illinois at Chicago

Interfaces in complex oxide materials have been an enduring theme in materials physics, where the interplay of the reduced dimensionality, proximity effects, and surface relaxation and reconstruction creates interfacial states that are distinct from their bulk counterparts. It has been recognized that perovskite oxides provide a unique opportunity to bring materials with diverse and mutually exclusive properties into intimate contact, and create interfaces with excellent structural and chemical compatibility and potentially novel properties.

In this presentation, I will summarize the aberration-corrected Z-contrast imaging and EELS studies conducted in my group at UIC, and how the combination with in-situ heating/cooling experiments can help solving fundamental problems associated with interfaces in perovskite-type oxides. More specifically, I will discuss our recent results, including the effects of the Co3+-ion spin-state transition and interfacial ferromagnetism in LaCoO3, the effects of charge transfer in the layered thermoelectrics, such as Ca3Co4O9, and the effects of promoter elements in nanoscale core-shell metal-clusters used in heterogeneous catalysis. All these experiments were performed using the FEI TEAM0.5 and VG HB501 at Lawrence Berkeley National Laboratory, the VG HB603U at Oak Ridge National Laboratory, and the UIC JEOL2010F.
LUNCH OPTIONS

The map shows the location of cafes & restaurants in the new University Village neighborhood south of the UIC Forum which are open at lunchtimes. Most are on Maxwell St between Halsted & Union.

If you are interested in trying the Original Maxwell St Polish Sausage Sandwich this is served at Jim’s (3) which was originally located at Maxwell & Halsted from 1939 to 2001. The two hot dog stands are takeaway only.

Key
1. Massa Italian Café
2. Francanello Italian Restaurant
3. Jim’s Polish Hot Dog Stand (The original Maxwell St one)
4. Express Grill Polish Hot Dog Stand
5. Jamba Juice
6. Kohan Sushi & Japanese
7. Caribou Café Coffee, Bakery
8. Morgan’s Bar & Grill American Traditional
9. Lalo’s Mexican Restaurant
10. Hash Brown Café All day breakfast & sandwiches
11. Perry’s Deli sandwiches
12. WOW Café & Wingery New Orleans inspired sandwiches
13. Quizno’s
14. Joy Yee Noodle Pan Asian Cuisine