User Science Division Report

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Organization and Mission

The User Science Division coordinates major facility activities related to users so that we can be more effective in communicating with the user community, strengthening existing scientific programs, fostering the growth of new scientific programs, and raising the visibility of the exciting science produced by our users both inside and outside the scientific community. The division consists of five sections: User Administration (Mary Anne Corwin), Information and Outreach (Lisa Miller), Beamline Development and Support (Steve Hulbert), Scientific Program Support (Ron Pindak), and Detectors and Controls (Peter Siddons). The major initiatives and accomplishments of the User Science Division and the NSLS user community for 2005 are summarized below.

2005 Activities

Beamline Reviews

With the cooperation of participating research teams (PRTs) and assistance from a large number of NSLS staff, all operational beamlines at the NSLS were reviewed in January-February 2005 by the NSLS Scientific Advisory Committee (SAC). In this round of review, beamlines were divided into seven groups: macromolecular crystallography, x-ray scattering/soft condensed matter, x-ray scattering/hard condensed matter, powder/single crystal diffraction and high pressure, imaging and microprobe, x-ray spectroscopy, and IR/UV/soft x-ray spectroscopy. Each group was reviewed by an ad hoc beamline review committee led by one or two members of the SAC. The goal of the review was to ensure the highest scientific productivity and safe operation of these beamlines. Specifically, the following areas were evaluated by the review committee for each beamline: (1) importance of the scientific program; (2) quality and quantity of scientific productivity; (3) quality of beamline instrumentation, including beamline optics/controls, endstations, detectors, and software; (4) funding for the beamline operation and beamline/endstation upgrades; (5) effectiveness in beamline usage; (6) level of beamline staffing; and (7) safety-related issues, including beamline operation procedures and user training. The evaluation and recommendations from the beamline review were accepted by NSLS management and communicated to the PRTs soon after the review. Most of the PRTs have been renewed for another three years. We are working closely with the remaining PRTs to resolve the issues raised by the review.

User Access Policy

Another major undertaking in 2005 was the establishment of facility beamlines (FBs) under the new NSLS User Access Policy. A set of approximately 15 beamlines were selected as FBs in the first phase, based on coverage of the primary research directions of the user community, available resources, and considerations of complementary capabilities provided by the PRTs. The goal of these beamlines is to allow general users to have access to all wavelengths and major synchrotron techniques. In addition, general users will also have better coordinated support from the facility's scientific and technical staff. Users will also benefit from enhanced instrumentation at these beamlines as a result of the beamline/endstation upgrade projects carried out over the last few years. These FBs were put into operation starting from the fall cycle of 2005. We are looking forward to working with and receiving feedback from users at these beamlines. Finally, we initiated a contributing user (CU) program on the FBs, with the first CU proposals submitted in September 2005. The CUs enhance the endstation capabilities on the FBs. In some cases, this enhancement involves specialized instrumentation to meet the needs of specific research communities, such as catalysis or environmental sciences. In other cases, the enhancement involves the implementation of a specific x-ray technique, such as x-ray standing wave or strain-mapping. The CUs also provide supplemental user support to strengthen the scientific impact of the FBs.

Education and Outreach

We continue to take steps to enhance user education, training, and outreach at the NSLS. In the area of user training, we organized a "High Resolution Powder Diffraction Data Collection and Analysis" short course, with the help of Peter Stephens (Stony Brook University) and Christie Nelson (NSLS). It was an intensive three-day course that included lectures on the basic physics of powder diffraction, indexing, and Rietveld refinement, as well as cutting-edge research using powder diffraction. The course also involved hands-on data collection carried out on NSLS beamlines X3B1, X7A, and X14A. Feedback on the short course was very positive, and we are considering running it annually. We also continued our annual EXAFS short course in 2005, which was entitled "EXAFS Course: Theory, Experiment, and Advanced Applications." We worked closely with users to organize additional focused scientific workshops, including "RapidData 2005," a workshop on "Strain Mapping in Engineering Materials with High-Energy Synchrotron X-Rays," a "BNL Workshop on Intense Coherent THz Pulses," a "Crystallization Workshop," "Bio-CD 2005," the "Synchrotron Environmental Sciences III (SES-III)" conference, a workshop on "Synchrotron Infrared Spectroscopy for High-Pressure Geoscience and Planetary Science," and three tutorials entitled the "X6A Workbench: Hands-On Training in Synchrotron Crystallography," These workshops were very effective in introducing the use of synchrotron techniques to a particular area of science to non-synchrotron users. Many new research opportunities and fruitful collaborations have resulted from them. Finally, a concerted effort has been made this year to coordinate closely with the Center for Functional Nanomaterials, in order to reach out to nanoscience researchers through joint seminars, workshops, and visits to interested universities and institutions.

Beamline Upgrades

There were a number of significant beamline upgrade projects completed this year by the NSLS and PRTs, including an upgrade of the X18B monochromator, the construction of a micro-beam diffraction endstation at X13B, an upgrade of the U13 photoemission spectrometer, the design and testing of Quick-EXAFS, the development of a double-focusing high-energy monochromator for X17, and the implementation of high field magnets at several beamlines around the ring. These highlights are described in detail below.

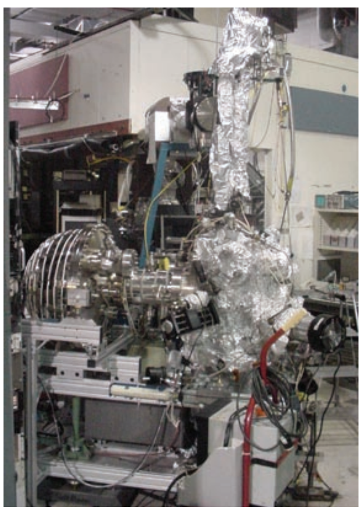


Figure 1. Photograph of the U13UB experimental endstation, the heart of which is a very high resolution Scienta SES200 photoelectron spectrometer.

New High-Resolution Electron-Energy Analyzer Installed on Beamline U13UB

The 1990s renaissance in the field of angle-resolved photoemission spectroscopy (ARPES) resulted from the combination of new parallel-detection (in energy and angle) photoelectron spectrometers with high-brightness VUV synchrotron beamlines. At the NSLS, such a beamline/endstation combination was constructed and commissioned at beamline U13UB in the late 1990s.

In 2004, the original Scienta photoelectron spectrometer that had been used since 1998 was replaced by a higher-resolution instrument from the same company. Shown in **Figure 1**, the new instrument has a measured energy resolution of 0.7 meV, which is a significant improvement over the ~5 meV resolution of the previous model. A photoelectron spectrum recorded at low temperature from an evaporated gold film in the vicinity of the Fermi edge is shown in **Figure 2**. The energy width of the Fermi edge in this spectrum is limited by the temperature of the sample.

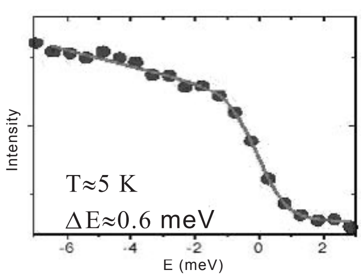


Figure 2. A photoelectron spectrum recorded at low temperature (~5 K) from an evaporated gold film in the vicinity of the Fermi edge, demonstrating the superior (sub meV) electron energy resolution of the new Scienta photoelectron spectrometer at beamline U13UB.

This new instrument has been used by U13UB PRT members and general users since late 2004. The U13UB PRT members (Brookhaven Lab's Physics Department, Boston University, Boston College, and Columbia University) are using the enhanced energy resolution of this new instrument to study detailed information on the electronic structure and dynamics of complex electronic systems. In particular, a recent study of the quasiparticle scattering rates around the Fermi surface of Sr_2RuO_4 was reported in Physical Review Letters by the Brookhaven group (PRL 94, 107003 (2005)). This study provided a microscopic picture of the origin of the crossover from non-Fermi liquid to Fermi liquid behavior observed in macroscopic transport measurements.

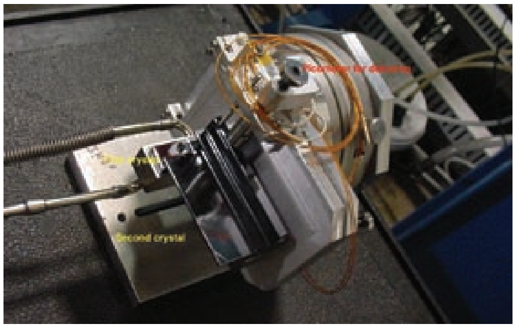
An X-ray Micro-Diffraction Instrument for Materials Research
A new x-ray micro-diffraction instrument was developed at X13B to take advantage of the small source size of the in-vacuum mini-gap undulator in the X13 straight section of the NSLS x-ray ring. This instrument combines sub-micron spatial resolution, exceptional reciprocal space access, a choice of focusing optics, and a modular design to accommodate different experimental needs.

A New High-Energy X-ray Side Station for High-Pressure Research
A new side-diffracting double-focusing high-energy x-ray monochromator was developed to provide 30-100 keV monochromatic x-rays to a new endstation in beamline X17B2, which includes a new high-pressure press and a MAR345 image-plate detector. It will enable high-resolution angle-resolved diffraction and pair-distribution function measurements to allow structure refinement for materials under high pressure.

A Fast Scanning Monochromator for X-ray Absorption Spectroscopy
A new monochromator drive and data-collection scheme has been developed to allow fast measurement of x-ray absorption spectra. This new method reduces the collection time of a typical extended x-ray absorption fine structure (EXAFS) spectrum to less than one second. It will be particularly important for the study of kinetics in catalytic reactions.

X18B: A New Monochromator for X-Ray Absorption Spectroscopy Between 4.9 and 40keV
A new monochromator has been installed at beamline X18B to lower its lower energy limit from 5.6 keV down to 4.9 keV. This change might sound small, but it opens significant opportunities for research — especially in solid-state physics, materials science, and catalysis research — since the K-edges of two important 3d-transition metals, titanium and vanadium, have their energies at 4.966 and 5.465 keV, respectively.

Beamline X18B is optimized for hard x-ray absorption spectroscopy. The original monochromator covered an energy range between 5.7 and 40 keV (Cr – Ce K-edges, Ce – U L-edges). An overlapping lower energy range (2-7 keV) is covered by beamline X19A, which is optimized for x-ray absorption spectroscopy below 4 keV. However, several research groups, especially in the field of catalysis research, do experiments at the K-edges of several 3d-transition metals. The typical duration for one of their experiments is two to three days, including a few hours of research at photon energies below 5.7 keV (i.e. the V and Ti K-edges).



New X18B monochromator with smaller first crystal.

Rather than attempt to schedule and setup experiments at both X18B and X19A, we decided to extend the lower energy range of X18B down to 4.9 keV via a simple modification of its monochromator. The monochromator at X18B is a standard channel-cut monochromator with a 3mm gap between the two crystals. The original length of the first crystal was 34 mm, which limited the maximum usable angle of operation of the monochromator, and in turn limited the low end of the photon energy range to 5.7 keV. The 34 mm length of the first crystal was chosen in order to intercept the entire beam vertically at higher energies, where the Bragg angle, and thus the vertical acceptance of the crystal, is smaller.

However, as it turns out, the vertical opening angle worsens the energy resolution at higher energies, so it is better to limit the vertical beam size at high energies, thereby shortening the required length of the first crystal and increasing the usable angular and photon energy ranges of the monochromator. By optimizing both the energy resolution and intensity, the footprint of the white beam on the monochromator remains almost constant over the energy range of this monochromator. This allowed us to shrink the length of the first crystal to 16 mm, which is still enough to prevent heat-load problems like the thermal bump.

The monochromator was first mounted in the beamline in the end of April, and tested and commissioned in April and May.

Beam Stability Tests at X18B

One important parameter in the success of experiments at the NSLS is the stability of the x-ray beam on the sample. Properties such as intensity, beam position, energy stability, and in a few cases degree of polarization play crucial rules in this success, and some can be neglected based on the experiment. Ideally, these properties, when normalized by their dependence on ring current, do not change over the course of an experiment. Change from the ideal performance can be caused by motions of the electron beam in the storage ring or by motions of the beamline optics. The latter are often thermally driven, their effect is not proportional to beam current, and they can cause any combination of beam position changes, intensity changes, or energy changes. Ideally, these motions are eliminated by stable supports (beamlines and storage ring), feedback systems (electron beam and, in some cases, beamline optics), and efficient cooling schemes. In practice, however, both the electron beam and the beamline optics move slightly over time, requiring regular realignment.

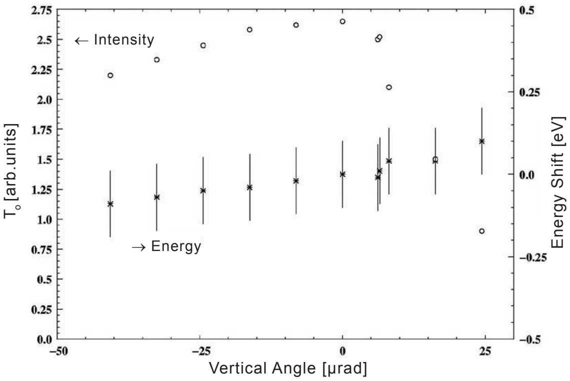


Figure 1. Intensity and energy shift for changes of the vertical angle. The stability of the beam is better than 5 μrad.

During a beam studies period, we measured the effect of beam motion on the intensity and energy of beamline X18B, which is equipped with a channel-cut Si(111) monochromator located at a distance of 18 meters from the source. X18B accepts a vertical (horizontal) divergence of 50μrad (0.5-1 mrad). Since X18B is optimized for x-ray absorption spectroscopy, we do not care too much about position stability. In contrast, we care a lot about source angular stability, since this directly affects the photon energy selected by our monochromator.

Based on the geometry of the X18B beamline (slit sizes and positions), we calculate that vertical offsets of the source (electron beam) by 60μm or angular changes by 5μrad cannot be observed in a typical experiment. In order to test these calculations, we aligned the beamline slits such that the beamline becomes sensitive to beam motions in one direction (angle or position), but not the other. We then moved the beam (angle and position) around the nominal values and measured the intensity and energy stability. We did not study the effects of orbit motion (position or angle) in the horizontal plane, since X18B is not sensitive to motion in this plane.

The results of the orbit studies described above on the intensity and energy of beamline X18B are shown in **Figures 1** and **2**. It is apparent that vertical angular changes have only a small effect on the energy calibration, independent of the slit position, but have a relatively large effect on intensity. Theoretically, the energy should remain constant as a function of vertical source angle, which demonstrates that the X18B beamline saw a positional shift in addition to an angular change during these studies. The photon energy shifts significantly more for changes in the vertical source position, about 0.1 eV for 100 μm, as expected. The intensity profile in **Figure 2** is an asymmetric function of vertical source position relative to the standard orbit value. The reason for this is that we aligned our three beamline slits/apertures (5 mm vertical Be-window, 1mm vertical white beam slit, 0.5 mm vertical hutch slit) such that the lower edges of their upper jaws were aligned, as viewed from the source point. In this situation, if the electron beam moves up, one of the slits intercepts the beam and the intensity is reduced immediately. Beam motions in the other direction, however, move the beam more towards the center of the three slits, so the intensity is much less strongly reduced, as can be observed in the data.

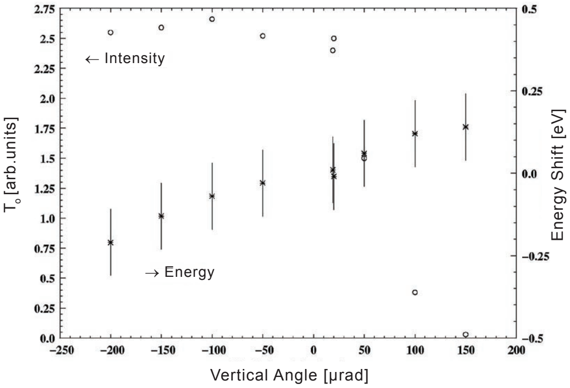


Figure 2. Intensity and energy shift for changes of the vertical position. The stability of the beam is better than 50 μm.

In summary, we confirmed our calculations for the sensitivity of the X18B beamline to electron beam motions and demonstrated that the intensity can vary significantly if the beamline is not aligned appropriately. For normal operations, we align the slits symmetrically about the nominal beam axis in order to maximize intensity and to maximize the intensity stability. During normal operations, the intensity varies less than 0.4%, and the energy is stable within 0.05 eV.

High Resolution Terahertz Spectroscopy in Magnetic Field

Over the past few years a new magneto-optical facility has been developed at the U12IR beamline at the NSLS. The main components are an Oxford Instrument superconducting magnet, and a Bruker IFS 125HR high-resolution spectrometer. -The principal reason for using the synchrotron source in far-IR spectroscopy is the brightness advantage over a conventional light source -- depending on the frequency range and on the sample geometry, the synchrotron results in a factor of 50 - 200 gain in the intensity, making a vast array of new measurements possible.

The Oxford Instruments magnet, installed next to the Bruker IFS 125HR spectometer. The stainless steel tubes contain the optical coupling to the VUV-IR ring and between the spectrometer and the magnet.

The Bruker spectrometer is essentially an interferometer where spectral resolution is proportional to the available path difference (between the two interferometer “arms”). This particular spectrometer has an extremely long path difference, yielding a 0.001 cm-1 (0.125 μeV) resolution. The available spectral range is from 5 cm-1 (0.63 meV) to over 7000 cm-1. The magnet can produce fields up to 16 Tesla and its 20 liter He reservoir has a hold time of nearly 1 week. A set of three wedged single crystal quartz windows at the bottom provide optical access to the sample from below, along the vertical axis of the magnet (see figure). The sample temperature can be varied between 1.8 K and room temperature.

The facility was tested extensively and the first results have been published on $LaMnO_3$, a well-known antiferromagnet and the parent compound of the so-called colossal magnetoresistance materials. Other projects currently in progress include: the study of the single molecular magnet Mn12-acetate (in collaboration with Myriam Sarachik, City College of New York), spin resonance on $NaNiO_2$ (with Sophie De Brion, Grenoble HMFL), the investigation of correlated magnetic systems, including $LiCu_2O_2$ and others (with Laszlo Forro, EPFL, Lausanne), and magneto-optical studies on superconductors, including carbon-doped MgB_2.

A 10T Superconducting Magnet for Magneto-Structural and Magneto-Electronic Research and Education

A horizontal-field, split-coil superconducting magnet has become available for magneto-structural and magneto-electronic research at the NSLS. The magnet can operate over the temperature range from 1.6 to 300 K and over the field range 0 to 10 T. The horizontal field enables x-ray absorption measurements with linearly polarized x-rays with the electric field of the beam parallel to or normal to the magnetic field. Care was taken in the design to ensure that the samples can be measured in transmission mode for energies down to 5 keV (and in small steel hutches).

In general, this magnet is being used to study the lattice-spin coupling in complex materials. It was designed to be used on beamlines X11A, X19A, X21A and X23B for structural measurements (XAFS and powder XRD) in magnetic fields. With the use of a quarter wave plate, hard x-ray circular dichroism measurements on these beamlines will be feasible. With a differential pumping configuration, a future upgrade will make possible x-ray magnetic x-ray magnetic circular dichroism (XMCD) measurements on beamlines X13A and U4B. This will enable the study of magnetism in hard magnets as well as the induced moments on oxygen sites in metal oxide systems. Optical access is possible by replacing the kapton windows with quartz windows and/or replacing the variable temperature insert windows and external windows with all-quartz windows. The magnet was funded by a NSF IMR Grant and is now operational.

The magnet set up at X19A. It is mounted on a xyz table, which can be motor-controlled for ease of alignment. Samples are loaded from the top on a vertical rod into the samples space and are cooled by helium vapor or liquid from the magnet reservoir.