

## 2004 NSLS Annual Users' Meeting Caps Off Another Successful Year

May 17-20, 2004

Almost 400 participants at the 2004 NSLS Annual Users' Meeting, held May 17-20 at Brookhaven National Laboratory (BNL), came together for a successful event full of interesting talks and important messages for the future. The meeting affirmed that the NSLS continues to be a facility that produces important science.



The new NSLS Users' Executive Committee Chair, Larry Shapiro, gave welcoming remarks at the main meeting.

In his welcoming remarks at the main meeting on May 18, the new Users' Executive Committee (UEC) Chair, Larry Shapiro, discussed the continuing excellence of NSLS research, such as the cellular ion channel structure determined by user Roderick MacKinnon, performed at the NSLS and the Cornell High Energy Synchrotron Source. This work earned MacKinnon the 2003 Nobel Prize in Chemistry.

Steve Dierker, NSLS Chairman and Associate Laboratory Director for Light Sources, described another

important crystal structure, a cellular protein channel, recently determined here and featured on the cover of *Nature* in January 2004.

He also described other research highlights – a new liquid crystal phase in polar ordered materials, featured on the cover of *Science* in August 2003; a new material texture type, appearing in *Nature* in December 2003; and a new



Among the main meeting speakers at the Annual NSLS Users' Meeting were (from left): Bob Casey (BNL-NSLS), Pedro Montano (DOE), Mark Croft (Rutgers University), Steve Dierker (BNL-NSLS), John Hill (BNL-Physics), Lois Pollack (Cornell University), Simon Billinge (Michigan State University), and Chris Jacobsen (Stony Brook University).

way to store hydrogen in molecular compounds, published in January 2004 in the *Proceedings of the National Academy of Sciences*.

"These give you a flavor of the diversity and quality of science that continues at this facility," he said. "The NSLS has a well-deserved reputation for outstanding productivity."

The main meeting's scientific talks focused on user research from a more in-depth perspective. Chris Jacobsen, of Stony Brook University (SBU), gave an overview of the many x-



Yvonne Akpalu (RPI) discussed the use of light and x-ray scattering on copolymers.

ray and infrared imaging techniques available to users at the NSLS.

Mark Croft, from Rutgers University, described his research on strain fields in macroscopic materials, in which he uses an energy dispersive x-ray diffraction method.

Simon Billinge, of Michigan State University, presented his work on the structures of complex materials that display order on the nanoscale, using the rapid acquisition pair distribution function technique he developed with his group. Brookhaven Lab's John Hill discussed how he

uses soft x-ray scattering to probe the behavior of electrons in solids.

Another speaker, Cornell University's Lois Pollack, described an apparatus designed by her research group, which allows them to observe, using small angle x-ray scattering (SAXS), how an RNA chain molecule compacts and folds into a three-dimensional structure. Following this, Yvonne Akpalu, of Rensselaer Polytechnic Institute, presented her work, which investigates how to solve the crystal structure of a copolymer using SAXS.

On the days before and after the main meeting, users divided up to listen to additional talks on specific topics. These talks were organized into the following workshops: "Better Ways to See the Light: Advanced Detectors for Synchrotron Radiation," organized by Peter Siddons (BNL) and Gianluigi De Geronimo (BNL); "Anatomy of a Virus," organized by Marc Allaire (BNL) and Paul Freimuth (BNL); "Grazing Incidence Small Angle Scattering," organized by Ben Ocko (BNL) and Detlef Smilgies (Cornell University); "Pharmaceutical Applications of Synchrotron Radiation," organized by Evgenyi Shalaev (Pfizer Inc.), Raj G. Suryanarayanan (College of Pharmacy), and Peter Stephens (SBU); "Advanced Optical Systems and Metrology for High Power and Coherent Beamlines," organized by Peter Takacs (BNL) and Steve Hulbert (BNL); "Applications of Synchrotron Based Methods to Hydrogen Storage Materials," organized by Trevor Tyson (New Jersey Institute of Technology) and Wolfgang Caliebe (BNL); "Nanoprobes for Nanoscience,"

organized by Cecilia Sanchez-Hanke (BNL) and Peter Sutter (BNL); and “Crystallization, Membrane Proteins,” organized by Naomi Chayen (Imperial College of Science, Technology & Medicine) and Vivian Stojanoff (BNL).

**Now and Ahead**

On the performance front, Dierker said the facility did well overall in the last year, with the VUV ring achieving 98 percent reliability and 108 percent availability. The x-ray ring



Brookhaven National Laboratory Director Praveen Chaudhari discussed his vision for the Laboratory in the next 20 years.

didn't perform quite as well, operating at 89 percent reliability and 99 percent availability. He noted that more than 60% of the downtime on the x-ray ring was due to three unusual major events, including the Northeast electrical power blackout last August.

In a message users are very familiar with, he stressed that the NSLS, designed 30 years ago and the only remaining second-generation DOE light source, is now performing at the limits of its capabilities. Pedro Montano, manager of the X-ray and Neutron Scattering Facilities program within the Department of

Energy's Office of Basic Energy Sciences, acknowledged this in his talk, referring to the NSLS as the “working horse” of the DOE light sources.

Dierker and Montano repeated the need and excitement for NSLS-II, the proposed third-generation light source that would replace the NSLS.



Members of the 2004 planning committee for the NSLS Users' Meeting include: (from left) Larry Shapiro (Columbia University), Liz Flynn (BNL-NSLS), Mary Anne Corwin (BNL-NSLS), Tony Lanzirotti (University of Chicago), Gretchen Cisco (BNL-NSLS), Lisa Miller (BNL-NSLS), Vivian Stojanoff (BNL-NSLS), Paul Stevens (Exxon Mobil Research & Engineering Company), and Dan Fischer (National Institute of Standards & Technology).

Currently, Montano said, an international panel of scientists is reviewing the proposal – the first step in the process. He urged the prospective users of NSLS-II to contact their representatives in Congress and tell them how vital the facility is.

“I think NSLS-II is necessary,” Montano said. “There are a huge number of scientists in the Northeast that would benefit from it.”

All of the day's speakers affirmed this idea – that NSLS-II is needed to broaden and enrich their research.



Center for Functional Nanomaterials (CFN) Director Robert Hwang gave the “CFN Update” at the main meeting.

**BNL and the NSLS**

In his “CFN Update” talk, Robert Hwang, director of the upcoming Center for Functional Nanomaterials (CFN) facility, discussed how the CFN will be “an interdisciplinary environment for nanoscience research,” performed in conjunction with several BNL departments, including the current NSLS and NSLS-II.

In this way, the research the CFN will enable promises to be very exciting. “The Northeast is becoming a hotbed for nanoscience,” said Dierker. “Brookhaven is becoming a focal point for much of that research.”

The future of Brookhaven Lab was the subject of Lab Director Praveen Chaudhari's talk. In his vision for the Laboratory in the next 20 years, Chaudhari said NSLS-II plays a significant part in his desire to see more integration between departments, and the emergence of a new culture and way of thinking at the Lab.

“In a research lab, departmentalizing can be a hindrance,” he said. “To reach the frontiers in the life, physical, and environmental sciences, we need to find the major challenges at these interfaces between disciplines, and begin to address them.”

Another Lab-wide initiative – safety – was discussed by Bob Casey, the NSLS Associate Chair for Environment, Health, Safety, and Quality. While the NSLS safety record hovers around the DOE average, “We're being asked to really improve,” Casey said. “We want to be best in class.”

The types of accidents that occur at the NSLS are sometimes considered routine, such as slips and muscle strains, but, as Dierker said in his talk, “Accidents have consequences.” Additionally, there have been near misses at BNL in the special-hazard category, which include more serious electrical, radiation, and laser accidents.



Poster prize winners at the 2004 NSLS Users' Meeting were (from left to right): Dario Arena (BNL-NSLS), Xianqin Wang (BNL-Chemistry), Marianna Kissell (SBU), Mehmet Aslantas (BNL-NSLS), and Alexei Grigoriev (Harvard University). Not pictured: Meghan Ruppel (SBU).

Casey made it clear that these must also be avoided, ending his talk with a series of photographs showing obvious safety violations in several areas of the NSLS. The pictures made it clear that safety really is an issue everybody must take seriously.



Tuesday night's Reggae Banquet was enjoyed by all including (from left to right): Gene Ice (ORNL), Pedro Montano (DOE), Peter Siddons (BNL-NSLS), Chi-Chang Kao (BNL-NSLS), and Chris Ryan (CSIRO, Australia).

### Honorable Mentions

Numerous honors and awards were presented at the close of the meeting. Outgoing UEC chair Tony Lanzirotti presented the UEC Community Service Award to Sue Wirick of Stony Brook University. Additionally, Lisa Miller, the meeting's poster session and publicity chair, gave out awards for the best posters. The winners were Dario Arena (BNL-NSLS), Mehmet Aslantas (BNL-NSLS), Alexei Grigoriev (Harvard University), Marianna Kissell (SBU), Meghan Ruppel (SBU), and Xianqin Wang (BNL-Chemistry).

At the evening banquet, Shapiro presented the meeting's organizers – Mary Anne Corwin, Liz Flynn, Gretchen Cisco, and Melissa Abramowitz – with framed letters of appreciation. This year's banquet was reggae-themed, complete with a reggae band, colorful hats, and island fare.

—Laura Mgrdichian

## Better Ways to See the Light: Advanced Detectors for Synchrotron Radiation Workshop

May 17, 2004

The workshop attracted 62 registrants, who enjoyed a wide range of talks about the latest in detector developments for synchrotron radiation (SR) applications. The following is a very brief resumé of the talks presented.

Many people think the center of this activity is in Europe these days, and to bring us up to date with what is happening over there, we heard Dr. Gareth Derbyshire (from the Rutherford-Appleton Laboratory in the UK) talk about "Detector Developments for Synchrotron Radiation in Europe." He described work underway at the Daresbury and Rutherford laboratories, as well as work being pursued by the Swiss Light Source in collaboration with the Centre Européenne pour la Recherche Nucléaire (CERN). In particular, he stressed the need to provide advanced detector systems to the UK's Diamond Light Source, currently under construction, ready for use on 'day 1.'

There are many interesting detector developments happening in fields other than SR. The next speaker, Dr. Oswald Siegmund, was from the Space Sciences Laboratory at the University of California Berkeley. In his talk, "Microlithographic Silicon-Based Microchannel Plates and Readout Techniques for Photon and Particle Detection," he told us of some interesting developments in microchannel electron multiplier device technology, in particular the use of micro-machining techniques to make precision microchannels in silicon wafers. Such devices offer much improved uniformity and reduced noise over conventional drawn-glass structures. He also described readout electronics they are capable of providing good spatial resolution in one and two dimensions, for use with these new channel plates.

There is a real lack of detector technologies that are suitable for hard x-ray detection. Perhaps the most promising at the moment is cadmium-zinc telluride (CZT). This material is not ideal, but the use of special signal processing techniques can be helpful. Gabriella Carini (Brookhaven National Laboratory) spoke about "A New Integrated Circuit for Coplanar-grid Detector Readout." The coplanar grid detector has a special electrode structure which can mitigate the poor hole mobility in these room-temperature semiconductor detectors. The talk described a new microcircuit designed by the BNL microelectronics group, which provides enhanced capabilities for such devices.

A key part of any detector system is the software used to control the detector and analyze the data produced. Dr. Chris Ryan, from the Commonwealth Scientific & Industrial Research Organization (CSIRO) in Australia, has developed "A New Technique for Real-Time Spectral

Deconvolution of Energy-resolving Detector Data,” which can generate quantitative elemental maps from fluorescence microprobe data, in real-time. Simple methods using pulse-height windows can lead to serious errors in elemental identification due to strong peak overlaps. Chris’ technique is able to account for all these overlaps, together with other artifacts such as escape peaks, and build up an elemental map photon-by-photon.

An exciting development that is beginning to become more accessible is that of very high-resolution x-ray detectors based on cryogenic superconducting technology. Kent Irwin (National Institute of Standards and Technology, Boulder, CO) in his talk “Cryogenic Microbolometer X-ray Detectors,” brought us up-to-date on these devices. They can reach single-digit eV resolution in the x-ray region, a remarkable achievement. The disadvantage of these detectors is that they are rather slow, and Kent described how his group is working to overcome this limitation by making large arrays of detectors, and how superconducting quantum interference device (SQUID) technology used to read them out can be integrated into such an array device.

Even the best detectors sometimes need help, and in the field of absorption spectroscopy this is particularly true. In his talk “Multilayer Optics for Fluorescence Detectors,” Ke Zhang (BioCAT, Argonne National Laboratory) described the development and application of multi-element analyzer systems based on synthetic multilayer optical elements. The instrument he presented provides an efficient filter that only allows a particular spectral line to pass to the detector. The multiple elements provide enhanced solid angle and hence efficiency.

Perhaps the most powerful driver for advanced detector development in the synchrotron community has been the demands of macromolecular crystallography. The talk by Dr. Edwin Westbrook (Molecular Biology Consortium Inc.) titled “Silicon Pixel Array Detectors for Protein Crystallography,” described some new ideas for fabricating detectors using non-traditional processing techniques. The result is a technology that can produce efficient arrays of detectors with excellent properties. One point that is unique to this technology is that the arrays do not have any dead area around the edges, so detectors can be easily tiled together to



Better Ways to See the Light: Advanced Detectors for Synchrotron Radiation Workshop attendees

make large area systems without any lost image regions.

Tae Joo Shin (NSLS) then told us about his work using 2-D position-sensitive proportional counters (PSPCs) for x-ray speckle experiments. Present speckle experiments typically use either a single-point detector and photon-counting with high time resolution, or a CCD area detector, which has a readout time of, at best, tens of milliseconds. PSPCs have the potential to combine the advantages of a photon counting detector and an area detector. Preliminary experiments to characterize and improve the performance of such detectors to make them suitable for speckle were described.

The final talk was to have been given by Dr. Mark Rivers (University of Chicago). Although circumstances prevented him from giving the talk at that time, it was rescheduled for a few days later. It also concerned the software implications of advanced detector systems. This area of development is extremely important, and the difficulties and required effort are frequently underestimated. Mark told us about the software solutions he has developed for interfacing area detectors, in particular CCD devices, to data acquisition systems based on the EPICS framework.

The breadth and depth of these talks only scratch the surface of what we believe is the last remaining barrier to the full utilization of our powerful synchrotron facilities. We look forward to hearing much more of these developments at future meetings.

—D. Peter Siddons and Gianluigi De Geronimo

## Anatomy of a Virus Workshop

May 17, 2004

It was a memorable day at the workshop, “Anatomy of a Virus,” where prominent experts in the field got together to talk about their favorite subject. Sponsored by the User Executive Committee at the National Synchrotron Light Source, the National Institute of Health (National Institute of Allergy and Infectious Diseases, National Institute of General Medical Sciences, and National Center for Research Resources) and Area Detector System Corporation (Poway, CA), this full day workshop highlighted the frontiers in the study of viral structure and the future needs in structural virology. With the impressive list of speakers that were present on that day (front row of the picture), we all had the feeling that the future of structural virology was already there.

The workshop started with no less than a historical perspective given by Donald Caspar (Florida State University), which was elegantly introduced by Dieter Schneider. Don related the important factors that were required for the success and the birth of structural studies



Anatomy of a Virus Workshop attendees

of viral capsids. The next speaker was Denis Leclerc (Laval University), who reported on his progress on the assembly of nucleocapsid-like particles of hepatitis C virus. Their results suggest that the first N-terminal half of the core protein would be sufficient for the formation of viral particles. Michael Chapman (Florida State University) presented his work on structural studies of a gene therapy vector, the adeno-associated virus. From their atomic x-ray structure, it was possible to predict the viral site where it attaches to the cellular receptor. Huilin Li (Brookhaven National Laboratory) talked about genomic RNA packaging in the vesicular stomatitis virus. By single-particle cryo-electron microscopy, a ring of density was revealed on the inner surface, and the density is proposed to be the RNA. Stacy Benson (the Wistar Institute) followed and reported on structural studies of the lipid-containing bacteriophage PRD1. The x-ray crystal structure of its major coat protein has revealed a molecule that contains two viral jelly rolls and appears to link PRD1 evolutionarily with the human adenovirus. Michael Rossmann (Purdue University) talked about “Membranes and Motors: Where Electron Microscopy Meets Crystallography.” He described a variety of examples in which crystallography and cryoEM were combined to construct detailed atomic models of large complexes. A striking example was the structure of the bacteriophage T4 base plate assembly in two different states. High-resolution structures of component proteins, combined with cryoEM reconstructions of the entire base plate complex in the extended and contracted states, revealed the extraordinary dynamic character of this complex and the large-scale motions of individual proteins.

The afternoon session started with Philip Dormitzer (Harvard Medical School) describing the structural rearrangements during rotavirus cell entry. His group's recent structure of the membrane penetration domain of VP4 reveals a fold-back rearrangement reminiscent of the enveloped virus fusion proteins that mediate enveloped virus cell entry. Wayne Hendrickson (Columbia University) gave his presentation on the structural biology of HIV attachment and entry into cells. His studies suggest that the flexibility of the glycoprotein GP120 of HIV is implicated in the escape to the immune response. The next talk, “Motors

and Membranes: Where Crystallography Meets Electron Microscopy,” was given by David Stuart (Oxford University). He presented his extensive work on the x-ray crystal structure analysis of the entire 66 MDa bacteriophage PRD1. These viral particles contain approximately 2000 protein subunits from 18 different protein species including integral membrane proteins associated with an internal lipid bilayer. Their crystal structure reveals, among other things, an ordered membrane structure that allows specific interactions with the genome and the coat proteins. John Johnson (The Scripps Research Institute) then closed the workshop with a lively talk about structure-based studies of auto-catalytic chemistry in virus particles. He described auto-catalytic cleavages that occur in the maturation of non-enveloped RNA insect viruses and proposed mechanisms based on atomic models from crystallography. The talk concluded with the description of a unique auto-catalytic ligation of a lysine side chain with an asparagine side chain to create catenated subunit rings that “chain-link” the capsid of HK97, a dsDNA bacteriophage.

—Marc Allaire

## Grazing Incidence Small X-ray Scattering Workshop

May 17, 2004

A workshop on Grazing Incidence Small-Angle X-ray Scattering was held on May 17, 2004 as part of the 2004 National Synchrotron Light Source (NSLS) Users Meeting. This technique, commonly referred to by the acronym GISAXS, is the surface analogue of Small-Angle X-ray Scattering (SAXS). Scientific topics discussed included thin polymer films, nanoparticles at interfaces, and semiconductor nanostructures. GISAXS measurements are sensitive to both the surface morphology and the internal structure of films, and provide information both lateral and normal to the surface on length scales extending from 1-100 nm. As a result, GISAXS provides an excellent complement to more conventional nanoscale structural probes such as atomic force microscopy and transmission electron microscopy. Moreover, GISAXS lends itself to in-situ and real-time studies. Eleven speakers presented their results, followed by a discussion on ways to improve the technique and access to GISAXS facilities.

The first speaker was Detlef Smilgies from the Cornell High Energy Synchrotron Source (CHESS). Detlef presented a short introduction on the history and applications of GISAXS. He reviewed the rapid development of the technique, starting with the pioneering measurements of Levine and coworkers in the U.S. and Naudon and coworkers in France during the late 1980s. Detlef provided a perspective on how three different x-ray communities, SAXS, diffuse

reflectivity, and Grazing Incidence Diffraction (GID), were converging through GISAXS. In the following talk, a youthful Sunil Sinha from the University of California presented an intriguing and mesmerizing talk on the underlying scattering theory associated with GISAXS with a focus on the Distorted Wave Born Approximation (DWBA). Examples were presented in which wave-guiding effects, induced by the film interfaces, significantly enhanced the small-angle scattering from the particulate matter inside or on the surface of the film. In the third presentation, Ian Robinson from the University of Illinois presented a stimulating talk on coherent GISAXS investigations of granular micro-structures in thin metal films, performed at the Advanced Photon Source (APS). Speckled diffraction patterns were reported for gold nanoparticles prepared by the dewetting of a thin gold film on a solid support. The shapes of the speckle patterns were used to investigate the changing geometry associated with different parts of the diffraction pattern.

After a short break, Till Hartmut Metzger from the European Synchrotron Radiation Facility (ESRF) presented a captivating review on combined GISAXS and GID studies of semiconducting nanostructures, so-called quantum dots, performed at HASYLAB and the ESRF. Particular emphasis was placed on the role of strain, size, and chemical composition and their relationship to growth, which are the crucial input parameters for the understanding the electronic and optical properties of the quantum dots. Specific examples included InAs islands on GaAs and Ge pyramids on Si, for which detailed maps of strain and composition within the quantum dots were obtained. The following speaker, Alain Gibaud from the Université du Maine, presented a visually stimulating talk on *in-situ* studies of surfactant-templated silica thin films at the NSLS, which included the premier of several “reciprocal space” movies. In these studies, time resolved GISAXS measurements were carried out simultaneously with gravimetric studies during the slow evaporation of ethanol from a film containing surfactant (CTAB), silica precursor (TEOS), and water to obtain silica mesostructures. The time evolution was exploited to probe the mechanism of the self-assembly process. Prior to lunch, Thomas Russell from the University of Massachusetts gave a lively and animated presentation on the assembly of nanoparticles at the interface of two im-



Grazing Incidence Small Angle Scattering Workshop attendees

miscible fluids. Tom noted that the nanoparticles mediate the interactions between the two fluids, thereby reducing the interfacial energy.

After lunch, Christine Papadakis from the Technical University of Munich awoke the audience with an enlightening presentation on the inner structure of lamellar diblock copolymer thin films, as studied at CHESS and ESRF. Christine showed how poly(styrene-*b*-butadiene) films undergo a morphological transition from parallel lamellae for short chains to perpendicular lamellae for long chains. Complementary *in-situ* time-resolved GISAXS measurements were presented after the injection of toluene into the sample cell, which revealed both changes in the sample thickness and the lamella orientation on a timescale of minutes. Continuing the polymer theme, Matthew Misner from Prof. Russell's group at the University of Massachusetts presented an informative talk on real-time studies of block copolymer thin films at the NSLS. Results for two different systems were examined: polystyrene-block-poly(ethylene oxide) diblock copolymers, where the orientation of the micro domains was normal to the surface, and poly(ethylene-*alt*-propylene-*b*-lactic acid), where the orientation was parallel to the substrate. Analysis of the time-resolved GISAXS provided information on the size, morphology, and orientation of the films during solvent evaporation. In the final polymer talk, Phong Du, a student from Cornell University working with Prof. Ober and Prof. Wiesner, presented an intriguing overview of their GISAXS studies at the CHESS. Poly(styrene-*b*-ethylene oxide) structures were exploited to create silica nanostructures, making use of the silica precursor being readily concentrated in the poly(ethylene oxide) block and successive calcination. In a second project, nanosieves were prepared from cylindrical diblock copolymer films by selective photoreactions, in order to remove one block, while cross-linking the other for stability. To close out the session, Oleg Gang from Brookhaven National Laboratory presented a fascinating talk on liquid films on nano-sculptured surfaces. By varying the chemical potential difference between the liquid-vapor coexistence, small nanometer pits were filled with an organic liquid. By combining GISAXS and x-ray reflectivity at the NSLS, detailed information could be obtained on both the liquid in the pits and the thin wetting film above the surface.

After a short break, Gilles Renaud from the Commissariat à l'Énergie Atomique (CEA) in Grenoble an inspiring whirl wind presentation of their work on real-time *in-situ* investigations of the morphology, organization, and internal structure of growing metal nanoparticles on oxide surfaces in ultra-high vacuum at ESRF. Results were presented for palladium, silver, and platinum on MgO(001) as well as for copper on alumina, gold on TiO<sub>2</sub>, and copper and silver on ZnO. Combined GISAXS and wide-

angle x-ray scattering provided a wealth of information on the growth modes and particle morphology. Quantitative information could be extracted by theoretical modeling within the framework of DWBA. In the final presentation, Jin Wang from Argonne National Laboratory gave a thought-provoking talk on the kinetics of nanocomposites obtained from both SAXS and GISAXS measurements at the APS. Jin showed that the motion of the nanoparticles is highly anisotropic.

Following the talks there was an enthusiastic discussion on the future of GISAXS methods, including the needs of the emerging community. There was wide agreement that one of the key features of GISAXS is the ability to carry out real-time, in-situ measurements. While there are currently few dedicated GISAXS beamlines, GISAXS capabilities can often be implemented on existing SAXS or GID beamlines through the addition of a 2D detector. There was a lively discussion on CCD detectors including the need for faster readout times. For soft matter applications, it was noted that it is often desirable to combine GISAXS with reflectivity and SAXS measurements. Simultaneous optical measurements of the film thickness can be very useful for in-situ experiments. For hard-matter applications, it was noted that it is essential to combine GISAXS measurements with GID studies, in order to obtain shape and internal structural information. Finally, it was remarked that future GISAXS facilities should be user-friendly and well supported in order to open up the technique to non-specialists.

—Ben Ocko and Detlef Smilgies

## Pharmaceutical Applications of Synchrotron Radiation Workshop

May 17, 2004

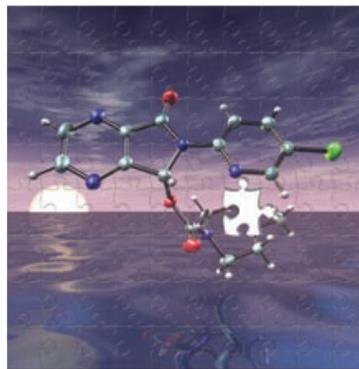
The workshop “Pharmaceutical Applications of Synchrotron Radiation” was held as part of the 2004 Annual Users’ Meeting of the National Synchrotron Light Source (NSLS) on May 17<sup>th</sup>. The main goal was to bring together people working in the pharmaceutical industry with those familiar with synchrotron radiation techniques. The workshop focused on issues related to later stages of the drug development cycle, i.e., polymorphism, selection of salts and hydrates, quantification of crystallinity, partial states of crystallinity, etc. Research in drug discovery, such as protein crystallography, was not covered, although that has grown to a very large user base at synchrotron facilities worldwide.

One of the co-organizers, Evgenyi Shalaev of Pfizer, opened the discussion with an overview of the drug development cycle. Of several million candidates screened, only one or two will be reduced to a profitable new drug, after a development period of approximately fifteen years. X-ray

techniques are crucial to many of the steps along the way, as well as the protection of intellectual property during the marketing lifetime of the drug.

Peter Stephens of Stony Brook University and the NSLS (also a workshop co-organizer) discussed the differences between laboratory and synchrotron x-ray sources, first from the standpoints of source properties and later in terms of access issues. He showed several comparisons of laboratory vs. synchrotron powder diffraction patterns and concluded that the additional information available was often dramatic in its ability to solve technical problems. He also emphasized that synchrotron sources are strongly motivated to attract new users, and while there may be some barriers to a new user getting a synchrotron research program started, they are generally fewer than popularly assumed, and the rewards are substantial.

Bill David and Kenneth Shankland, both of Rutherford Appleton Laboratory (UK), discussed many aspects of structure determination of small molecules from powder x-ray data. David emphasized the value of detailed structural crystallography, illustrated with the structure determination of a transient phase that existed for only one minute during the hydration of paracetamol (acetaminophen). He also showed the remarkable detail of information available, primarily through changes in diffraction peak shapes, of the transformation process in a dehydration reaction of the hypnotic zopiclone (see **figure below**). Shankland discussed the importance of various steps taken in experimental design and data collection to ensure the accurate solution of structures, emphasizing that these techniques, both experimental data collection and analysis through simulated annealing methods, are widely available to those who care to use them. His message is that you can solve, from powder diffraction, structures that represent the typical complexity



The molecular conformation of the hypnotic drug zopiclone in its monoclinic anhydrous form, solved from powder diffraction data taken at ESRF BM16. The puzzle alluded to in the illustration, that of significant batch-to-batch variation in the physical properties of commercially prepared zopiclone tablets, was solved only when XRPD was brought to bear. The experiments revealed the structural relationships between the previously unsolved monoclinic anhydrous form, the monoclinic dihydrate form, and the stable orthorhombic anhydrous form. Picture courtesy of Alastair Florence and Kenneth Shankland.



Pharmaceutical Applications of Synchrotron Radiation Workshop attendees

of most pharmaceutical compounds.

Mike Pikal of the University of Connecticut discussed the degradation of pharmaceutical compounds, such as freeze-dried proteins in the form of glassy solids. The stability of pharmaceutical solids can be extremely sensitive to formulation, and the relationship between thermodynamic measurements and structural relaxation is an important topic of research. The current state of understanding is largely empirical, and the development of other methods to characterize the state of glassy solids is an important challenge.

Shalaev discussed the nature of the disordered states, such as amorphous states, and crystalline mesophases, of pharmaceutically relevant solids. Amorphous solids can have advantages in solubility and bioavailability, but disadvantages in stability; amorphous phases can also arise during certain standard processing techniques such as milling, drying, and compaction. He challenged the audience to help to clarify the possible existence of more than one amorphous phase of a given system, so-called polyamorphism, and the extent to which nominally amorphous solids, e.g., protein/sugar mixtures, may be heterogeneous. Another important issue is the relationship between local structure of amorphous solids and the structure of crystalline phases of the same compounds.

The third co-organizer, Raj Suryanarayanan of the University of Minnesota, discussed x-ray measurements of the degree of crystallinity of pharmaceuticals, especially from the standpoint of monitoring the physical instability of products that are prepared as non-crystalline phases. He discussed *in situ* measurements of the crystallization of amorphous sucrose, performed with synchrotron radiation and an area detector. These experiments avoid the difficulties associated with the preparation of a large number of mixtures required for the traditional preparation of a calibration curve.

Satyendra Kumar, of Kent State University, discussed model systems related to the issue of delivering water-insoluble drugs. Such materials may be physically encapsulated in micelles or at the level of individual molecules, which then

have interesting mesophases. He reported on synchrotron x-ray scattering studies of aqueous solutions of cyclodextrin and small molecule dyes, which have a variety of self-organized structures with varying degrees of orientational and structural order. He demonstrated how the use of external electric, magnetic, or mechanical (shear) fields can reveal important new structural details that otherwise cannot be obtained.

The final talk of the session was given by Heinz Amenitsch of the Austrian Institute of Biophysics and X-ray Structure Research outstation at the ELETTRA synchrotron source in Trieste. His topic was the use of simultaneous small- and wide-angle x-ray scattering to study nanostructural features of relevant solids. The size of domains of the different components of a pharmaceutical formulation, the nature of the internal surfaces, and the degree of dispersion are all accessible to these techniques. He showed that these measurements can be made with sub-millisecond time scales, allowing real-time study of the relevant structural changes during such processes as spray-drying.

At the end of the workshop, Bruno Hancock of Pfizer moderated a round-table discussion of collaboration between pharmaceutical scientists and facilities such as the NSLS. Some participants expressed concerns that the federal government might claim ownership of intellectual property derived from experiments at synchrotron sources, even if the user follows procedures for proprietary access. Obviously, this is a matter to be addressed by lawyers, not scientists, but the large investment in protein crystallography by major drug companies at synchrotron sources indicates that this might not be a serious impediment to the commercial use of these facilities. Participants who are not experienced with synchrotron radiation as a research tool probably came away thinking of synchrotron sources as valuable commodities, not heroic investments. And synchrotron experts should have seen that their expertise may be of real value to some important practical problems.

—Peter Stephens

## Advanced Optical Systems and Metrology for High Power and Coherent Beamlines Workshop

May 19, 2004

Future light source beamline instrumentation will challenge users and instrument designers to take full advantage of the brightness and coherence of the x-rays delivered by the new sources. Since Brookhaven National

Laboratory has a strong tradition in the development of metrology instrumentation in support of synchrotron radiation optics, it is appropriate that this session on optical systems and metrology reviewed the current state-of-the-art in optical technology and metrology, and explored avenues that may lead to improved capabilities for the future.

Chris Jacobsen from Stony Brook University, who has extensive experience in the generation and use of coherent x-rays with existing sources, provided a summary of the capabilities of various groups around the world to produce small spot sizes from present sources of coherent x-rays. A Japanese group at SPring8 has demonstrated 90nm spot sizes with Kirkpatrick-Baez focusing optics, and 15nm line spacing in zone plates have been reported by workers at the Center for X-ray Optics at Berkeley.

Following Jacobsen's introduction to coherent imaging at x-ray wavelengths, the workshop heard from two speakers who are primarily involved with normal incidence UV and visible optics for large programs at Lawrence Livermore National Laboratory (LLNL) and at NASA Marshall Space Flight Center. The surface figure and finish requirements on normal incidence optics for DUV (Deep Ultraviolet) and EUV (Extreme Ultraviolet) lithography and for large space telescopes are nearly the same as those for grazing incidence optics, so it was appropriate to hear from John S. Taylor of LLNL and from Phil Stahl of NASA about mirror fabrication and metrology technologies that are being supported by these projects. Taylor discussed the development of diffraction-limited multilayer-coated optics for normal incidence EUV lithography systems that have been developed over the past several years by the Virtual National Laboratory consortium of three national laboratories and three companies involved in developing tools for the semiconductor manufacturing industry. Slope errors on these optical components are in the 200 nrad range, which is where synchrotron optics will need to be for effective utilization of the next generation of high-brightness x-ray sources. Stahl gave an overview of future NASA space telescope missions and the technology that will be required



Advanced Optical Systems and Metrology for High Power and Coherent Beamlines Workshop attendees

to build large, lightweight mirrors and structures. Over the past few years he has organized a symposium, "NASA Tech Days," for NASA contractors and university grant recipients to present progress in technology development programs funded by government agencies. Of interest to the Synchrotron Radiation (SR) community are the various materials-development projects investigating the use of Be, SiC, and other ceramic and composite materials as high power mirror substrates, and the fabrication technologies that accompany these new materials.

Don Golini from QED Technologies in Rochester, NY, gave a presentation on a new surface finishing technique that looks quite promising as an alternative to conventional pitch polishing for the production of spherical and aspherical surfaces. His company has developed various polishing machines that use magnetorheological fluids as the polishing medium. These fluids change viscosity by orders of magnitude when a magnetic field is applied and can conform to exotic surface shapes not possible with hard laps, such as ellipsoids and toroids. A great deal of research has gone into characterizing the polishing process, which is highly deterministic. With the appropriate metrology, it is possible to quickly correct for figure errors and converge rapidly to the desired surface shape.

Refractive optics for focusing x-ray beams are a new addition to the techniques available to users for producing small spot sizes. Ken Evans-Lutterodt (National Synchrotron Light Source) gave an overview of the subject and discussed the production of the optimum elliptical hole shape with an e-beam writer and the use of diamond as a substrate for high heat load situations. Ali Khounsary, Argonne National Laboratory, followed with a discussion of high heat load optics. He emphasized the need for a systematic approach to the design of optical systems that incorporate knowledge of the source parameters, mirror substrate characteristics, mechanical design, and testing.

Following the lunch break, Peter Takacs (BNL/ Instrumentation) discussed metrology issues related to the Long Trace Profiler (LTP). The LTP is widely used to measure the surface quality of large aspheric x-ray optics. In order to reach the 100 nrad measurement accuracy level, improvements are needed in the quality of the internal optical components in the system. Custom-made glass prisms with superpolished surfaces will be needed to reduce the systematic error to below the current 1  $\mu$ rad level. Wayne McKinney (Advanced Light Source, Lawrence Berkeley National Laboratory) followed with a discussion of issues that are driving the direction of SR metrology. Although the LTP has served well in the past, other alternative metrology techniques need to be explored, such as stitching interferometry, Hartmann sensors, and multi-beam autocollimator techniques such as those proposed by Polack's group at SOLEIL in France and by Weingärtner's group at the

Physikalisch-Technische Bundesanstalt (PTB) in Braunschweig, Germany.

The final session of the day began with a description of Kirkpatrick-Baez (KB) mirror nanofocusing systems by Gene Ice from Oak Ridge National Laboratory. The practical limit to the focal spot size is in the range of 10 to 30 nm, which can be produced by highly demagnifying (~1000:1) elliptical cylinders. These mirrors need to have slope error tolerances of less than 200 nrad in order to perform correctly. The technique of differential deposition, which he has pioneered, appears to be a promising tool for converting cylindrical surfaces into accurate elliptical shapes.

The final two talks were presented jointly by Riccardo Signorato (ACCEL, France) and Daniel Hausermann (Advanced Photon Source, Argonne National Laboratory) as they related to the fabrication and use of modular piezoelectric bimorph mirrors as adaptive optics. The piezoelectric bimorph has been developed by Signorato and is currently in use at a number of sites, including beamlines at the APS. Hausermann uses a pair of bimorphs in a KB configuration to focus a 500 $\mu\text{m}$  x 500 $\mu\text{m}$  hard x-ray beam down to a 10  $\mu\text{m}$  spot. The longest size currently available is 1050 mm in length with N=33 actuator segments. Signorato is looking into adding a Shack-Hartmann wavefront analyzer to the system to enable automated closed-loop focus control for the mirror.

Following the final talk, a quick tour of the Optical Metrology Laboratory in the NSLS Instrumentation Division was offered to those participants who remained. The workshop was successful in bringing together members of several different communities, including conventional optics fabricators, end users, and metrology specialists, to share thoughts and ideas on the current state of optical component technology, and to chart a course toward future capabilities.

—Peter Z. Takacs and Steven Hulbert

## Applications of Synchrotron-Based Methods to Hydrogen Storage Materials Workshop

May 19, 2004

A workshop on “Applications of Synchrotron-based Methods to Hydrogen Storage Materials” was held in conjunction with the 2004 National Synchrotron Light Source (NSLS) Users’ Meeting on May 19, 2004. The purpose of the workshop was to bring together scientists who synthesize materials with applications in hydrogen storage and scientists with expertise in the application of synchrotron radiation methods, who currently study these materials.

The workshop was opened by Doon Gibbs, the Associ-



Applications of Synchrotron Based Methods to Hydrogen Storage Materials attendees

ate Laboratory Director for Basic Energy Sciences at Brookhaven National Laboratory (BNL). He emphasized the importance of the Hydrogen Energy Project (production, storage, utilization) as one of the missions of the Department of Energy (DOE), and encouraged collaborations between scientists within BNL and with outside groups by utilizing facilities like the NSLS and the Center for Functional Nanomaterials (CFN) for research.

John Petrovic, from the Hydrogen Storage Team in the DOE Office of Hydrogen, Fuel Cells, and Infrastructure Technology, described in his talk titled “The DOE National Hydrogen Storage Project: Addressing Key Performance Targets for Materials-based Hydrogen Storage Systems” the targets for automotive research over the next 15 years, and the funding efforts by the DOE to reach these targets. He presented different projects and Centers of excellence that actively work in the field of hydrogen storage and that pursue different paths trying to create a successful system. Currently, none of the proposed hydrogen storage systems achieves the proposed targets for 2005!

James Reilly from the Department of Energy Science and Technology at BNL described in his talk “The Application of Hydrogen Driven Metallurgical Reactions to Prepare Reactive Nanoscale/Nanocomposite Materials” several ways to create nano-composite metal-hydrides, and discussed their properties. By doping some materials with fairly large amounts of different metals, the storage capacity can be increased, and the temperature for release is lowered.

Zafar Iqbal of the Department of Chemistry and Environmental Science at the New Jersey Institute of Technology talked about “Electrochemically-Induced Hydrogen Storage in Metal-Functionalized Carbon Nanotubes.” He described different ways to synthesize single-wall nanotubes (SWNTs), ways to functionalize the SWNT with metals, and their properties. Experimental investigation with Raman and Fourier Transform Infrared (FTIR) spectroscopy indicate a chemisorption process in Co-

doped SWNT, while (weaker) physisorption is present in undoped nanotubes.

Tom Vogt of the CFN talked about “Hydrogen Storage and Nano-Structure.” He emphasized the importance of clearly characterizing the materials to better understand their storage and release mechanisms. High-brightness synchrotron radiation sources like the proposed NSLS-II are ideal sources for real-space and dynamical imaging. Also, probes at the CFN, such as proximal probes, are well suited for studying SWNTs, for example.

Alexander Ignatov of the Department of Physics at the New Jersey Institute of Technology presented in his talk “X-ray Absorption Studies of Hydrogen Storage Materials – Strength and Limitations” experimental results of EXAFS and XANES experiments studying the Ti K-edge in Ti-doped  $\text{NaAlH}_4$ . The data indicate that Ti does not substitute Na in the lattice. Instead, amorphous  $\text{TiAl}_3$  is formed, and H is found in the first coordination sphere about Ti. The EXAFS-analysis allows extraction of the position of H, but the H coordination numbers have large errors.

Yan Gao from the GE Global Research Center talked about “Characterization of  $\text{NaAlH}_4$  by High-Pressure X-ray Diffraction and in-situ EXAFS.” He presented the first powder-diffraction data of the re-hydrogenation process measured with high time resolution. The data clearly indicate that two steps in the dehydrogenation and re-hydrogenation process have to be completed individually before the next step can start. He also observed an increase in particle size, which slows down the reactions. His Ti XAS-data also indicate the formation of  $\text{TiAl}_3$ , and the growth of  $\text{TiAl}_3$ -crystals with the number of cycles.

The final speaker, Najeh Jisrawi from the Department of Physics and Astronomy at Rutgers University, discussed “Synchrotron XRD Studies of Hydrogen Absorption in Metallic Multilayers and Nano-Particles.” He applied surface x-ray diffraction to study charge-discharge curves of Pd and Nb/Pd/Nb thin films, and of polyhedral clusters with magic-number sizes. Molecular dynamics calculations support his experimental results and demonstrate that a cluster with a size of 8 nm shows similar behavior to a thin film.

The workshop was concluded with a brief discussion of techniques and equipment that are required for further successful studies of hydrogen storage materials at the NSLS.

—Wolfgang Caliebe and Trevor Tyson

## Nanoprobes for Nanosciences Workshop

May 19, 2004

A workshop on synchrotron-based nanoprobes was held on May 19<sup>th</sup>, 2004 as part of the National Synchrotron Light



Nanoprobes for Nanosciences Workshop attendees

Source (NSLS) and Center for Functional Nanomaterials (CFN) annual users’ meetings. The goal of the workshop was to explore the scientific opportunities offered by synchrotron-based nanoprobes, with emphasis on combined low-energy/photo-electron microscopy (LEEM/PEEM), soft x-ray microscopy, and x-ray micro-diffraction.

Bob Hwang, director of the CFN at Brookhaven National Laboratory (BNL), opened the workshop by giving an overview talk about the status of the CFN, current CFN capabilities that are open for users, including scanning probes, transmission electron microscopy (TEM) and NSLS beamlines, and the plan to establish close collaborations with other BNL research departments. He also outlined the vision to provide the nanoscience user community 24/7 access to a broad range of “off the shelf” instruments, as well as develop new and novel characterization methods in conjunction with partner users to achieve a more efficient transfer of information and technology.

After the introduction, the workshop was divided into three dedicated sessions. In the soft x-ray microscopy session, Harald Ade from North Carolina State University presented an extensive review of his work on the characterization of polymers using soft x-ray transmission microscopy (XTM). The chemical sensitivity in near edge x-ray absorption fine structure (NEXAFS) and linear dichroism are exploited to provide a unique contrast mechanism to discriminate different components in the sample as well as characterize the crystallinity and morphology of the sample. The second speaker of the session was Chris Jacobsen from Stony Brook University. Chris first gave an excellent review of x-ray microscopy using zone-plates as well as the advances made in recent years in the fabrication of high-resolution x-ray zone-plates. In the second half of his talk, Chris talked about diffraction imaging, a new technique that has attracted a great deal of attention recently because the possibility of achieving higher spatial resolution than that of zone-plate-based x-ray microscopy. The key in diffraction imaging is, of course, recovering the phase information that is lost in the measurement. Chris gave a detailed account of a very efficient iterative algorithm recently developed.

After a short break, Cev Noyan from IBM and Columbia University started the session on hard x-ray micro-spectroscopy and micro-diffraction. Cev presented a study of strain in SiGe crystals grown on Si crystals. It was a very nice demonstration of the power of micro-diffraction. In this case, he was able to map out the strain in both the SiGe layer as well as that of the substrate. He also discussed in some length the challenges in finding and focusing on a single grain using x-ray microbeams. The second talk in the session was given by Barry Lai from the Advance Photon Source (APS) at Argonne National Laboratory. Barry talked about the applications of micro-fluorescence and x-ray absorption near edge spectroscopy (XANES) spectroscopy to life science problems, in particular the role of trace amounts of metals and metal oxides in immunology and intracellular processes. At the 2ID at the APS, x-rays down to 200 nm x 200 nm, with intensities up to  $2 \times 10^9$  ph/sec, are achieved using a hard x-ray zone-plate. These hard x-ray zone-plates are extremely difficult to produce because of the narrow width and the height/width aspect ratio required. Barry also shared his experience in how to accurately align optics and position samples, a very challenging problem as the spatial resolution of the experiment improves to sub-micron level.

The third speaker of the session was Ken Evans-Lutterodt of NSLS. Ken gave an overview of the microdiffraction project at the X13B beamline at the NSLS. The project is funded by the Department of Energy to serve the increasing needs of nanoscience users. The instrument, taking advantage of the small source size at the X13 straight section of the ring, is designed to perform sub-micron x-ray micro-diffraction and imaging. It has the unique capability of allowing experimenters to choose the focusing optics to optimize their measurement. The beamline will also serve as a test bench for new emerging focusing optics and x-ray imaging techniques.

The third session, focused on LEEM/PEEM, began after lunch. Rudolph Tromp from IBM gave a broad overview of the history, current state-of-the-art, and future possibilities of this powerful technique. For example, structural information with 4 nm spatial resolution can be achieved with LEEM today. There are also a variety of contrast mechanisms that can be exploited in LEEM, for example, using bright and dark field imaging, as well as work function and electron energy dependence. A LEEM instrument can also be combined with a photon source to perform PEEM to obtain chemical contrast. The potential of combining the structural information from LEEM and chemical information from PEEM is extremely exciting. Finally, by using a spin-polarized electron source, LEEM can be used to study magnetic surfaces and interfaces (SPLEEM). Time-resolved study of magnetization dynamics has also been demonstrated. Stefan Heun from ELETTRA Trieste/Italy presented the capability and results from the X-PEEM instrument located at Sincrotrone Trieste. The unique feature of the instru-

ment is the energy filtering of photoelectrons. It enhances the chemical sensitivity significantly. The session ended with a talk on the next generation of PEEM developed at the Advanced Light Source by Jun Feng from Lawrence Berkeley National Laboratory (LBNL). Jun talked about the design of the PEEM3 project at LBNL. The goal of PEEM3 is to achieve spatial resolution down to 5 nm, almost an order of magnitude better than the PEEMs currently operating at various synchrotron facilities. Jun discussed in detail the critically important aberration correction system, including an electron mirror aberration corrector and an aberration-free magnetic beam separator.

Following the three focused sessions, there was a lively discussion period, chaired by Peter Sutter of CFN/BNL, Ken Evans-Lutterodt, and Chris Jacobsen. These valuable ideas will be taken into consideration in the design of the new LEEM/PEEM end station and for the future upgrade of the x-ray microscopy programs at the NSLS.

—Cecilia Sanchez-Hanke

## Crystallization: Focus on Membrane Proteins Workshop

May 19-20, 2004

The Crystallization workshop organized in conjunction with the 2004 National Synchrotron Light Source Annual Users' Meeting focused this year on crystallization techniques available to membrane proteins. This two-day workshop covered different crystallization methods from the most standard vapor diffusion to more advanced techniques. A morning of introductory talks was followed by five practical sessions, of two hours each, where participants had the opportunity to carry out different crystallization methods in the laboratory. The main purpose of this workshop was to bring the practical aspects of the crystallization methods to the participants, the exchange of experiences and ideas being its essence.

Naomi Chayen, Imperial College London, in her talk "Tackling the bottleneck of protein crystallization: Practi-



Crystallization: Focus on Membrane Proteins Workshop attendees

cal techniques with a difference,” introduced the phase diagram and discussed the several different crystallization techniques developed for soluble proteins and the necessary modifications for crystallization of membrane proteins. The laboratory practical sessions included setting up microbatch and vapour diffusion trials using oils as a tool to aid crystal growth. Participants had the opportunity to try several different crystallization configurations.

Peter Nollert, deCODE Genetics, in his talk “Miniaturization of the cubic phase,” introduced the cubic lipid phase method and discussed the difficulties of membrane protein crystallization. The cubic lipid phase method was demonstrated in the laboratory and participants were able to set up crystallization trials for bacteriorhodopsin. The application to soluble proteins was also introduced. A number of participants tried the method with their own proteins with success.

Petra Fromme, Arizona State University, talked about “Overcoming the Crystallization Problems of PSI and PSII.” She discussed the importance of the phase diagram to the crystallization of PSI and PSII proteins. The contribution of the dialysis method was introduced and participants were able to assemble their own crystallization reactors. Most experiments lead to the crystallization of PSI within 24 hours and by the end of the workshop participants were able to harvest some of the crystals.

Marie Claude Marchand, NEXTAL Biotechnologies, in her presentation on “The Vapour Diffusion Method,” discussed the vapour diffusion method, as well as seeding, derivatization, and optimization of the phase diagram. Some participants were able to screen for the best crystallization conditions of their own protein.

Ana Belen Moradela Merlo, University of Granada, discussed “Protein Crystallization by the Counter,” diffusion, and introduced the counter diffusion method. Participants were able to set up crystallization trials of soluble proteins in the “Granada Box.” Possible applications to membrane proteins were discussed.

The demand for the course was overwhelming. It was oversubscribed by over 100%. At the end of the workshop students were asked to evaluate the course and suggest changes and additions; two thirds responded to the survey. Overall the course was rated as extremely useful as reflected in one of the answers to the survey: “I learned a great deal about crystallization methods. Most importantly I now understand the more practical aspects of each method that I was not aware of. These ideas cannot be gained from other formats.” All participants who answered the survey thought that the workshop should be offered again.

—Vivian Stojanoff and Naomi Chayen

## UEC Community Service Award Presented to Sue Wirick

May 20, 2004

The National Synchrotron Light Source Users’ Executive Committee (UEC) presented the 2004 UEC Community Service Award to Sue Wirick of Stony Brook University’s Physics Department. Sue is a beamline scientist for the X1A insertion device beamline. This award is given for service, innovation, and dedication to users of the NSLS, and she is well deserving of that honor.

Members of the NSLS user community nominated Sue for this award. Here are some of the comments users made about her wonderful contributions:



UEC Chair Tony Lanzirotti presents the award to Sue Wirick.

- “Since joining the X1A effort more than a decade ago, Sue Wirick has played a central role in the development of the scientific program and in supporting user access to the spectromicroscopy facilities. Quite simply put, she is the key to outside users’ ability to carry out experiments.”
- “Our research team owes very much to the assistance that Sue Wirick was able to give us. She was available day and night, gave excellent advice, steered us through the experimental process with ease, and provided a positive and friendly environment that made it a pleasure to work at the beamline.”
- “Sue Wirick actively participates in meetings of the user community to improve the user support at the NSLS. She contributed heavily in the past to making the annual user meeting a success by helping in the planning and chairing of sessions. Sue is known by users for helping with technical problems even for beamlines that are not her responsibility.”
- “Sue Wirick has been a godsend to the NSLS community. She is one of the rare people who will help anyone, including the most difficult personalities, when they have

a problem. She will do this anytime, even on weekends and evenings. She is the person that I recommend first to outside users who want to find out more about NSLS and Brookhaven. She always provides detailed and concise advice on instrumental capabilities, and also on the more mundane aspects of how to get beam time, availability, etc.”

Tony Lanzirotti, the Chair of the UEC, presented the award to Sue at the NSLS Users' Meeting banquet on the evening of Tuesday, May 18th. Sue received a \$250 gift certificate and her name was engraved on the plaque on display in the NSLS lobby. Congratulations Sue and well done!

—Tony Lanzirotti

## The PASS System Arrives

June 23, 2004

We are very happy to announce the arrival of the new Proposal, Access, Safety and Scheduling (PASS) system, which debuted in May for the submission of general user proposals for the fall cycle.

### PASS System

Proposal Allocation Safety Scheduling

PASS was developed to facilitate the submission and review of general user and proprietary proposals and the allocation and scheduling of beamtime at the NSLS. To meet DOE reporting requirements and to ensure safety compliance, the safety approval form is also incorporated into the system.

The first phase of development (proposal submission, safety approval, and the scheduling of general user proposals) is complete and online. Proposals (and Px Forms when appropriate) are submitted online in one system and routed for beamline review for feasibility and safety purposes. PASS then routes the form to proposal review panel (PRP) members for peer review and ratings, to allocation panel members for beamtime allocations, to NSLS safety staff for review and approval, and finally for beamtime scheduling. Each principal investigator and reviewer is granted individual access according to permissions assigned.

Instructions and frequently asked questions are provided throughout the system to ensure user friendliness and accessibility. The production version will soon include a link to send comments or report functionality issues or problems.

Further development will take place over the next few months to integrate proprietary proposals, safety approval for Participating Research Team (PRT) experiments, to further develop scheduling for all experiments, and to add

more functionality and capabilities. The final phase will incorporate a reduced lead-time to provide for rapid access.

Some proposal process changes introduced in the PASS system are:

- PASS randomly assigns each proposal to three PRP reviewers who have reviewed the lowest number of proposals for the given cycle to date. This ensures that all reviewers review approximately the same number of proposals and that members are not overburdened with requests, and plans are in place to increase the pool of reviewers. PASS does not assign proposals to a PRP member whose institution is the same as the PI.
- PASS notifies PRP members by email immediately after submission of the proposal by the PI, allowing reviews to take place as soon as the PRP member is able.
- PRP reviews are completed independently of other PRP members and all ratings are averaged for a final rating.
- Lifetime days are no longer requested. The PI requests the number of days needed in a given cycle, and this is the information that is vital for review, allocation and scheduling.
- Penalties for canceling beamtime after allocation have been eliminated. Our goal is to re-assign canceled beam time to other users who were not allocated due to high demand.
- The one-half point credit given for one cycle to lower-rated proposals not allocated beam time is no longer given. Alternatives available are for the PI to submit a new proposal based on an existing one and to thereby address concerns or questions that were raised by reviewers in the previously submitted proposal. Another planned capability is to provide PRTs the ability to offer PRT beamtime to rated proposals that did not receive an allocation of beam time since higher-rated proposals filled up the general user obligation for that beamline.
- PIs must assign a lead experimenter to serve as lead person for the experimental team while present on the NSLS experimental floor, who will in turn receive an email with links to his/her roles and responsibilities.

—Mary Anne Corwin