Workshop A: Frontiers in Imaging & Spectroscopy
Workshop held on May 3, 2011
Organizers: Jeffrey Guest (CNM), Gayle Woloschak (APS, Northwestern University), Ilke Arslan (EMC, University of CA at Davis)

Central to many of the fundamental research and engineering challenges of our time is the ability to understand, modify and control systems in a variety of environments on ultrasmall length scales and - in some cases - fast time scales. Success hinges not only on the ability to image structure in nanoscale systems, but also correlation of the associated electronic, magnetic, optical and chemical properties and behavior. The APS, CNM and EMC possess a diverse and complementary array of imaging and spectroscopy microscopy platforms to push this frontier. Going forward, it will become more important to establish multimodal imaging capabilities and novel tool combinations to address this next generation of research challenges. The goal of this workshop was to bring scientists together who are at the technical cutting edge and the scientific forefront to share not only the reach of their respective capabilities, but also their grand challenges addressed and obstacles encountered. The hope of the organizers was for sharing of knowledge, making connections, and cross-pollination of experimental approaches and scientific problems. Overwhelmingly positive feedback was received in the end. The most satisfying comments came from participants who said, referring to the speakers from other fields, that the work was “amazing” and that they were not even aware that such measurements were possible until this event took place.

The workshop was opened by keynote speaker Nestor Zaluzec (University of Illinois at Chicago, ANL), who has decades of experience in the field of microscopy. He reviewed the history of electron microscopy and introduced the types of information scanning and transmission electron microscopy can provide. Nestor showed examples from biological and materials science, and demonstrated the state of the art energy dispersive spectroscopy (EDS) with very high signal to noise ratios that he developed at Argonne. He also discussed the scientific motivation for performing electron microscopy in a controlled atmosphere - rather than in vacuum - and recent efforts to develop this new capability.

Jorg Maser (APS, CNM) described the current status and future plans for hard x-ray probes at the Advanced Photon Source. By providing extremely high spatial resolution for x-ray imaging, diffraction, and fluorescence probes, these capabilities are leading to new discoveries in nanoelectronic devices, energy materials, and environmental systems. Next generation hard x-ray optics (multi-layer Laue lenses) are being explored to bring spatial resolution below 10 nm. Jorg discussed the current science investigated at the CNM/APS hard x-ray nanoprobe and the progress in the development of a second beamline dedicated to biological and health science applications. It was noted that high throughput approaches remain a challenge, however. Jorg also discussed efforts to combine scanning tunneling microscopy with x-ray illumination in order to provide chemical sensitivity at atomic resolution.

Two speakers then focused on science using scanning probe techniques. Weida Wu (Rutgers University), a frequent user of CNM’s scanning probe microscopes, discussed his recent work on imaging multifunctional domains and domain walls by *in situ* scanning probe microscopy

(SPM). Atomic force microscopy (AFM) is sensitive to not only the structure of surfaces, but also magnetic, electronic, and piezoelectric properties. This makes the technique ideal for exploring multiferroic materials. Weida used state-of-the-art SPM techniques with *in situ* high magnetic/electric fields or multimodal capabilities to explore aspects of multiferroic materials. For example, in one multiferroic system he used conductive AFM to directly image domain walls, piezo force microscopy to measure the polarization of individual domains, and electro-force microscopy to map the local Fermi level of the domains. He also used polarized electron microscopy to cross correlate these surface measurements with buried domain properties. Weida then gave an overview of the CNM UHV SPM capabilities which generated follow-up inquires to CNM staff.

Pegor Aynajian (Princeton University) discussed experiments on heavy fermionic materials with ultrahigh vacuum scanning tunneling microscopy and spectroscopy (UHV STM). Collective electronic phenomena have been discovered in materials with partially filled *f*-orbitals (where the excitations act as heavy fermions). At low temperatures, hybridized many-body states form exotic quantum phases such as unconventional superconductivity and the "hidden order" phase in URu_2Si_2. A microscopic understanding of the mechanism behind these phase changes does not currently exist. By using the atomic-scale imaging and spectroscopy available with UHV STM, he explored the spatial and energetic structure of the electronic states in $CeCoIn_5$ and URu_2Si_2. Pegor argued that, even though STM is a surface sensitive technique, he could extrapolate his observations to bulk properties because the temperature dependence matched that of bulk measurements. He also discussed environmental requirements for their measurements, including extremely low drift instruments and acoustically isolated rooms.

Two speakers discussed applications of x-ray fluorescence microscopy in mammalian, marine and microbial systems regarding studies of environmental contaminants, mechanisms of drug resistance, and basic biological pathways such as the role of metals in brain cell development. James Penner-Hahn (Univ. Michigan) discussed yeast and mammalian systems with both toxic and therapeutic applications. This work may impact studies of Cd toxicity in humans as well. Studies of Zn distribution in malarial parasites also were presented. Derk Joester (Northwestern University) examined metal sequestration in microbes, particularly radionuclides that might be important contaminants of the environment. The organisms are extremely resistant to the effects of radiation and thus could be used for clean-up of nuclear waste sites. Speakers noted the importance of improving resolution, preservation, and throughput at synchrotron beamlines.

Steve Pennycook discussed research on energy materials high resolution microscopes at ORNL, including transmission electron microscopy (TEM). He showed atomic resolution imaging of yttria-stabilized zirconia (YSZ) in YSZ/strontium titanate epitaxial heterostructures. He also presented images of LiFePO4 where they were able to resolve Li columns. The atomic and electronic structure measurements in the microscope, coupled with theoretical calculations, provided an understanding of the fundamental properties of those materials that can lead to better device nanofabrication.

Nigel Browning closed the workshop with novel research on thin films and nanomaterials using dynamic TEM (DTEM). This new technology allows for high temporal resolution studies of dynamic processes in materials, specifically 15 ns temporal resolution with a concurrent spatial resolution of 5 nm. While DTEM is novel in itself, he further focused on its combination with in-situ methods, such as gas and liquid stages. With a liquid stage they were able to observe growth of PbS nanoparticles in solution. In a gas environment, they observed growth of catalyst particles and nanowires using a drive laser. The speakers all discussed the need to correlate images with chemical information (EDS or EELS), and also stressed that these advanced techniques should be coupled with in-situ stages to understand phenomena in real time.