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Magnetic Materials Probed with Polarized X-ray Spectroscopies

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Highlights from Workshop on Early Science at NSLS-II

The First Experiments Workshop for NSLS-II drew more than 260 participants.

Exciting opportunities for early science at the National Synchrotron Light Source II (NSLS-II) drew over 260 participants to a workshop at Brookhaven National Laboratory, August 12–13, 2013. NSLS users planning to continue their work at the new facility joined prospective NSLS-II users to hear talks and debate possible research projects, beginning the process of creating a pool of experiment proposals and forming research teams.

The experiments chosen, which will run on an initial suite of seven beamlines (out of about 58 beamlines when the facility is fully built), will aid in the NSLS-II science commissioning phase, expected to begin in October 2014. Each breakout session featured several presentations, yielding discussions on which experiments promise to have the greatest science impact and take best advantage of NSLS-II's world-leading capabilities.

After welcoming remarks from Brookhaven Lab Director Doon Gibbs and an introduction from Brookhaven's Associate Laboratory Director for Photon Sciences Steve Dierker, who briefed attendees on the NSLS-II construction status and early-operations timeline, a series of science talks delved into the "grand challenge" problems in science that NSLS-II will help to address: Sunil Sinha, University of California, San Diego, "Complexity and Dynamics in Condensed Matter Systems"; Gayle Woloschak, Northwestern University, "Nanoparticles in Functional Biological Systems"; and John Sarrao, Los Alamos National Laboratory, "Materials Co-design at the Mesoscale: Opportunities for NSLS-II." They were followed by Paul Zschack, Deputy Director of the Photon Division within the Photon Sciences Directorate, who discussed the capabilities of the initial suite of NSLS-II beamlines.

The essence of the workshop was found in the six breakout sessions on day two, where the most important conversations took place. The sessions represented six of the beamlines: Hard X-ray Nanoprobe (HXN), Submicron Resolution X-ray (SRX) spectroscopy, Coherent Hard X-ray (CHX) scattering, Coherent Soft X-ray scattering branch beamlines 1 and 2 (CSX-1 and CSX-2), and X-ray Powder Diffraction (XPD). A separate session for the Inelastic X-ray Scattering (IXS) beamline was held at BNL on October 1.

The topics covered were wide-ranging, from fundamental physics ideas on materials complexity and dynamics to basic needs for conducting advanced studies of functional materials systems, such as fuel cells and energy-storage schemes. Some specific science themes emerged, however, such as "mesoscale" science, which examines the properties and phenomena that occur at the border between the quantum world of assemblies of single atoms and the complex world of functional systems. This is a new frontier for research at NSLS-II.

The breakout session discussions were also a starting point for the formation of research teams that would best achieve the research goals at NSLS-II. Special attention was paid to representation in the areas of theory and analysis, ancillary measurements, and specimen environment development.

All of the breakout sessions were well attended and yielded some excellent dialogue. For example, at the HXN session, 50 registered participants took in presentations by 14 invited speakers. Due to the interdisciplinary nature of X-ray microscopy, a wide range of scientific fields was represented, including materials science, condensed matter physics, high-pressure science, nanoelectronics, diffraction theory/techniques, and X-ray diffraction microscopy techniques.

HXN beamline scientists Yong Chu and Hanfei Yan explained HXN's planned scientific capabilities, which include high spatial resolution (10 nm or better); the ability to take parallel measurements of two or more techniques; in-situ sample environments (temperature, pressure, and electric or magnetic fields); and excellent stability, which allows routine applications of ptychography and other advanced X-ray microscopy techniques. Many of these capabilities were repeatedly cited during the presentations and the follow-up discussions, which yielded several possible directions for first experiments.

Frontiers in X-ray and Electron Imaging

More than 100 attendees participated in the workshop on "Frontiers of X-ray and Electron Imaging."

The following is a summary of the "Frontiers of X-ray and Electron Imaging" workshop held at Brookhaven National Laboratory on May 20, 2013, as part of the 2013 annual users meeting for the National Synchrotron Light Source and Center for Functional Nanomaterials. Presentations and conversations on the direction of X-ray and electron imaging complemented the mission of the NSLS-II early science workshop.

About 100 people attended a one-day workshop in May 2013 to discuss the future of synchrotron X-ray and electron imaging techniques, a future in which NSLS-II will play a significant role. Many of the projects discussed here will make use of the Hard X-ray Nanoprobe (HXN) and Submicron Resolution X-ray (SRX) spectroscopy beamlines – two of the initial suite of beamlines under construction at NSLS-II – and the Full-field X-ray Imaging (FXI) beamline, which will be built later.

Steve Pennycook (Oak Ridge National Laboratory) opened the workshop with an introduction to scanning transmission electron microscopy and electron energy loss spectroscopy (STEM-EELS). He discussed new opportunities for in-situ studies of solar cells by integrating the cathodoluminescence and electron-beam-induced current system. This allows researchers to correlate the electrical and optical properties of individual defects with atomic structure and composition.

Stephan Hruszkewycz (Argonne National Laboratory) gave a talk on Bragg projection ptychography, which extends lensless imaging to the Bragg regime and enables the investigation of extended objects. With this technique, he successfully imaged the 180° ferroelectric stripe domains in $PbTiO_3$.

Amy Clarke (Los Alamos National Laboratory) talked about using in-situ X-ray imaging to map the phase space of aluminum alloys during solidification. She also introduced an imaging technique using high-energy protons at the pRAD facility at Los Alamos National Laboratory. This technique gives her group the ability to penetrate cm-thick samples with a spatial resolution of approximately 10 μm.

Wilson Chiu (University of Connecticut) provided an overview of energy storage materials research using transmission X-ray microscopy (TXM). Three-dimensional nano-tomography and nano-XANES enabled him and his group to link the material's micro/nano structure to device performance. Ray Egerton (University of Alberta) discussed the complementary nature of X-ray and electron-beam analysis. He remarked that the basic limitations of both techniques arise mainly from radiation damage, particularly for organic specimens.

Kamel Fezzaa (Argonne National Laboratory) described the high-speed imaging program at the Advanced Photon Source (APS). He described how his group takes advantage of the six different storage-ring fill patterns at APS to conduct experiments that span more than six orders of magnitude ($1–10^6$ Hz) in equivalent frame rate and more than three orders of magnitude in exposure time (500 ns to 100 ps).

Can Erdonmez (Brookhaven National Laboratory) presented in-situ TXM measurements on the zinc anode of a flow battery, revealing the real-time process of zinc electrodeposition and dissolution during battery cycling. The morphological change during the process of zinc electrodeposition and dissolution is strongly related to current density and flow velocity.

Finally, Stefan Vogt (Argonne National Laboratory) described trace element imaging for the life sciences and relevant instrument development, including the Bionanoprobe at APS. He highlighted the implementation of fly-scans, which is a significant development for 3D microfluorescence measurements.

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Kyle Brinkman (Savannah River National Laboratory) identified possible early-science experiments on mixed ionic and electronic membrane systems; Wenge Yang (Carnegie Institute of Washington) later elaborated on the scientific opportunities for high-pressure science. Wilson Chiu (University of Connecticut), Esther Takeuchi (Stony Brook University and Brookhaven), and Kyung-Wan Nam (Brookhaven) discussed scientific problems facing the development of solid-oxide fuel cells and battery materials, some of which may be addressed at HXN.

Qingteng Zhang (University of Wisconsin) and Stephan Hruszkewycs (Argonne National Laboratory) discussed state-of-the-art experiments on ferroelectric materials and possible early-science experiments on ferroelectric domain dynamics and nanostructure. In related work, Oleg Shpyrko (University of California, San Diego) talked about future scientific opportunities for investigating magnetic domains. Conal Murray (IBM) discussed how he could use the HXN beamline to solve industrial problems facing nanoelectronics.

The XPD session was equally robust, attended by a large audience, with over a dozen participating via webcast. The XPD beamline will offer unprecedented resolution performance while maintaining high levels of photon flux in the 30–70 keV X-ray energy range. It will be a versatile, modular, high-energy X-ray diffraction and imaging instrument for real- and reciprocal-space diffraction studies of real materials in real time under real conditions.

A recurring theme during the session was the frontier of materials science research. Many scientists are working to develop transformational materials for energy production and energy storage, and the tools at XPD will further these types of studies. With that in mind, John Parise (Stony Brook University) discussed the high-pressure synthesis of novel oxynitride photocatalysts, which are potential water-splitting agents for the production of hydrogen fuel from water and sunlight. Ruibo Zhang (Binghamton University) and Feng Wang (Brookhaven) discussed how XPD tools could be used to monitor changes in the structure and composition of battery materials at their interfaces and in bulk phases.

The XPD beamline will have the ability to enable quantitative structural studies of polycrystalline samples, experiments that are unmanageable almost everywhere else. As discussed by Branton Campbell (Brigham Young University), this is important because many samples of interest cannot be grown as single crystals.

Session participants also talked about XPD's role in the multi-agency Materials Genome Initiative (MGI), which aims to halve the current time and cost for transitioning breakthroughs from the laboratory to the marketplace by tightly integrating theoretical predictions, syntheses, and testing of material properties – designing materials with specific properties rather than "chance" discoveries. XPD will provide valuable insight into some of these structures.

Many additional possible first experiments were proposed. These include studying nanocatalysts to fuel the water-gas shift reaction in hydrogen fuel cells; measuring the corrosion of zirconium alloys, which are used as cladding materials in nuclear reactors; several proposals in the area of high-pressure studies, which will be a key part of the XPD program; and studies into a possible hydrogen storage material, magnesium borohydride, under real working conditions.

The CSX sessions focused on those branch beamlines' signature capabilities – the highest coherent flux in the soft X-ray region (CSX-1) and fast switching of the incident X-ray polarization (CSX-2) – and the resulting science possibilities. CSX-1 will allow measurements of electronic textures (inhomogeneities) in strongly correlated electron systems with a resolution better than 50 nm by either scanning zone plate imaging or coherent diffraction imaging techniques, such as measuring stripe domains in high-temperature superconductors using resonant soft X-ray scattering. Attendees also discussed possibilities for X-ray photon correlation spectroscopy from magnetic systems based on recent work at the Advanced Light Source by Sujoy Roy et al., where information on the dynamics of magnetic domains close to a phase transition can be obtained.

During the CSX-2 session, participants discussed how its unique fast switching capabilities will dramatically increase the sensitivity of X-ray circular magnetic dichroism to measure such materials as dilute magnetic semiconductors. The possibility of a future program to focus on ambient pressure X-ray photoelectron spectroscopy was presented with an emphasis on catalysis science.

At the SRX session, presentations reflected that beamline's ability to host experiments using several techniques, including X-ray fluorescence imaging, X-ray absorption near-edge spectroscopy, X-ray spectromicroscopy, X-ray tomography, and X-ray micro-diffraction. It will offer high-flux and high-resolution modes, which will be useful for a variety of studies. Examples are investigations of nano-inclusions in solar cells, suggested by Steve Heald (Argonne National Laboratory), and platinum nanoparticle catalysts, as discussed by Yuanyuan Li (Yeshiva University).

Tim Glotch (Stony Brook University) described the potential role of SRX in the Solar System Exploration Research Virtual Institute proposal submitted to NASA. This is a multi-group project to study how materials on asteroids, the Earth's moon, and the Mars moons Phobos and Deimos weather bombardment by micrometeorites and solar wind. SRX will allow these samples to be studied with a resolution under 100 nanometers.

SRX may host research related to shale gas production, focusing on the nanoscale impact of minerals via the study of tiny grains – another case in which SRX's excellent resolution would be advantageous. This was presented by Jeff Fitts (Princeton University).

For a full wrap-up of the early-science workshop, including summaries of the CHX and IXS sessions, go to http://go.usa.gov/DJkj. ■

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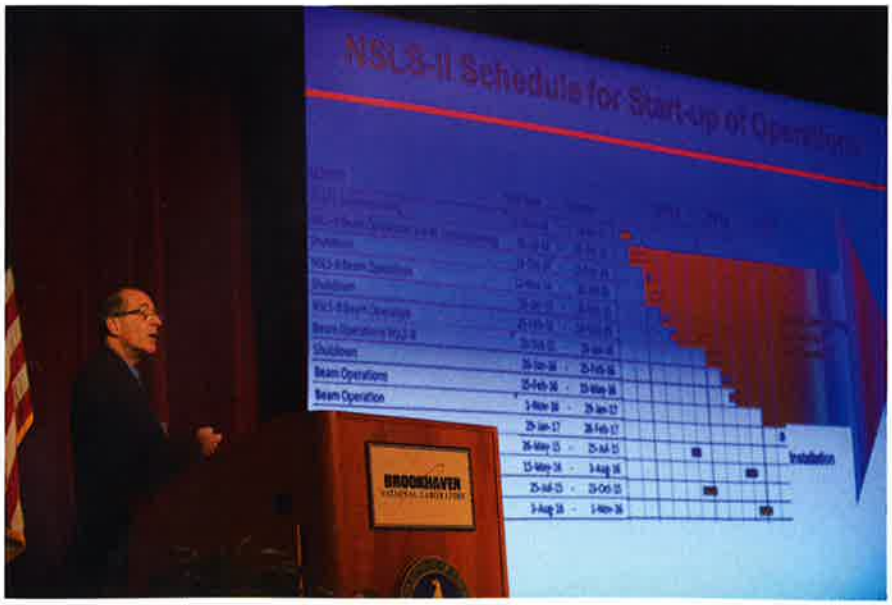

Paul Zschack, Brookhaven National Laboratory, outlined the schedule for NSLS-II commissioning and early operations.