NSLS' Youngest Scientists Learn from Light on "Take Our Daughters and Sons to Work" Day

April 24, 2003

On April 24, about 30 daughters and sons learned about some of the scientific programs at the NSLS, and even performed their own scientific experiments. The one-day visit was part of the national "Take our Daughters and Sons to Work Day."

At the NSLS, the children learned that the facility produces many types of light, from microwaves to x-rays, which have many applications in many fields, including electronics, catalysis, microscopes, and medicine. NSLS scientists Marc Allaire, Steve Hulbert, Lisa Miller, and Vivian Stojanoff offered a tour of the experimental floor to the boys and girls, who discovered how synchrotron light is used to design non-stick coatings for aluminum pans, study bone diseases like osteoporosis, and develop new drugs using protein crystallography.

After the tour, the daughters and sons had the chance to perform their own scientific experiments. Marc Allaire demonstrated simple reflection of light from a mirror and contrasted that with the process of diffraction, which was illustrated by reflecting red laser light from a CD-ROM -- the world's most popular diffraction grating. But perhaps the most exciting moment was when the boys and girls discovered that they could created their own rainbow patterns by diffracting visible white light from the CD-ROM.

The boys and girls then had the opportunity to learn from Lisa Miller about the wonders of liquid nitrogen. By immersing an inflated balloon in liquid nitrogen, they discovered that the air inside of the balloon contracts, and then re-expands when warmed up. Much to the amazement of the entire crowd, the balloon survived dozens of repeated freeze-thaw cycles without bursting. But perhaps one of the most memorable experiments involved freezing natural versus artificial daffodils in liquid nitrogen. Both the children and their parents learned that it is much more fun to freeze and crumble a living flower than to take it home as a souvenir.

— Lisa Miller

Simple reflection demonstrated with a flashlight, mirror, and a white board.

NSLS 2003 Annual Users' Meeting Highlights Scientific Successes, Exciting Future Plans

May 19-21, 2003

A spirit of optimism pervaded the 2003 annual meeting of National Synchrotron Light Source (NSLS) users, held at BNL May 19-21, 2003, with presentations on scientific successes and plans for new facilities.

About 30 sons and daughters visited the NSLS for "Take Our Sons & Daughters to Work" day.

"A lot of good things have happened at BNL in the last year," said Doon Gibbs, BNL's Interim Associate Laboratory Director for Basic Energy Sciences, as he welcomed NSLS users from around the country and the world to the Tuesday morning main meeting, chaired by Tony Lanzirotti of the University of Chicago, Chair-Elect of the Users' Executive Committee (UEC). Gibbs pointed out that BNL had made "great strides" toward establishing a new Center for Functional Nanomaterials (CFN) and toward significantly upgrading the NSLS. He also noted that several highly qualified people had been brought into BNL leadership positions, including Praveen Chaudhari, the new Laboratory Director.

Among speakers and attendees at the Annual National Synchrotron Light Source Users' Meeting are: (from left) Tony Lanzirotti, University of Chicago; Steven Dierker, BNL; Patricia Dehmer, DOE; Doon Gibbs, BNL; Pedro Montano, DOE; Peter Paul, BNL; Leemor Joshua-Tor, Cold Spring Harbor Laboratory; and Chi-Chang Kao, BNL.

In introducing Peter Paul, Deputy Director for Science & Technology, Gibbs also took the opportunity to thank Paul for his steadfast leadership as Interim Director during the past two years.

Paul, whose task was to give an overview

of BNL, echoed a statement made by Chaudhari at the previous week's RHIC & AGS Users' Meeting, that DOE program managers take a great risk when they build new facilities with the hope that users will come and do good science.

"Fortunately it has always seemed to work out, but we can't take it for granted," Paul said, emphasizing how important it is to have an active user community, such as that at the NSLS, to keep a facility strong. With such involved users and the new leadership at the Lab — including Chaudhari, Gibbs, James Misewich as Materials Science Department Chair, Robert Hwang as CFN Director, and Alex Harris as Chemistry Department Chair — "We are all set to move forward," Paul said.

After describing improvements in support services, housing, and other facilities for users, Paul spoke of the CFN, recent findings at the Relativistic Heavy Ion Collider, and the proposed NSLS-II, a third-generation light-source ring that would be the future center of synchrotron activity at BNL and in the Northeast. "The Laboratory will commit all the resources we can muster to make this a reality," he said.

Bob Hwang then presented details of the CFN, recognizing that "the current excitement in nanoscience is based on work that has been going on for decades at synchrotrons like the NSLS, and you, the users, are a big part of that." He asked NSLS users for help in shaping the new center, noting that the CFN, like DOE's four other nanoscience research centers, was co-located within an existing DOE research facility, in this case the NSLS, to build on existing strengths.

Like the NSLS, the CFN will be a user facility, with a similar process for reviewing proposals. With a range of complementary facilities focused on six scientific areas, the CFN will address the goal of tailoring materials' responses to achieve specific functionality based on an understanding of nanoscale phenomena.

Offering one example of what nanoscience might yield, Phaedon Avouris of IBM's T.J. Watson Research Center then gave the meeting's keynote address on "Carbon Nanotube Electronics."

With a break from science to focus on funding, UOP's Simon Bare, lobbying coordinator for the UEC, then urged all NSLS users to learn about the federal funding process and to get involved.

Users could help to "educate" their own legislators and the congressional committee members vital to science funding — via letters, phone calls, office visits, and even op-ed articles in newspapers — about the importance of research sponsored by DOE's Office of Science. Several bills that propose increased funding for the Office of Science are pending, he said, so to take action now is vital. For more information, see: http://www.nslsuec.org.

Robert Hwang (left), who is Director of the Lab's new Center for Functional Nanomaterials. Keynote speaker at the meeting was Phaedon Avouris (right) of IBM's T.J. Watson Research Center.

The meeting's next session was chaired by Ron Pindak, Head of Science Program Support for the NSLS. Patricia Dehmer, Associate Director of the Basic Energy Sciences (BES) within DOE's Office of Science, started the session. "After hearing this morning's talks," she said, "it strikes me that this is the beginning of a transition period for the Lab, and I'm very optimistic about the future of this institution."

Members of the 2003 planning committee for the NSLS Users' Meeting include: Ron Pindak, BNL; Annie Heroux, BNL; Dan Fischer, National Institute of Standards & Technology; Lisa Miller, BNL; Liz Flynn, BNL; Lydia Rogers, BNL; Mary Anne Corwin, BNL; Sue Wirick, Stony Brook University; and Tony Lanzirotti, University of Chicago.

Long-range planning within BES has resulted in a recommendation for a general upgrade to provide a full return on capital investments at existing light sources, Dehmer explained. Another recommendation was for the NSLS-II upgrade. "This rated very high," she said, encouraging the spontaneous applause that erupted, adding, "You can thank Steve [Dierker, NSLS Chair] for doing such a good job at the presentation."

Referring to the five DOE Nanoscale Science Research Centers, she said, "We are extremely happy that one of those is at Brookhaven. These [nanocenters] are

going to be a very, very important component of the BES family of facilities."

Dehmer then gave her "Totally Unsanctioned Safety Seminar," drawing partly from her own lab experience. The bottom line: "It is possible – and required – to run your laboratory safely, and Pat will become a pest [with investigations and possible cuts in funding] if you mess up."

Following Dehmer, Steve Dierker gave an overview of recent NSLS successes, including Roderick MacKinnon's "spectacular piece of work" on voltage-dependent potassium ion channels, featured on the cover of the May 1 issue of Nature; studies of materials that expand under pressure; and a paper on cell membrane fusion that explains "one of the most basis processes" of cell division. "This has been an action-packed year, with a lot of exciting developments," he said.

Dierker gave credit to the NSLS's support staff, saying, "None of these advances would have been possible if we could not deliver the photons to the end of the beamline. It takes a dedicated and talented staff and a determined effort to keep both rings running reliably."

Dierker then reviewed the many beamline and instrumentation improvements of the past year, and talked about the proposed NSLS-II.

This $400 million upgrade, featuring a new x-ray storage ring three times larger than the current NSLS, would be constructed on Brookhaven Avenue, across from the existing structure, featuring 21 superconducting undulator beamlines and providing the highest brightness of any existing light source, with much shorter pulses.

"Our goal is to build the ultimate medium-energy storage ring," Dierker said. "We would see a huge impact from these enhanced capabilities, especially in the areas of nanoscience and protein crystallography, as larger cells and smaller crystals could be analyzed."

The meeting continued with scientific talks on nanoscience, thin films, x-ray crystallography, and new x-ray sources. During the afternoon session chaired by Lisa Miller, Coordinator of the NSLS's Information & Outreach Office, the UEC Community Service Award was presented to Michael Sullivan, Chief Beamline Engineer for Albert Einstein College of Medicine, for service, innovation and dedication to NSLS users. The winners of the Student/Post Doc Poster Contest were also announced.

Poster prizewinners at the 2003 NSLS Users' Meeting are: (from left) Ally S.-Y. Chan, Rutgers University; David Linkous, George Mason University; Hidenori Tashiro, University of Florida; Henrik Loos, BNL; Daisuke Kawakami, Stony Brook University (SBU); and V.G. Alexandratos, SBU.

Users were then invited to hear more about the BNL nanocenter and encouraged to meet with CFN scientific and facility leaders before adjourning for the meeting's Western-theme banquet in Berkner Hall.

—Karen McNulty Walsh

Frontiers in Powder Diffraction Workshop

May 19, 2003

The theme of the workshop, "Frontiers in Powder Diffraction," held on May 19th at the 2003 NSLS Users' Meeting, was the growing practice and utility of powder diffraction and related techniques in a variety of contexts. Speakers covered work done with x-rays and neutrons, performed at the NSLS, Advanced Photon Source, European Synchrotron Radiation Facility, ISIS (spallation neutron source at Rutherford Appleton Lab, UK), the Intense Pulsed Neutron Source (Argonne National Lab), and the Institut Laue-Langevan, as well as laboratory x-ray instruments.

Enjoying the "Western" flavor banquet held at the 2003 NSLS Users' meeting are the NSLS User Office staff, past and present: (standing, from left) Gretchen Cisco, Eileen Pinkston, Susan Hatzel, Liz Flynn; (seated, from left) Lydia Rogers, Nancye Wright, Brian Bindert, Mary Anne Corwin, and Melissa Abramowitz.

The first speaker was Cam Hubbard of Oak Ridge National Lab, who spoke on *in situ* powder diffraction measurements at high temperatures. He discussed a variety of experimental systems, studied both in the High Temperature Materials Lab at ORNL and at the NSLS in

which the ability to follow phase transformations at high temperatures, under synthetic conditions, was key to solving practical problems in ceramics and other high performance materials. Richard Harlow (of Harlow, Inc.) followed with discussions of work performed at the APS on Fe metal catalysts that are used by DuPont in commercial scale manufacturing. These catalysts are activated at high temperature and pressure at the beginning of the process batch, and there was inadequate understanding of the chemical basis for the observed lot-to-lot variation of their performance. High energy x-rays were necessary to penetrate the stainless steel tube used to house the catalyst under process conditions, and high angular resolution was required to distinguish the processes of interest in the catalyst. Insights gained from the study of the state of the activated catalyst have given information useful to optimize the process in the chemical plant.

The next two talks addressed an extension of the domain of powder diffraction that is becoming increasingly important, pair distribution analysis. Briefly, this technique transforms the entire diffraction pattern into a radial distribution function. Instead of analyzing only the Bragg peaks to learn the periodically repeating component of the crystal structure, pair distribution analysis reveals the distribution of local environments throughout the sample. Accordingly, it is particularly valuable in materials that are only partially crystalline, such as nanoscale phases. Valeri Petkov of Central Michigan University provided an introduction to the technique, and discussed recent results from studies of nanophase $LiMoS_2$, $Ag_{0.4}MoS_2$, $(NH_4)_{0.5}V_2O_5$, magnetic $GdAl_2$, and Cs intercalated into zeolite. Jonathan Hanson (Brookhaven National Laboratory, Chemistry Department) continued with the theme of radial distribution structural refinements, combined with "conventional" Bragg peak analysis of diffraction patterns in studies of the reduction of (nominal) CuO and CeO_2, with measurements performed *in situ* at high temperature. This work shows the complementary information available from the two techniques, and the importance of both in unraveling complicated behavior in mixed phase materials with partial occupancy of several crystallographic sites.

After lunch, Bill David (Rutherford Appleton Laboratory, UK) woke the audience up with some startling new comments on a concept taken for granted by most practitioners: least squares analysis. While that would be the correct approach if the data errors obeyed a normal probability distribution governed by counting statistics and the hypothesized model was a correct description of the sample diffraction properties, these conditions are often not met. Starting with a formal description of least-squares analysis, David reviewed principles of experimental design to meet those criteria. He then presented some new results on techniques to deal with problems frequently observed: unknown impurities in a powder diffraction pattern handled with a new minimization criterion, and a maximum likelihood approach to analyze incomplete structures in which some atoms have not been located.

Frontiers in Powder Diffraction Workshop attendees.

The two following talks covered various perovskite-related materials in which the interplay of structural distortions, charge ordering, and magnetism require complementary application of neutron and x-ray powder diffraction. El'ad Caspi of Argonne National Laboratory discussed the phase diagram of the colossal magnetoresistance system $(Ca^{2+}_{1-x}Ce^{4+}_{x})MnO_3$. This is a two-electron doped system (in contrast to more familiar one-electron doped systems such as $(Ca^{2+},Bi^{3+})MnO_3$), and the faster change of electronic charge with ion substitution leads to a much more complicated interplay among charge ordering, orbital ordering, and spin ordering, which in turn causes phase separation over a much larger range than one-electron systems. Patrick

Woodward of Ohio State University opened his talk with several demonstrations that neutrons are often superior to x-rays in *ab initio* structure solutions of oxides and fluorides, even though the latter are much more widely used. He then discussed several neutron and x-ray experiments: Fe charge disproportionation in $CaFeO_3$, Mn orbital ordering in $NdSrMn_2O_6$, and the Verway transition on oxygen deficient double perovskites, $RBaFe_2O_{5+w}$ (R = Rb, Y, Ho, and Nd).

In the last session, Tom Vogt (BNL, Physics Department) discussed work on the high pressure chemistry of zeolites. The theme of his talk was the surprising discovery of materials that expand under pressure, due to increased incorporation of water into the zeolite cavities. Sodium aluminosilicate natrolite undergoes a reversibly pressure-induced lattice expansion, whereas a synthetic analog, potassium gallosilicate natrolite, expands irreversibly, retaining the expanded high pressure phase upon returning to ambient pressure. Vogt presented structure determinations showing the role of non-framework metal ions in distinguishing the two cases. Finally, Peter Stephens presented a talk largely prepared by Robert Von Dreele (Los Alamos and Argonne National Lab) on their work applying high resolution x-ray powder diffraction to proteins.

In all, the broad range of powder diffraction, pair distribution function, and single crystal analysis, and the large and growing user community at synchrotron and neutron facilities points towards increasing growth at the frontiers of powder diffraction. This in turn indicates continuing demand for improved instruments as well as improved access to the current generation of operating instruments.

—Peter Stephens

Workshop on Spectroscopy in High Magnetic Fields: ESR, Infrared, and Other Applications

May 19, 2003

The availability of high field magnets, combined with the development of high resolution/low energy spectroscopic techniques, provides new opportunities for probing materials with synchrotron light. In this workshop, a few selected applications of x-ray and infrared radiation for the study of superconductors, magnetic perovskites, semiconductor quantum wells and other systems were reviewed. x-ray scattering and spectroscopy, electron spin resonance, optically detected Hall effect, and far IR spectroscopy in high magnetic fields were also discussed. The speakers included current users as well as other leading experts from the U.S. and Europe.

Spectroscopy in High Magnetic Fields: ESR, Infrared and Other Applications Workshop attendees.

—Laszlo Mihaly

Processes in Environmental Sciences Workshop

May 19, 2003

Reliable long-term prediction of heavy element mobility in natural multi-component systems or construction of intelligent reactive barrier systems for waste confinement requires a fundamental process understanding. Application of a combination of macro- and microscopic techniques including EXAFS, XANES, FTIR, XRD, XRF and soft x-ray microscopy can provide atomic scale chemical information as well as information of nano- to microscopic spatial distribution in complex matrixes. New single crystal approaches on well defined crystallographic planes furthermore gives

Processes in Environmental Sciences Workshop attendees.

insight in redox-kinetics and sorption relevant mineral surfaces. The scope of this workshop will be to give a discussion platform as well as an overview of recent applications of synchrotron based techniques to elucidate important pathways in natural and anthropogenic influenced environmental systems.

—Thorsten Schaefer

Bio-Matters: from IR to X-rays Workshop

May 21, 2003

For the past two decades the NSLS has been increasingly contributing to structural biology. With the advent of a new facility the aim of this workshop was to discuss the contributions of different synchrotron radiation-based methods to the understanding of molecular structure and biomolecule function. The second goal was to focus on the complementary aspects between these techniques and different methods such as cryo-electron microscopy and neutron scattering methods. The workshop consisted of oral presentations, a poster session, and a panel discussion session on the future requirements and expectations of the NSLS user community. The talks presented are summarized below:

Wayne Hendrickson, Columbia University, *"Synchrotron Crystallography in Biological Discovery,"* introduced the subject of the workshop. In his talk he described the impact of synchrotron radiation on the field of biological crystallography, a number of technical advances, and the problems of radiation damage with the advent of more intense sources. Several examples were discussed in relation to the speed of solution provided by crystallography at synchrotron radiation sources, and the impact to biochemistry and molecular biology.

Chris Jacobsen, Stony Brook University, "*Soft X-ray Imaging and Spectromicroscopy,"* presented high resolution views of chemical contrast through the combination of soft x-ray microscopes and near-edge spectroscopy methods. This approach was illustrated with biomedical examples including microspectroscopy studies of human sperm, and imaging of several cell types.

Rob Scarrow, Haverford College, *"EXAFS Studies of Metalloproteins and the Usefulness of Model Coordination Complexes,"* discussed the application of EXAFS (Extended X-ray Absorption Fine Structure) analysis to a variety of metalloproteins. The determination of the nature of ligand atoms, the number and lengths of bonds, metal-metal distances, and how small molecule crystal structure databases are useful in the interpretation of the results was discussed using lipogenase and porphobilinogen synthase as examples.

Joannna Krueger, University of North Carolina at Charlotte, "*Small-Angle Scattering: Solutions in Protein Structural Analysis,"* discussed x-ray and neutron small angle scattering focusing on the complementary aspects of these techniques and other structural and biochemical approaches such as that obtained from selected-site mutagenesis, circular dichroism, NMR, and electron microscopy.

Bio-Matters from IR to x-rays Workshop attendees.

Udupi A. Ramagopal, Albert Einstein College of Medicine, and Zbigniew Dauter, NIH, "*SAD: Happy Phasing with Weak Anomalous Scatterers,"* described the single-wavelength anomalous diffraction (SAD) as an alternative to the multiple wavelength diffraction method (MAD) applied to sulfur-containing proteins and to radiation sensitive samples.

Uwe Bergmann, Stanford, "*Advances in High-Resolution Hard X-ray Spectroscopy: From Vibrational Studies to Identify ligands to the Local Structure of Water,*" explained that hard x-ray spectroscopy became possible in recent years due to intense sources and improvements in x-ray instrumentation. The application of x-ray fluorescence spectroscopy (XFS) of weak lines, resonant inelastic

x-ray scattering (RIXS), (non resonant) x-ray Raman scattering (XRS) and nuclear resonant vibrational spectroscopy (NRVS) to studies of the oxygen K-edge of water, metalloproteins and Fe containing systems was shown.

Mark Chance, Albert Einstein College of Medicine, "*Structure and Dynamics of Macromolecular Machines,"* described synchrotron footprinting to study the dynamics and interactions of proteins and nucleic acid structures with millisecond time resolution and high structural resolution using nanomoles to picomoles of material. He gave examples for the L-21 ribozyme from *Tetrahymena*, cofilin and time-resolved activation of the actin binding protein gelsolin.

Lisa Miller, NSLS, "*Chemical Imaging of Biological Tissues using a Combination of Infrared, UV-Visible Fluorescence, and X-ray Micro-Spectroscopy,"* discussed the application of synchrotron infrared (IR) micro-spectroscopy and fluorescence techniques for examining the inherent chemical makeup of biological cells and tissues at spatial resolutions not achieved by conventional IR microscopes. Comparisons with other techniques such as immunofluorescence and x-ray micro-spectroscopy were presented in light of Alzheimer's disease, scrapie, and bone disease.

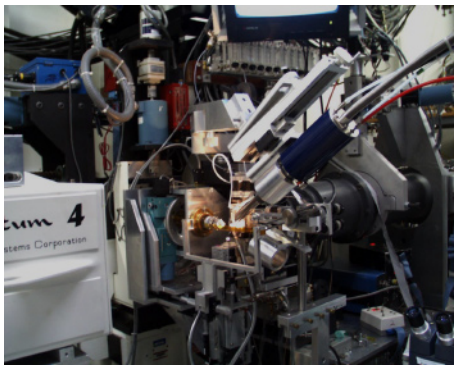
Thomas C. Terwilliger, Los Alamos National Laboratory, "*Structural Genomics: Technology for Structural Biology,"* presented the future needs of structural genomics and the current status. He focused on the technological improvements needed from protein production to structure determination. Several of these developments are underway, one of the most important being the automation of data collection and analysis at x-ray beamlines worldwide. Other technologies such as the engineering of proteins for optimal solubility, automated structure solution, and phase improvement by x-ray crystallography were also discussed.

Several posters on different subjects ranging from imaging to scattering and from the NASA radiological program to Cryo Electron Microscopy were discussed over coffee and a lunch break.

Members of the panel (Wayne Hendrickson, Columbia; Thomas Terwilliger, Los Alamos; Joachim Frank, Wadsworth Center; and Naomi Chayen, Imperial College), and workshop participants addressed several technological problems such as instruments, detectors, methods, and software developments to subjects such as multiple assemblies and unstable systems. The main recommendations were related to the development of detectors, brighter sources, instrumentation to handle smaller crystals, software for automated structure determination, modeling, and docking.

Prior to the Bio-matters workshop, a workshop was held on the basic and advanced methods in protein crystallization. The aim of this one-day workshop was to allow participants to have a hands-on experience with the different crystal growth methods available to protein crystallographers. Naomi Chayen (Imperial College) explained the microbatch method and the oil method; Miroslawa Dauter (NIH) discussed the hanging drop method, co-crystallization of heavy atoms, and seeding; Zbigniew Dauter (NCI-NIH) presented strategies in choosing an optimal derivative and data collection; and Grahemen Williams (Brookhaven-Instruments) discussed the application of the light scattering technique to protein crystallization. Two parallel sessions were organized in the morning and in the afternoon where the 22 participants could experience the different crystallization methods. We thank our sponsors Nextal Biotechnologies, Brinkmann Instruments, Millipore, Fisher Scientific, Brookhaven Instruments Corporation, and New York New Jersey Scientific, Inc., for their kind support, without which the Crystallization Workshop, a satellite meeting to the NSLS Annual Users' Meeting, would not have been possible.

—Vivan Stojanoff



Many life science users at the NSLS perform protein crystallography experiments like the one shown here.