Synchrotron Beam Line Operation and Development

The synchrotron radiation project is focused on the operation and continued development of unique experimental facilities at the Advanced Photon Source (APS) at Argonne National Laboratory and at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory (BNL). The emphasis is on the development and application of microstructural characterization tools and techniques that allow researchers from industry, universities and government laboratories to perform leading edge measurements on technologically advanced materials.

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At the APS, the Ceramics Division, through UNICAT (a NIST/University of Illinois/Oak Ridge National Laboratory/UOP collaboration), supports activities in ultra-small-angle x-ray scattering (USAXS) and USAXS imaging on the insertion device line and x-ray absorption fine structure (XAFS) and diffraction topography on the bending magnet line. A hard x-ray microscope is under development. As in previous years, the USAXS instrument received the highest demand of any UNICAT instrument, with nearly 50 % of the available beam time being used for USAXS experiments. The USAXS instrument has been upgraded with an overconstrained weak-link rotational stage, which has significantly improved stability and reproducibility. The software for data reduction has been completed and the first version of software for data analysis is available on the Web. Examples of research supported by this facility include: in-situ production of nanoparticles in a flame; fuel cell microstructure: fillers for human medical implant glues: and imaging of human and artificial tissues.



Figure 1: XAFS data for SrTiO₃ thin films.

On the bending magnet beamline, NIST researchers have used XAFS and diffraction to study SrTiO₃ single-crystals and thin films grown on Si (100) surfaces. Figure 1 shows the Ti-K near-edge XAFS for a series of thin films of differing thickness. The increase in the intensity of the pre-edge peak for films thinner than about 80 Å indicates hybridization of the Ti 3d and 4p states. This hybridization occurs because inversion symmetry is broken due to strain from the substrate. This "clamping effect" leads to a local ferroelectric transition in the films.

At the NSLS, the NIST/Dow beamline (U7A) is recognized as a leading materials science experimental facility on the UV ring. For the last reporting period from the NSLS, beamline U7A accounted for over 1/3 of the total number of publications reported for the UV ring.



Figure 2: Cover of Applied Physics Letters.

Research from this facility was featured on the cover of the January 13, 2003 issue of *Applied Physics Letters*, as shown in Figure 2, "Combinatorial near-edge x-ray absorption fine structure: simultaneous determination of molecular orientation and bond concentration on chemically heterogeneous surfaces." This year, a larger and more versatile main experimental chamber was commissioned, enabling all of our unique fluorescence and electron detection capabilities to be installed simultaneously.

Contributors and Collaborators

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