



Our User Science Shapes the Future

2012 Users Meeting
Advanced Photon Source
Center for Nanoscale Materials
Electron Microscopy Center

Thematic Workshop Summary: Imaging at All Length and Time Scales

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The ability to understand, modify, and control systems in a variety of environments across multiple time and length scales is central to many fundamental contemporary research and engineering challenges. Success in these efforts hinges on the ability to image not only the structure in systems, but also the electronic, magnetic, optical and chemical properties and behavior associated with this structure. The APS, CNM and EMC possess a diverse and complementary array of imaging, microscopy and spectroscopy platforms to push this frontier of multiscale and time-resolved investigation. Going forward, it will become more important to establish multimodal imaging capabilities focusing on new techniques and the use of novel tool combinations to address the next generation of research challenges. This workshop featured a collection of eight invited presentations by pioneers in their respective fields.

Sheng-Nian Luo from Los Alamos National Laboratory presented the first results of bulk-scale gas gun shock experiments with dynamic x-ray Phase Contrast Imaging (PCI) and diffraction measurements at the beamline 32ID-B. The highly transient nature of shock loading and pronounced microstructure effects on dynamic materials response call for in situ, real time, in-depth, temporally and spatially resolved, x-ray-based diagnostics. He first showed the x-ray beam characteristics, experimental setup, x-ray diagnostics, and static and dynamic test results, with unprecedented temporal (<100 ps) and spatial (2 μm) resolutions. The results not only substantiate the potential of synchrotron-based experiments for addressing a variety of shock physics problems, but also allow us to identify the technical challenges related to image detection, x-ray source and dynamic loading.

Wilson Chiu from the University of Connecticut talked about the progress, challenges and opportunities of in situ hard x-ray microscopy of fuel cells. Substantial losses arise in the solid oxide fuel cell (SOFC) due to degradation during operation. His presentation focused on the development of in situ x-ray imaging and spectroscopy techniques that will allow the direct observation of the SOFC under operational conditions and provide a scientific and engineering understanding of degradation. Progress was presented on x-ray fluorescence spectroscopy mapping of sulfur-treated SOFC Ni-YSZ anodes using the hard x-ray nanoprobe at 2-ID-D and the in situ x-ray imaging of 3D microstructural and chemical changes in SOFC materials using the transmission x-ray microscope at 32-ID-C. Challenges and opportunities of these techniques were discussed. The long-term goal of this work is to develop hard x-ray in situ techniques to further the understanding and enhance SOFC long term performance and reliability to enable SOFCs as a viable technology for efficient and sustainable energy conversion.

Xianghui Xiao from the X-ray Science Division of Argonne presented some of the pioneering work being done 2-BM on fast x-ray micro-tomography and micro-imaging, which also serves as a test bed for some of the wide-field imaging long beamline (APS-U) planned activities. He presented a few examples of

dynamic tomography of corrosion in aluminum, and in situ morphological structure evaluation of an amorphous anode in a Li-ion battery.

Peter Sutter of Brookhaven National Laboratory discussed the growth and synthesis of graphene on various transition metal substrates. These systems were characterized with both low-energy electron microscopy (LEEM) and scanning tunneling microscopy (STM). The LEEM analysis is quite unique as it allows for the real-time imaging of graphene growth. Peter also showed very exciting unpublished research that incorporates the growth of hexagonal boron nitride with the growth of graphene on a single surface. Phillip First of the Georgia Institute of Technology focused on graphene physics and utilized epitaxially grown graphene on SiC(0001). A highlight was the experimental observation of Landau Levels in strained graphene that correspond to a magnetic field on the order of hundreds of Tesla. Phil's research is at the cutting edge of graphene physics and most recently has focused on the effects of external magnetic fields on this amazing two-dimensional material.

Three speakers then covered recent advances in the field of transmission electron microscopy (TEM). Improvements in electron optics, coupled with better detectors and computer control have resulted in impressive gains in the areas of spatial, temporal, and chemical resolution. Paul Kotula from Sandia National Laboratory described his work on atomic resolution chemical characterization; through the use of brighter sources, spherical aberration correction, and efficient detectors with a much larger solid angle than previously possible, the collection efficiency has increased by at least an order of magnitude over what used to be the norm in analytical TEM. He showed examples acquired on their new Titan G2 80-200 microscope; inc. the low end of the resolution scale (CMOS spectral images); the medium end (electrical contacts with spinodal decomposition as well as Pd-Rh hydrogen isotope storage materials); and the high end with atomic resolution x-ray analysis in perovskite and pyrochlore. He concluded by saying that quantification is now being pushed in the 1000 ppm range with sufficient counting statistics; current efforts are underway to combine the spectral techniques with tomographic analysis.

Geoff Campbell from Lawrence Livermore Laboratory covered the temporal aspect and discussed how the study of complex dynamics at the nanoscale is approached by combining an older model TEM with high end lasers, one to drive the sample into an excited state, the other, with a variable delay, to eject electrons from the cathode in 15 ns pulses. He provided an update on the status of dynamic TEM (DTEM) as well as examples of pulsed laser annealing of amorphous NiTi films and GeTe; amorphous-to-solid transitions in Al 7at% Cu thin film, which revealed an interesting transition between two columnar growth modes; and a study of transition zones in a-Ge. Current developments focus on achieving the ability to record dynamic movies by sequential sector-by-sector exposure of a large CCD camera.

Christian Kisielowski from Berkeley National Laboratory concluded the workshop by answering the question: Where are we going with TEM? He provided several example areas of interest, such as imaging of single molecules that are undergoing catalytic reactions; single atom imaging by combined Cs+Cc correction; low atomic number atomic imaging; and adsorbents of graphene. In STEM mode, the depth resolution becomes an issue due to dechanneling of the beam, but this can be used to provide depth sectioning. He discussed the drive towards quantitative measurements, which in principle allow for inverse reconstructions (beyond the reconstruction of the wave function). Atomic resolution at low accelerating voltage (20kV) has recently been achieved. Dr. Kisielowski emphasized the importance of understanding the interactions between the electron beam and the sample.