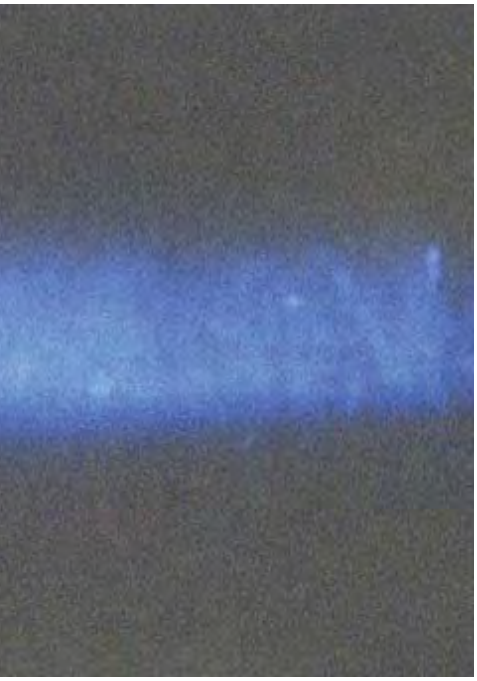
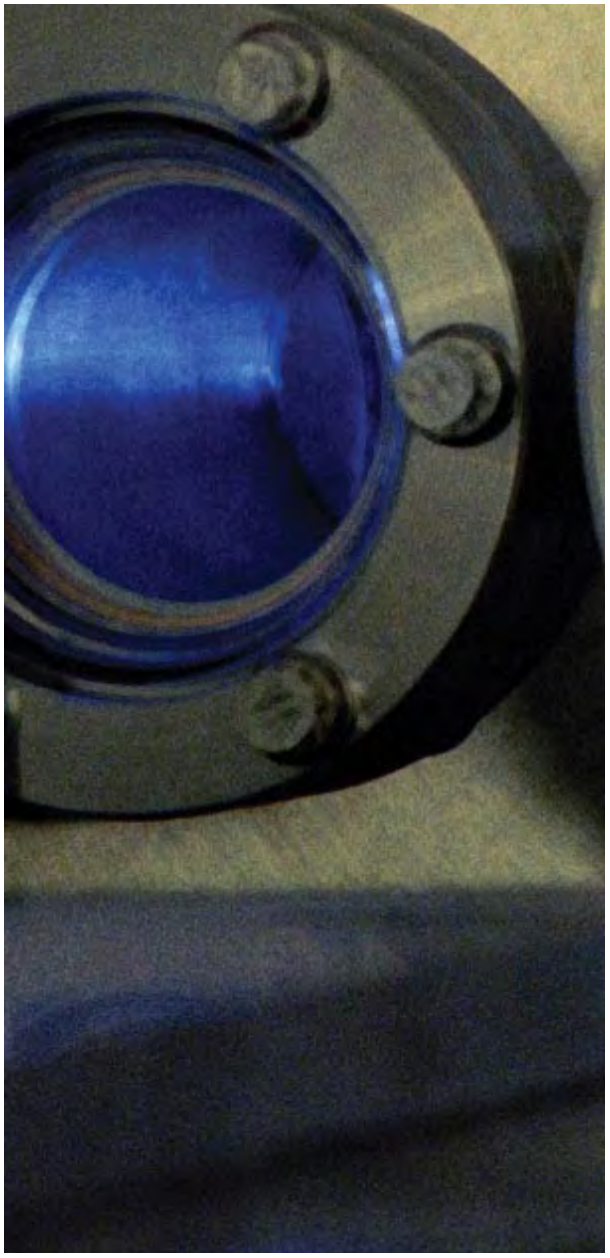




Celebrating 25 Years

National Synchrotron Light Source

2007 Activity Report

1970 - Renata "Rena" Wiener Chasman (right) and G. Kenneth Green (left) work with NSLS Chair Marty Blume. Chasman and Green were responsible for the ingenious design of the two storage rings at the NSLS, what is commonly known as the "Chasman-Green lattice."

NSLS Celebrates its Silver Anniversary

Thursday, November 22, 2007 – Thanksgiving Day – was the 25th anniversary of the NSLS building dedication. The first facility designed and built specifically for producing and exploiting synchrotron radiation, the NSLS was conceived in 1970, officially proposed in 1976, and had its ground-breaking in 1978. It was a rough start, but at the end of the four-year construction period, the project was on schedule and within its set cost of $24 million. And most importantly, both the VUV and X-Ray rings had beam.

The 1982 dedication event featured Lab Director Nicholas Samios as the master of ceremonies. Speakers included NSLS Chair John McTague, local Congressman William Carney, Don Stevens, Director of the Division of Materials Science of the U.S. Department of Energy, and George A. Keyworth, President Reagan's science advisor. During the celebration, Samios also announced that the NSLS Division was being upgraded to a department. McTague predicted that within a year, 50 experiments in a wide variety of fields would be running at the NSLS.

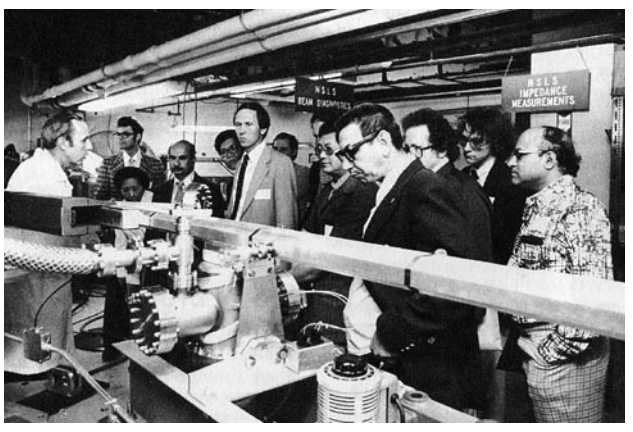
Funding difficulties and technical problems caused delays, so it wasn't until April 1984, after a machine shutdown and overhaul, that the design energy of 700 MeV was met in the VUV Ring. The X-Ray Ring followed the next year, meeting its design energy of 2.5 GeV in August 1985. Today, 25 years after its dedication, the NSLS holds its status as one of the world's most productive scientific user facilities. Home to nine R&D 100 Awards and a Nobel Prize, the NSLS hosts more than 2,200 users every year, who, combined with NSLS scientists, produce more than 900 yearly publications on 65 beamlines in diverse fields ranging from biology and physics to environmental and materials sciences.

Now that's something to be thankful for.

A baby mammoth, which has been frozen in ice for 40,000 years, is discovered in the Soviet Union.

1977

The National Synchrotron Light Source (NSLS) project starts.



1978

← NSLS groundbreaking

1979

The First Users' Meeting →

1980

Ring assembly begins on the Vacuum Ultraviolet (VUV) Ring floor.

Construction starts on the Linear Accelerator (Linac) to the Booster Ring.

The Linac beam reaches 70 MeV.

Solar One, the world's largest solar-power generating station, goes into operation, generating up to 10 megawatts.

1981

NSLS staff celebrate the first turn of beam around the VUV Ring.

1982

NSLS dedication takes place. The NSLS Division also is upgraded to a department.

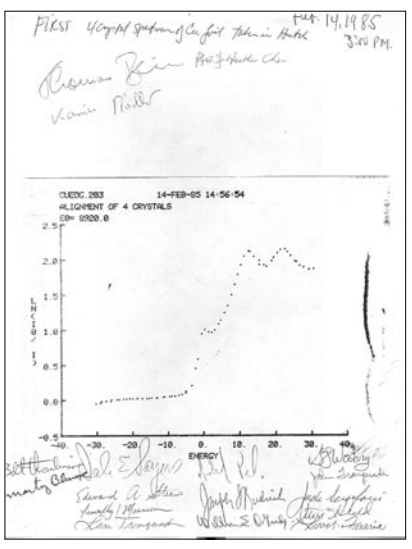
The first turn of beam is made around the X-Ray Ring.

First visible light for machine testing emerges from the NSLS VUV Ring. →

1983

The first Activity Report is distributed.

1984



The concept of a high-gain self-amplified spontaneous emission free electron laser is pioneered at the NSLS.

Human growth hormone produced by genetically engineered bacteria is made available for treatment of children with growth problems.

1985

← The first x-ray absorption spectrum from a copper foil is taken in the X11 hutch with signatures.

Groundbreaking takes place for the "Phase II" construction project to expand the facility by 52,000 sq ft for new offices, laboratories, beamlines, and room for new experimental equipment.

1986

Researchers from the National Bureau of Standards and the University of Tennessee, Knoxville, win an R&D 100 Award for the development of a soft x-ray emission spectrometer installed at the NSLS.

1987

Brookhaven's Accelerator Test Facility (ATF) is started as a facility for accelerator and beam physicists, operated by the NSLS and BNL's Center for Accelerator Physics.

1988

Researchers from BNL and the University of Chicago receive an R&D 100 Award for developing an x-ray microprobe/microscope.

An R&D 100 Award is given to researchers from AT&T Bell Laboratories for the development of a high-resolution soft x-ray monochromator at the NSLS.

1989

An R&D 100 Award is given to a team of NSLS physicists and engineers who developed a real-time, harmonic closed-orbit feedback system.

NSLS engineers develop a new method for determining transverse electron beam position through radio frequency receivers, which have been widely adopted at other facilities and are the basis for a commercial product.

The Human Genome project is set up as a collaboration between scientists from 16 countries to work out the human genetic code.

1990

Researchers from the NSLS and Fairleigh Dickenson University win an R&D 100 Award for developing a wavefront dividing infrared interferometer at the NSLS.

The first infrared beamline is built at the NSLS to study surface vibrational dynamics.

Scientists from BNL, AT&T Bell Laboratories, and the NSLS pioneer resonant x-ray scattering techniques for probing magnetism.

1991

Researchers from Stony Brook, BNL, LBNL, and IBM win an R&D 100 Award for developing a high-resolution scanning photoelectron x-ray microscope.

Researchers from McGill University, MIT, IBM, the University of Florida, and the NSLS perform pioneering speckle experiments using coherent x-rays.

The prototype small-gap undulator is developed by the NSLS and SPring-8.

1992

NSLS researchers and collaborators first demonstrate the use of nuclear forward scattering to perform Mössbauer spectroscopy in the time domain.

1993

A time-varying elliptically polarized wiggler is constructed and operated in the NSLS X-Ray Ring.

1994

↑ Groundbreaking takes place for the Structural Biology addition and expansion of the facility's experimental floor space.

The NSLS Source Development Laboratory (SDL) is established.

Researchers from the University of Michigan, AT&T Bell Laboratories, the University of Illinois, and the NSLS perform pioneering x-ray photon correlation spectroscopy experiments using coherent x-rays.

Flat-screen TV sets are demonstrated for the first time.

1995

A small gap in-vacuum undulator with a full aperture as small as 3.3 mm is developed at the NSLS.

NSLS scientists develop high-energy-resolution x-ray analyzers to extract new information from and improve the resolution of x-ray absorption spectroscopy, as well as probe electronic excitations using inelastic x-ray scattering.

1996

The fourth harmonic radio frequency system for bunch lengthening is used in the VUV Ring.

Dolly, the first cloned sheep, is born.

1997

Brookhaven and Quantar Technology, Inc. researchers win an R&D 100 Award for developing a device at the NSLS called a Fluorescence Omnilyzer.

The X-Ray Ring begins running at 2.8 GeV.

1998

A low-emittance lattice is operated at the NSLS X-Ray Ring at 2.584 GeV.

The building of a new international space station to replace Mir begins.

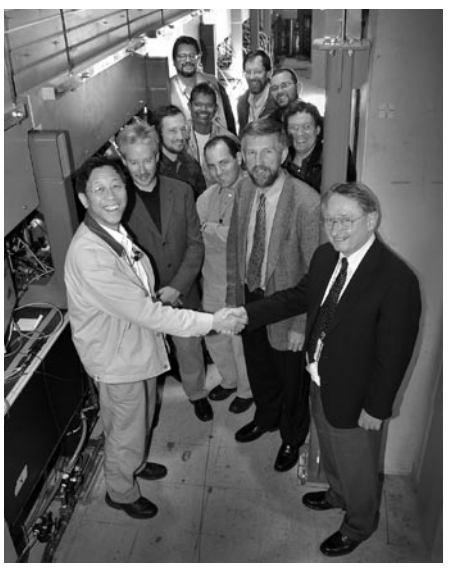
1999

Stony Brook University and Bell Laboratories researchers win an R&D 100 Award for developing a cryo scanning transmission x-ray microscope.

NSLS researchers observe coherent emission from the VUV Ring.

↑ Researchers from Stony Brook University and IBM demonstrate coherent x-ray diffraction (or "lensless") imaging.

2000



At the ATF, NSLS staff achieve second harmonic high-gain harmonic generation.

Scientists report that the age of the universe is at least 12.5 billion years old, give or take 3 billion years.

2001

2002

← At the SDL self-amplified spontaneous emission at 400 nm and third harmonic high gain harmonic generation at 266 nm are achieved.

Two spacecraft from the United States land on Mars. The European probe, Beagle 2, will be officially declared missing early in 2004, presumed to have undergone a "hard landing" on the surface of the planet.

2003

NSLS user Roderick MacKinnon wins the 2003 Nobel Prize in Chemistry for work done partly at the NSLS explaining how a class of proteins helps to generate nerve impulses.

2004

2005

The Department of Energy (DOE) grants "Critical Decision Zero" (CD-0) status to the National Synchrotron Light Source-II (NSLS-II).

2006

NSLS scientists win a 2006 R&D 100 award for developing the Sagittal Focusing Laue Monochromator, which is able to focus a large spread of high-energy x-rays. →

2007

The Center for Functional Nanomaterials (CFN) officially opens on the first day of the 2007 joint NSLS-CFN Users' Meeting. CFN researchers will work with those at the NSLS, and eventually at the NSLS-II, to help solve the nation's energy challenges.

July - DOE grants "Critical Decision 1" (CD-I) status to NSLS-II.

December - DOE grants "Critical Decision 2" (CD-2) status to NSLS-II.

National Synchrotron Light Source
2007 Activity Report

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The National Synchrotron Light Source Department is supported by the Office of Basic Energy Sciences, United States Department of Energy, Washington, D.C.

Brookhaven National Laboratory, Brookhaven Science Associates, Inc., Upton, New York 11973
Under contract no. DE-AC02-98CH10886

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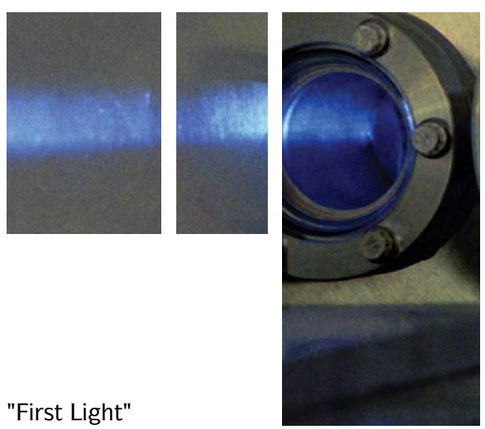

"First Light"

Early synchrotron light from the NSLS Vacuum Ultraviolet (VUV) ring viewed at beamline U14 in 1982.

Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

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"The NSLS is one of the most widely used and productive scientific facilities in the world."

Chi-Chang Kao
Chairman, National Synchrotron Light Source

Chairman's Introduction

Twenty-five years after its dedication, the NSLS continues to shine. This year, we celebrated our silver anniversary and a history that can teach us many lessons: resourcefulness in the face of a tight budget, ingenuity when things don't turn out as planned, and most importantly, a strong commitment to scientific and technical excellence. The tumultuous start to the NSLS eventually gave way to what we have today – one of the world's most productive and cost-effective user facilities. With 2,219 individual users, about 100 more than last year, and a record-high 985 publications, 2007 was no exception.

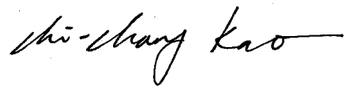
In addition to producing an impressive array of science highlights, which are included in this Activity Report, many NSLS users were honored this year for their scientific accomplishments. These include: the University of Colorado, Boulder's Noel Clark and Brandeis University's Chris Miller, who were among 72 new members elected to the National Academy of Sciences; Cold Spring Harbor researcher Leemor Joshua-Tor, who won the 2007 Dorothy Crowfoot Hodgkin Award; and Yale biophysicist Thomas Steitz, who shared one of the five 2007 Gairdner International Awards.

Throughout the year, we made major strides in the development of our scientific programs by strengthening strategic partnerships with major research resources and with the Center for Functional Nanomaterials (CFN). Of particular note, the Consortium for Materials Properties Research in Earth Sciences (COMPRES) received renewed funding for the next five years through the National Science Foundation. COMPRES operates four high-pressure NSLS beamlines – X17B2, X17B3, X17C, and U2A – and serves the earth science community as well as the rapidly expanding segment of researchers using high-pressure techniques in materials, chemical, and energy-related sciences. A joint appointment was made between the NSLS and Stony Brook University to further enhance our interactions with COMPRES.

We've also made major progress on two key beamline projects outlined in the Five-Year Strategic Plan: the X25 beamline upgrade and the construction of the X9 small angle scattering (SAXS) beamline. The X25 overhaul, which began with the installation of the in-vacuum mini-gap undulator (MGU) in January 2006, is now complete. X25 is once again the brightest beamline for macromolecular crystallography at the NSLS, and in tandem with the X29 undulator beamline, it will keep the NSLS at the cutting edge in this important area of research. Upgrade work associated with the new MGU and the front end for the X9 SAXS beamline – jointly developed by the NSLS and the CFN – also was completed. Beamline X9 will host the SAXS program that currently exists at beamline X21 and will provide new microbeam SAXS capabilities and much-needed beam time for the life sciences, soft condensed matter physics, and nanoscience communities.

Looking toward the future, we have made a significant step in expanding the user base and diversifying our work force by holding the first Historically Black Colleges and Universities (HBCU) Professors' Workshop. The workshop, which brought 11 professors to the NSLS to learn how to become successful synchrotron users, concluded with the formation of an HBCU User Consortium. I look forward to welcoming the return of these professors to the NSLS with their students and colleagues to conduct experiments and take advantage the connections they've made.

Finally, we have made significant contributions in optics and detector development to enhance the utilization of the NSLS and address the challenges of NSLS-II. In particular, x-ray detectors developed by the NSLS Detector Section have been adopted by an increasing number of research programs both at the NSLS and at light sources around the world, speeding up measurement times by orders of magnitude and making completely new experiments feasible. Significant advances in focusing and high-energy resolution optics have also been made this year. These developments are a clear demonstration of the ingenuity of the NSLS staff, and it is this long tradition of innovation that underlies the continued success of our facility.

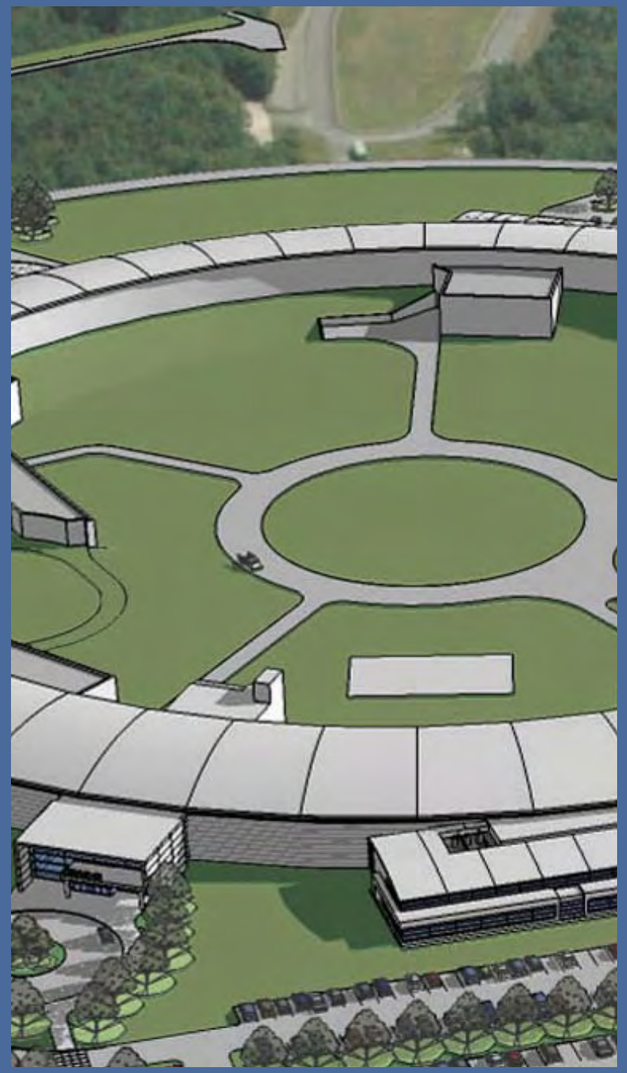


A Beacon for Research

Funded by the U.S. Department of Energy's Office of Basic Energy Sciences, the National Synchrotron Light Source (NSLS) is a national user facility that operates two electron storage rings: x-ray (2.8 GeV, 300 mA) and vacuum ultraviolet (VUV) (800 mev, 1.0A). These two rings provide intense light spanning the electromagnetic spectrum — from very long infrared rays to ultraviolet light and super-short x-rays — to analyze very small or highly dilute samples. The properties of this light, and the specially designed experimental stations, called beamlines, allow scientists in many diverse disciplines of research to perform experiments not possible at their own laboratories.

Each year, more than 2,200 scientists from more than 400 universities and companies use the NSLS for research in such diverse fields as biology, physics, chemistry, geology, medicine, and environmental and materials sciences. For example, researchers have used the NSLS to examine the minute details of computer chips, decipher the structures of viruses, probe the density of bone, determine the chemical composition of moon rocks, and reveal countless other mysteries of science. The facility has 65 operating beamlines, with 51 beamlines on the X-Ray Ring and 14 beamlines on the VUV-IR Ring. It runs seven days a week, 24 hours a day throughout the year, except during periods of maintenance and studies. Researchers are not charged for beam time, provided that the research results are published in open literature. Proprietary research is conducted on a full-cost-recovery basis.

With close to 1,000 publications per year, the NSLS is one of the most prolific scientific facilities in the world. Among the many accolades given to its users and staff, the NSLS has won nine R&D 100 Awards for innovations ranging from a closed orbit feedback system to the first device able to focus a large spread of high-energy x-rays. In addition, a visiting NSLS researcher shared the 2003 Nobel Prize in Chemistry for work explaining how one class of proteins helps to generate nerve impulses.



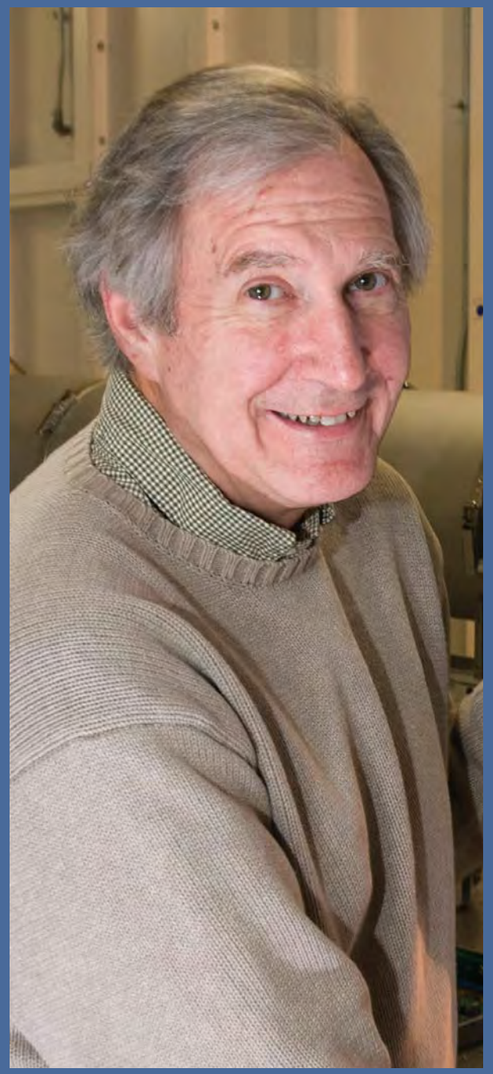
A New Light on the Horizon

Since its first operations in 1982, the NSLS has continually updated its technology and expanded its scientific capabilities. However, as the boundaries of scientific discovery have been expanded, many researchers are looking for capabilities beyond those provided by the NSLS. And while newer synchrotrons surpass the performance of the present NSLS, no synchrotron anywhere in the world will enable scientists to image and characterize materials down to billionth-of-a-meter (nanometer) resolution.

To address this need, Brookhaven is building ¬the National Synchrotron Light Source II (NSLS-II). The new facility, which will replace the NSLS, will be a medium-energy storage ring designed to deliver world-leading brightness and flux. It will provide advanced tools for discovery-class science in condensed matter and materials physics, chemistry, and biology. For example, major advances in energy technologies – such as the use of hydrogen as an energy carrier; the widespread, economical uses of solar energy; or the development of the next generation of nuclear power systems – will require scientific breakthroughs in developing new materials with advanced properties.

The combination of capabilities at NSLS-II will have broad impact on a wide range of disciplines and scientific initiatives in the coming decades, including new studies of small crystals in structural biology, the development of probes for nanoscience, coherent imaging of the structure and dynamics of disordered materials, greatly increased applicability of inelastic x-ray scattering, and properties of materials under extreme conditions. This high-brightness light source will also foster research in areas such as structural genomics and drug design as well as extend the studies of early disease detection.

The leading-edge ability of NSLS-II to analyze materials will help guide the development of new materials at Brookhaven's Center for Functional Nanomaterials. The synergy of these two facilities is expected to lead to breakthroughs in the use of renewable energy through improved energy conversion, transmission, and storage – science that ultimately will enhance national and energy security and help drive abundant, safe, and clean energy technologies.



"In 2007, NSLS users and staff began to combine synchrotron techniques with the characterization and fabrication facilities available at nanoscience research centers for fundamental studies that could impact applications from improved electronic devices to more efficient energy conversion and storage."

Ron Pindak
Physical and Chemical Sciences Division Head

Science Highlights

In 2007, NSLS users and staff began to combine synchrotron techniques with the characterization and fabrication facilities available at nanoscience research centers, such as Brookhaven's Center for Functional Nanomaterials, to explore novel catalytic materials and nanoscale assemblies important for applications ranging from electronic devices to more efficient energy conversion and storage. There were also new insights on problems that have puzzled scientists for decades, including the nature of high-temperature (Tc) superconductivity. Key discoveries are listed below and described more fully in the science highlights that follow.

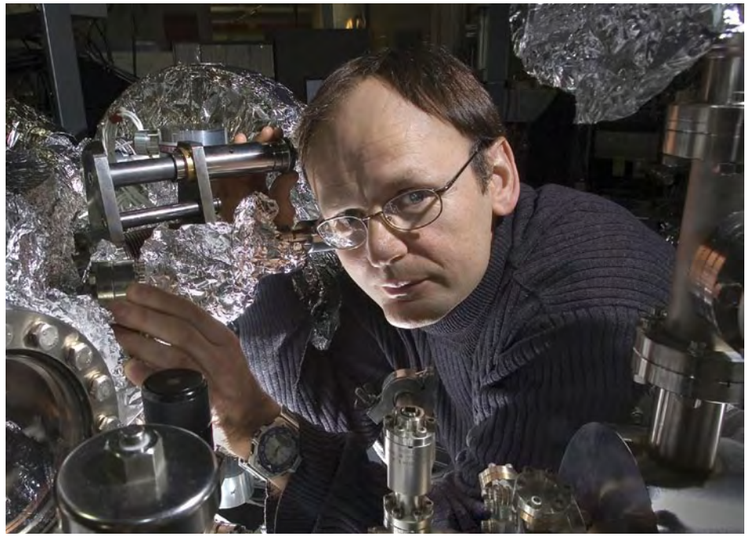
In the field of condensed matter physics, there were research advances in high-Tc superconductors, magnetic interface ferromagnetism, and semiconductor electron quantization. Angle-resolved photoemission spectroscopy (ARPES) measurements revealed a second kink in the energy band of a high-Tc superconductor at an energy that could only be associated with spin fluctuations. This result demonstrated the important role that spin fluctuations play in this phenomenon. ARPES measurements also led to the discovery of a quantized electron accumulation layer near the surface of the semiconductor indium nitride. Usually, quantized states are only observed in engineered materials or electronic devices. Finally, a combination of soft x-ray reflectometry and magnetometry experiments measured the depth of the magnetization across an antiferromagnetic-ferromagnetic interface, providing new information on the origin of exchange bias – an effect used in spintronic devices.

In the materials science realm, NSLS research focused on nanotube assemblies and nano-patterned films. Temperature-dependent x-ray diffraction measurements demonstrated that phase-change materials maintain their bulk properties when solution-processed into thin films or nanodots. This result was important for the incorporation of these materials in phase-change random access memory devices. Small-angle x-ray scattering was applied to determine the height and spatial distribution of the diameters of multiwall carbon nanotubes (CNTs) assembled perpendicular to a substrate in a dense forest. These densely packed CNT arrays show promise for nanoelectronics, superhydrophobic films, and power applications. A third important advance involved introducing cavities into nanorods of titanium oxide, making them 25 percent more efficient in absorbing ultraviolet radiation. This has applications in solar light conversion, sunscreens, and hydrogen storage.

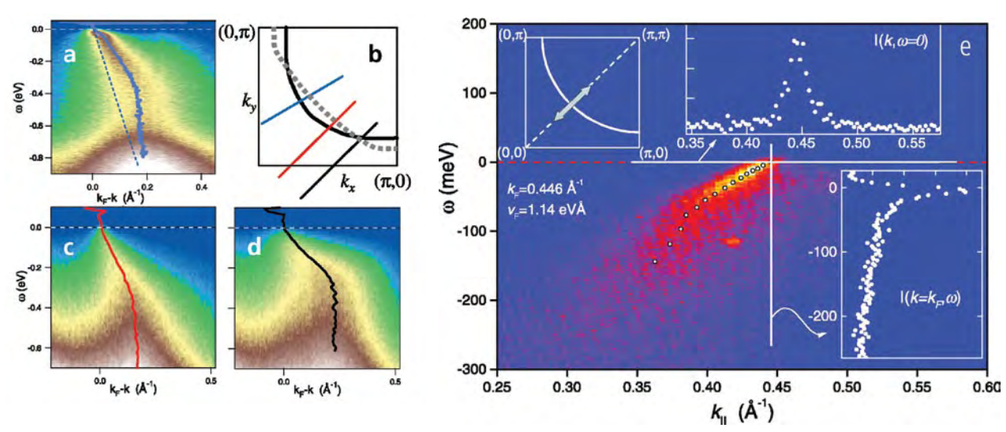
In the area of chemical sciences, catalysis was the dominant research theme and x-ray absorption spectroscopy (XAS) was the primary technique. Using XAS, researchers demonstrated that applying gold clusters to the surface of platinum nanoparticles substantially improved their stability, maintaining their effectiveness as a catalyst in hydrogen fuel cells. XAS and electron diffraction were also used to demonstrate how local disorder and orbital symmetry could be tailored to produce a high-dielectric constant material, CCTO, crucial for the production of the ultra-small capacitors needed as electronic circuits continue to shrink in size. A third XAS measurement, combined with first principles calculations, elucidated the role of titanium (Ti) as a catalyst in the reversible hydrogen cycle of sodium aluminum hydride. The Ti was found to be coordinated by more than 10 aluminum (Al) atoms and localized at or near the surface, rather than in interstitial or substitutional sites. The study also explained the high Al mobility observed in the complex metal halides.

In x-ray optics, it was demonstrated, by focusing x-rays beyond the critical angle, that there are no fundamental limits for using refractive optics to focus x-rays to nanometer-sized spots. And in accelerator physics, researchers succeeded in producing ultra-fast pulses of terahertz radiation that were sufficiently intense to introduce non-linear optical effects. These ultra-fast pulses can be used to study molecular processes.

Physical and Chemical Sciences Introduction



Tonica Valla



"Big kink" (left) vs. "small kink" (right). a) OP91 BSCCO (T = 10K). b) Brillouin zone (BZ) and Fermi surface (FS) for BSCCO (solid line) and LBCO (dashed line). Straight lines represent the momentum lines probed in the spectra with correspondingly colored dispersion. c) and d) LBCO spectra at x = 0.125 for two different momentum lines as indicated in b). e) ARPES intensity from the nodal line in superconducting state in optimally doped BSCCO. The velocity or rate of dispersion changes in the vicinity of the Fermi level, resulting in a "kink" in dispersion at ~60 meV bellow the Fermi level. "Small kink" image from "Evidence for Quantum Critical Behavior in the Optimally Doped Cuprate $Bi_2Sr_2CaCu_2O_{8+\delta}$," T. Valla, A. V. Fedorov, P. D. Johnson, B. O. Wells, Q. Li, G. D. Gu, and N. Koshizuka, *Science* **285**, 2110 (1999).

Illuminating a Second "Kink" in High-Tc Superconductors

There's another kink in the mystery of high-temperature (Tc) superconductors – literally. Using photoemission studies at the NSLS, a group of researchers has revealed a new anomaly, or "kink," in the energy spectrum of high-energy electrons in two different families of cuprate superconductors, further complicating their quest to discover exactly how the materials conduct electricity with zero resistance

In 1999, a group of researchers led by BNL physicist Tonica Valla discovered a low-energy kink in the energy bands of electrons in high-Tc superconductors just as they went through the transition temperature from their normal to superconducting state. This spectral abnormality is thought to be signature of an interaction or "coupling" between an electron and an excitation such as a phonon, which is a vibration of the ions that form the lattice of a superconductor's crystal.

"In conventional low-temperature superconductors, this is really the interaction that causes superconductivity," Valla said. "And there was a hope that when we saw a similar kink in high-temperature superconductors that maybe the mechanism of superconductivity would be resolved. But it's not that easy."

While some groups hold that the mechanism is the same as in conventional superconductors — that is, that phonons are responsible for electron pairing – other scientists believe that changes in the spin alignment, or magnetic polarity (magnons), of adjacent electrons are responsible. However, because both excitations are found in the energy range of the low-energy kink – between 40 and 70 meV – it's difficult to determine which mechanism is correct.

And recently, Valla's group has thrown another complication into the mix, unveiling a second and much larger kink in the high-Tc superconductor "LBCO" (named for the elements it contains: lanthanum, barium, copper, and oxygen) and "BSCCO" (containing bismuth, strontium, calcium, copper, and oxygen). Using high-resolution spectrometers at NSLS beamline U13UB, Valla's group measured the energy and the angle at which the electrons were emitted from the superconducting crystals, allowing them to reconstruct the original electrons' state. This time around, the research team found a large anomaly in the energy spectrum of the materials at 350 meV, the details of which are published in the April 20 edition of *Physical Review Letters.*

"It was a piece of cake to see because it's so huge, but you have to look deep enough," Valla said, adding that the lower energy kink makes just a small contribution to the total kink. "Now, the question is, 'What's the cause of this big one?'"

Because they only exist at lower energies, the researchers immediately ruled out phonons as a possible culprit. In fact, Valla's group now believes that both the large and small kinks are caused by something different: spin fluctuations. This excitation occurs when the spin of one atom is changed, causing a domino effect as its neighbors flip in order to get back into the proper alignment, whether it's ferromagnetic (spins pointing in the same direction) or antiferromagnetic (spins pointing in opposite directions). Spin fluctuations occur at up to 400 meV, and they appear to die out when a material's superconductivity disappears, providing a further link for their involvement in the mechanism of high-Tc superconductors.

"For conventional superconductors, it is clear that the observation of a kink is related to superconductivity," Valla said. "In high-Tcs we are seeing kinks all over the place and some of them might also be closely related to superconductivity. Maybe the low-energy kink is the only important one, but it also might be that the total interaction at higher energies is important as well."

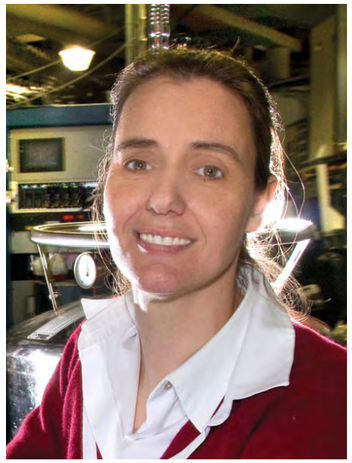
Valla said that future studies will extend to other materials to verify that the high-energy kink is present in all high-Tc cuprate superconductors and to further investigate the role of spin fluctuations in the system.

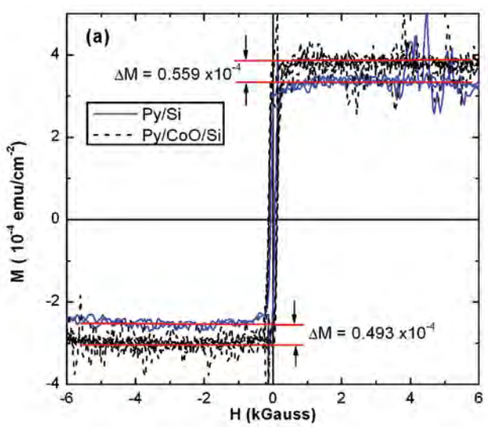
Other researchers involved in the work include: Tim Kidd (BNL and the University of Northern Iowa); Weiguo Yin, Genda Gu, and Peter Johnson (BNL); and Zhihui Pan and Alexei Fedorov (Lawrence Berkeley National Laboratory). Their work was supported by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science.

T. Valla, T. Kidd, W. Yin, G. Gu, P. Johnson, Z. Pan, A. Fedorov, "High-Energy Kink in the Electron Dispersion of High Temperature Cuprate Superconductors," *Phys. Rev. Lett.,* **98**: 167003 (2007).

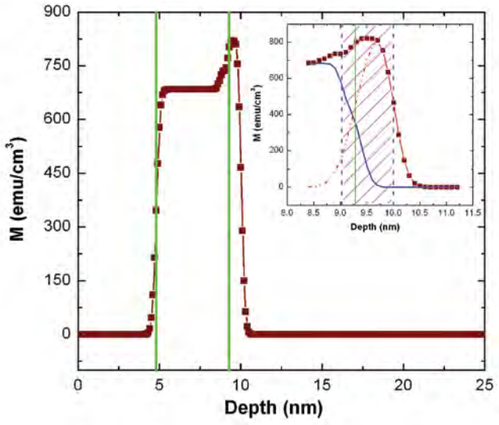
Sujoy Roy

Cecilia Sánchez-Hanke





Room temperature hysteresis measurements for same thickness of Py deposited on Si (blue) and CoO/Si (black dashed). Red lines indicate the magnetization value at the positive and negative saturation for Py/Si and Py/CoO/Si, respectively.



Depth-dependent magnetic density profile in absolute units in a Py/CoO bilayer system. Green solid lines represent structural/chemical interfaces. The inset shows the Py/CoO interface region where the structural and magnetic features are modified. Solid blue and dashed-dot red curves represent the individual Py and CoO contribution to the net magnetization. The hatched area within violet dashed lines represents the magnetic interface between Py and CoO.

Studying Magnetic Interface Ferromagnetism

The development of various magnetic-based devices, such as read-heads found inside your computer, depends on the discovery and improvement of new materials and magnetic effects. In particular, researchers are interested in the magnetic behavior at the interfaces between different materials, especially in devices presenting so-called "exchange bias" – an effect produced by combining ferromagnetic and antiferromagnetic materials. Because these materials sometimes have very small magnetizations, characterizing them at atomic- and nanometer-length scales isn't easy. However, using soft x-ray resonant magnetic scattering at the NSLS, a group of researchers has found a way to investigate how magnetism varies with the depth of a thin layer of material, specifically at the interface.

Many state-of-the-art materials and devices for magnetic reading and writing rely on the interplay between magnetic properties. This is especially true for quantum mechanical spins, the origin of the atoms' magnetic behavior. In a ferromagnet, the atoms' magnetic moments are aligned in parallel, whereas in an antiferromagnet, the moments are antiparallel. The coupling between an antiferromagnetic and a ferromagnetic material may give rise to exchange bias, which locks the ferromagnet spins (a major contributor to the magnetic moment). This effect is of fundamental importance to modern magnetic devices since it forms a reference layer with a fixed direction of magnetization.

However, there are still ongoing controversies about the microscopic origin of exchange bias. Previous studies have revealed the existence of "unpinned spins" (spins in one direction not matched by an opposite spin and responding to applied field) at the antiferromagnetic interface. To examine how these spins are distributed and how they interact with the ferromagnetic spins across the interface, the researchers determined the depth dependence of the net magnetization in an exchange-biased sample consisting of permalloy, a nickel iron magnetic alloy (the ferromagnet), and cobalt oxide (the antiferomagnet). This was done at room temperature, where no "pinned" spins exist in the antiferromagnet and the exchange bias does not occur.

"Not many instruments can do this," said lead researcher Sujoy Roy, from the University of California, San Diego. "You can use microscopes to look at the surface of a material, but we wanted to scan the films from top to bottom, especially the interface."

To do this, the researchers used x-ray reflectometry at NSLS beamline X13A and magnetometry at University of California, San Diego. These techniques allowed them to determine the distributions of free spins and of spin orientation, providing an atomic-level picture of the exchange bias mechanism.

"This was a very unique method," Roy said. "We have been able to pinpoint exactly how the magnetism is varying as a function of depth in absolute units."

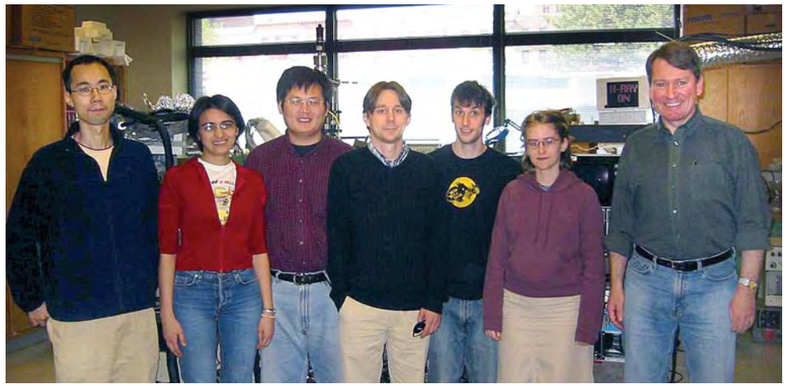
The team found that a region at the permalloy/cobalt oxide interface is modified both chemically and magnetically. This modification is due to the formation of an oxide layer containing both cobalt and permalloy in between the bilayer. While there is no significant magnetization in the cobalt oxide, the temperature dependence of the interfacial layer's net magnetization is different than the permalloy. Their results were published in the January 31, 2007 edition of *Physical Review B.*

The magnetization in this interfacial region, and how it interacts with the antiferromagnetic spins in the cobalt oxide at low temperatures, is expected to play a key role in determining the exchange bias properties of this bilayer. Future work will repeat the experiment at lower temperatures, where exchange bias develops in the sample, in order to determine how the interface properties change.

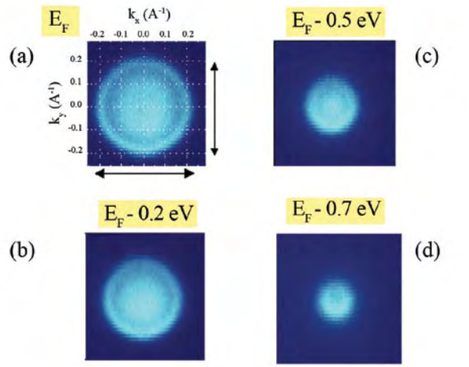
Other researchers include: Cecilia Sánchez-Hanke and Chi-Chang Kao (NSLS); Sungkyun Park (Los Alamos National Laboratory and Korea Basic Science Institute); Mike Fitzsimmons (Los Alamos National Laboratory); Sunil Sinha (University of California, San Diego, and Los Alamos National Laboratory); Y. Tang, Jung-Il Hong, Xuerong Liu, M. Brian Maple, and Ami Berkowitz (University of California, San Diego); and David Smith (Arizona State University).

This research was supported by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science.

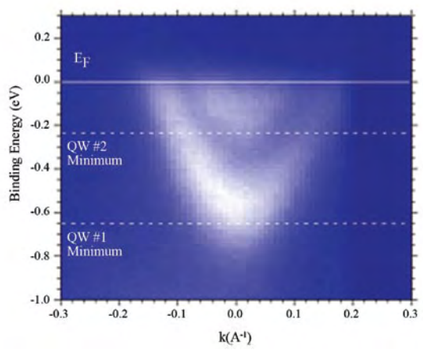
S. Roy, C. Sanchez-Hanke, S. Park, M. Fitzsimmons, Y. Tang, J. Hong, D. Smith, B. Taylor, X. Liu, et al., "Evidence of Modified Ferromagnetism at a Buried Permalloy/CoO Interface at Room Temperature," *Phys. Rev. B,* **75**: 014442 (2007).



From left, Yufeng Zhang, Leyla Colakerol, Shancai Wang, Lukasz Plucinski, Tim Learmonth, Sarah Bernardis, and Kevin Smith.



ARPES photocurrent intensity map of states within 1.5 eV of E_F. hν = 69 eV, and the sample temperature was 177 K. The sample was annealed to 300 °C in UHV for 30 minutes. The false color intensity reflects the photocurrent, with lighter intensity indicating higher current. The momentum direction is along ΓΣM, in the surface plane.



Fermi surface and constant binding energy contours. The photocurrent is plotted as kx and ky are varied, while the binding energy relative to EF is kept constant at 0 eV (Fermi surface), 0.2 eV, 0.5 eV and 0.7 eV. hν = 70 eV, and sample temperature was 60 K. The diameter of the outer Fermi surface is 0.4 $Å^{-1}$.

Unveiling Electronic Properties Near a Semiconductor Surface

Electrons near the surface of semiconducting indium nitride have been shown to exist in "quantum well" states, which are remarkably simple fundamental energy states. It is highly unusual to observe such states in ordinary materials; usually they are observed only in engineered materials or electronic devices. Recently, a team of researchers from Boston University, the University of Warwick, and the Advanced Light Source discovered the quantum well states by irradiation of indium nitride with intense x-rays, which caused the electrons in these states to be ejected from the material into vacuum, where their energy and momentum was measured. This technique, known as photoemission spectroscopy, allowed them to make the first definitive determination of these properties.

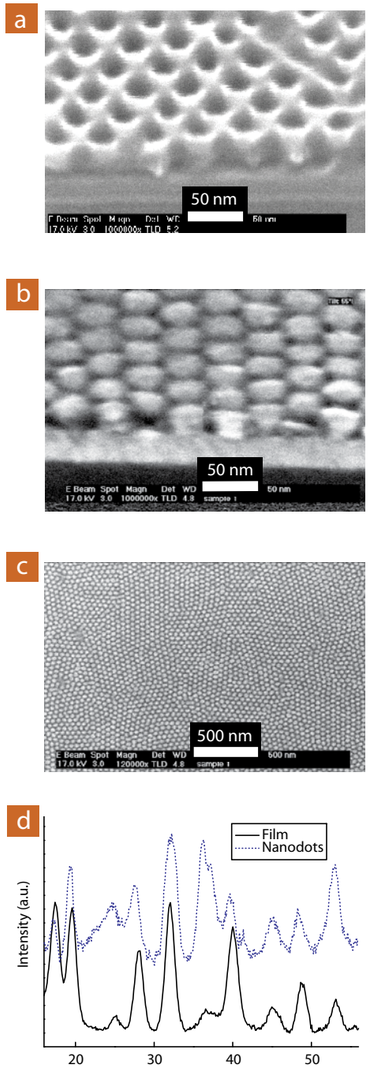
Using angle resolved photoemission spectroscopy at the Advanced Light Source and NSLS beamline U5UA, the researchers directly observed a quantized electron accumulation layer near the surface of the narrow gap semiconductor indium nitride (InN). Electron accumulation is a phenomenon observed in certain semiconductors whereby a higher density of electrons is observed in a layer near the surface of the solid. It is postulated that the surface region in InN has a higher charge density than the bulk due to N vacancies or donor-type surface states. This causes the surface Fermi level to lie in the conduction band. The research team discovered that not only are electrons observed far above the conduction band minimum, but these electrons are found to be quantized perpendicular to the surface, i.e. the electrons in the accumulation layer have been determined to reside in quantum well states.

Their results, which were published in the December 4, 2006 edition of *Physical Review Letters*, are the first unambiguous observation that electrons in the InN accumulation layer are quantized, and the first time the Fermi surface associated with such states has been measured.

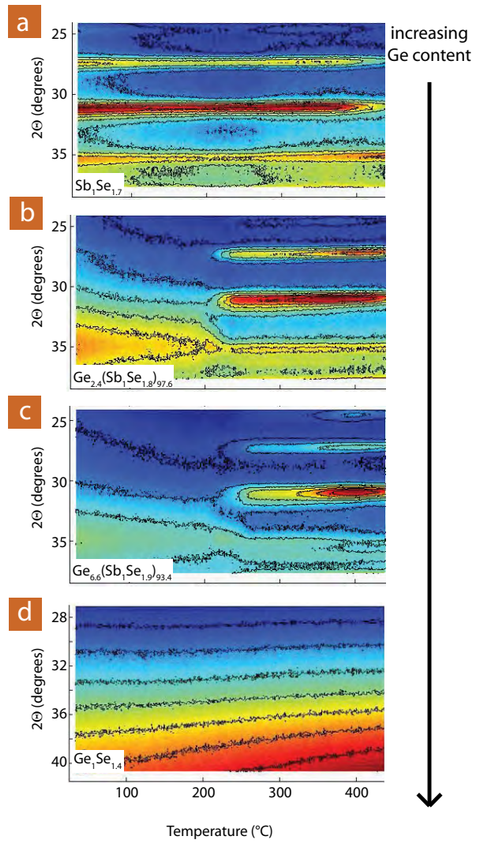
Researchers involved in the study included: Leyla Colakerol, Hae-Kyung Jeong, Lukasz Plucinski, Alex DeMasi, Timothy Learmonth, Per-Anders Glans, Shancai Wang, Yufeng Zhang, Kevin Smith, Tai-Chou Chen, and T. D. Moustakas (Boston University); T.D. Veal, L.F.J. Piper, P.H. Jefferson, and C.F. McConville (University of Warwick); and Alexei Fedorov (Advanced Light Source, Lawrence Berkley National Laboratory).

Funding was provided by the National Science Foundation, Army Research Office, and the Air Force Office of Scientific Research.

L. Colakerol, T. Veal, H. Jeong, L. Plucinski, A. DeMasi, T. Learmonth, P. Glans, S. Wang, Y. Zhang, et al., "Quantized Electron Accumulation States in Indium Nitride Studied by Angle-Resolved Photoemission Spectroscopy," *Phys. Rev. Lett.*, **97**: 237601 (2006).



Nanopatterns of GeSbSe phase-change material formed using a block copolymer template.



Thermal crystallization of GeSbSe films followed by temperature-dependent x-ray diffraction.

Solution-Based Approach for Phase-Change Material Deposition

Chalcogenide films with reversible amorphous-crystalline phase transitions have been commercialized as optically rewritable discs, and intensive effort is now focused on integrating them into electrically addressed nonvolatile memory devices (phase change random access memory, or PCRAM). While optical data storage is accomplished by laser-induced heating of local spots within continuous films, electronic memory requires integration of discrete nanoscale phase-change material features with read/write electronics. Currently, phase-change films are most commonly deposited by sputter deposition, and patterned by conventional lithography. However, at the NSLS, a team of IBM researchers is exploring an alternative, solution-phase deposition method for metal chalcogenide phase-change materials.

Solution processing of phase-change material is not only a low-cost, simple alternative to sputter deposition – it also offers potential advantages for patterning and eventual fabrication of PCRAM devices. In their study, the IBM researchers looked at solution processing of GeSbSe, a well-known phase change material.

Using characterization techniques at NSLS beamline X20C, the researchers demonstrated a versatile, solution-based, low-temperature approach made on the basis of the synthesis of soluble precursors, which can be combined to tune the composition and properties of the final material. Spin-on GeSbSe films show a tuneable crystallization temperature in the range of 200–250 °C and a minimum crystallization time of the order of 100 nanoseconds for melt-quenched material.

Their results, which were published in the April 8, 2007 edition of *Nature Materials*, are a promising indication that this process is capable of yielding materials of practical use for phase-change data storage. Solution deposition also enables facile filling of high-aspect-ratio vias, offering a potential advantage in device fabrication over conventional sputter-deposition methods. Next, the researchers plan to integrate spin-on materials into prototype PCRAM devices and to explore new applications for spin-on metal chalcogenide materials.

Researchers involved in the work include Delia Milliron, Simone Raoux, and Robert Shelby (IBM Almaden Research Center), and Jean Jordan-Sweet (IBM Watson Research Center).

D. Milliron, S. Raoux, R. Shelby, J. Jordan-Sweet, "Solution-Phase Deposition and Nanopattering of GeSbSe Phase-Change Materials," *Nat. Mater.*, **6**: 352-356 (2007).

Using SAXS to Investigate Multiwall Carbon Nanotube Films

At the NSLS, a team of researchers from the Massachusetts Institute of Technology showed that small-angle x-ray scattering (SAXS) is a powerful tool for investigating the morphologies of multiwall carbon nanotube (CNT) films. CNT films have attracted significant interest from the engineering community because of their remarkable thermal, electrical, and mechanical properties, and because they show promise for use in nanoelectronics, energy-absorbing foams, superhydrophobic films, and power applications. The team demonstrated the utility of SAXS for quantitative structural analysis of CNT films, indicating the potential to reveal new information about the CNT growth process, and relating variations in morphology to evolution of the catalyst and reaction conditions.

Multiwall CNT films can be grown by thermal chemical vapor deposition, resulting in films that range from vertically aligned to entangled and tortuous. The extent of CNT alignment can only be assessed qualitatively using scanning electron microscopy, but using NSLS beamline X27C, the researchers showed that it could be characterized quantitatively in various regions of the CNT film by examining the relative SAXS intensities as a function of azimuthal angle. Additionally, by fitting the SAXS data to a cylindrical form factor model, the average CNT diameter, and an estimate of the standard deviation, can be determined. The diameters determined through SAXS correspond well to high-resolution transmission electron microscopy (HRTEM) data. SAXS provides the advantage of sampling millions of CNTs in a single image, and also provides spatial resolution not accessible through HRTEM. The ability to characterize the CNTs as a function of position within the film allowed the researchers to observe a systematic increase in the diameter as a function of height from the substrate as well as smaller diameter CNTs near the edge, relative to the center of the film.

In order to provide insights into the fundamental mechanisms of the CNT growth process, the researchers are conducting systematic studies of the morphologies of the CNT forests as a function of variations in the growth conditions. Developing a complete understanding of this process is critical for enabling materials' structures and properties to be optimized and customized for specific applications. SAXS provides rich morphological information that is not accessible through conventional microscopy techniques. This type of analysis can easily be applied to single-wall CNTs, zinc oxide nanowires, and a variety of other similar systems.

The group's results were published in the March 30, 2007 and the August 2, 2007 editions of the *Journal of Physical Chemistry C*. Researchers include Eric Verploegen, Benjamin Wang, Ryan Bennett, Anastasios Hart, and Robert Cohen, all of MIT.

Funding was provided by the National Science Foundation, MIT's Institute for Soldier Nanotechnologies, the Fannie and John Hertz Foundation, and DURINT on Microstructure, Processing and Mechanical Performance of Polymer Nanocomposites.

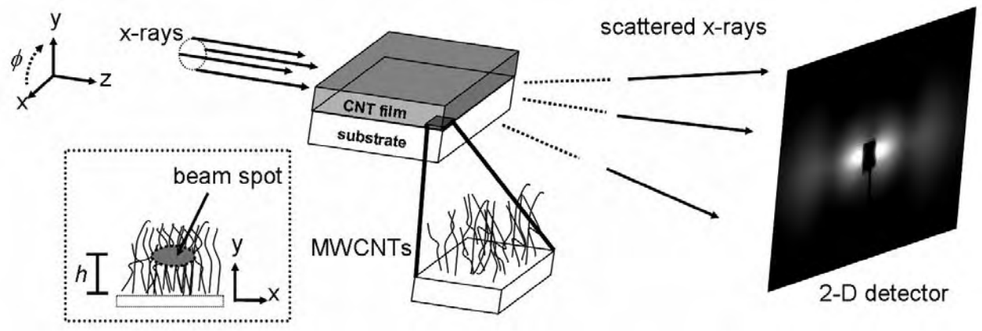
B. Wang, R. Bennett, E. Verploegen, A. Hart, R. Cohen, "Quantitative Characterization of the Morphology of Multiwall Carbon Nanotube Films by Small-Angle X-ray Scattering," *J. Phys. Chem. C,* **111** (16): 5859 (2007).

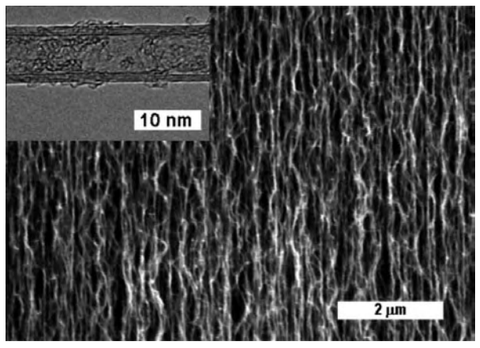
B. Wang, R. Bennett, E. Verploegen, A. Hart, R. Cohen "Characterizing the Morphologies of Mechanically Manipulated Multiwall Carbon Nanotube Films by Small-Angle X-ray Scattering," *J. Phys. Chem. C,* **111** (48): 17933 -17940 (2007).

Anastasios Hart

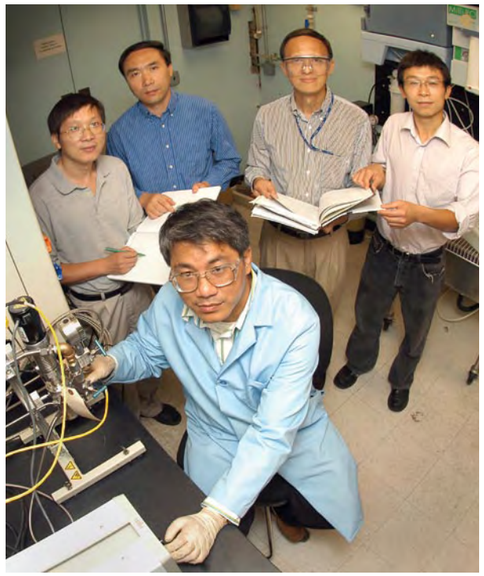
Eric Verploegen

(below) Schematic of the experimental setup for small-angle x-ray scattering of multiwall carbon nanotube forests. A motorized stage provides spatial resolution allowing for the morphology to be investigated as a function of position within the film.

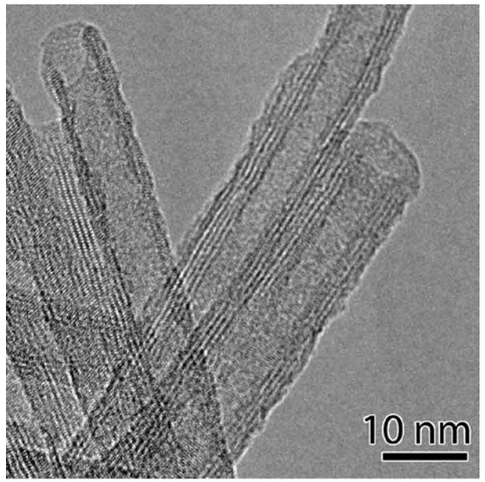


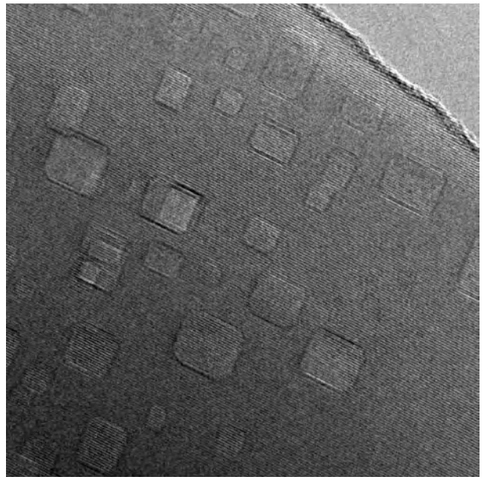


(left) Scanning electron microscopy (SEM) image of a vertically aligned multiwall carbon nanotube forest. Inset: transmission electron microscopy image of a single multiwall carbon nanotube. Small angle x-ray scattering (SAXS) provides quantitative characterization of the alignment, where only qualitative assessments of orientation can be made with SEM. SAXS measurements yield a "locally averaged" measurement of the CNT diameter, where TEM requires imaging of individual nanotubes.



Authors, (sitting) Wei-Qiang Han, (standing, from left) Lijun Wu, Zhenxian Liu, Yimei Zhu, and Wen Wen





Transmission electron micrographs of nanocavity-filled titanate nanorods (bottom) and iron-doped titanium oxide nanotubes (top). Both are being investigated as photocatalysts for reactions to produce hydrogen gas. The improved light-absorption of the nanocavity-filled nanorods also makes them ideal new materials for sunscreen.

Tiny Tubes and Rods Show Promise as Catalysts, Sunscreen

Scientists at Brookhaven have developed new ways to make or modify nanorods and nanotubes of titanium oxide, a material used in a variety of industrial and medical applications. The methods and new titanium oxide materials may lead to improved catalysts for hydrogen production, more efficient solar cells, and more protective sunscreens. The research is published in two papers, one in *Advanced Materials* (published August 22, 2007), and the other in the *Journal of Physical Chemistry C* (September 8, 2007).

In the first study, the scientists enhanced the ability of titanium oxide to absorb light.

"Titanium dioxide's ability to absorb light is one the main reasons it is so useful in industrial and medical applications," said Wei-Qiang Han, a scientist at Brookhaven's Center for Functional Nanomaterials (CFN) and lead author on both papers.

It is used as a photocatalyst for converting sunlight to electricity in solar cells and also has applications in the production of hydrogen, in gas sensors, in batteries, and in using sunlight to degrade some environmental contaminants. It is also a common ingredient in sunscreen.

Many scientists have explored ways to improve the light-absorbing capability of titanium oxide, for example, by "doping" the material with added metals. Han and his coworkers took a new approach. They enhanced the material's light-absorption capability by simply introducing nanocavities, completely enclosed pockets measuring billionths of a meter within the 100-nanometer-diameter solid titanium oxide rods.

The resulting nanocavity-filled titanium oxide nanorods were 25 percent more efficient at absorbing certain wavelengths of ultraviolet A (UVA) and ultraviolet B (UVB) solar radiation than titanium oxide without nanocavities.

"Our research demonstrates that titanium oxide nanorods with nanocavities can dramatically improve the absorption of UVA and UVB solar radiation, and thus are ideal new materials for sunscreen," Han said.

The cavity-filled nanorods could also improve the efficiency of photovoltaic solar cells and be used as catalysts for splitting water and also in the water-gas-shift reaction to produce pure hydrogen gas from carbon monoxide and water.

In the second paper, Han and his collaborators describe a new synthesis method to make iron-doped titanate nanotubes, hollow tubes measuring approximately 10 nanometers in diameter and up to one micrometer (one millionth of a meter) long. The scientists demonstrated that the resulting nanotubes exhibited noticeable reactivity in the water-gas-shift reaction.

"Although the activity of the iron-doped nanotubes was not as good as that of titanium oxide loaded with metals such as platinum and palladium, the activity we observed is still remarkable considering that iron is a much less expensive metal and its concentration in our samples was less than one percent," Han said.

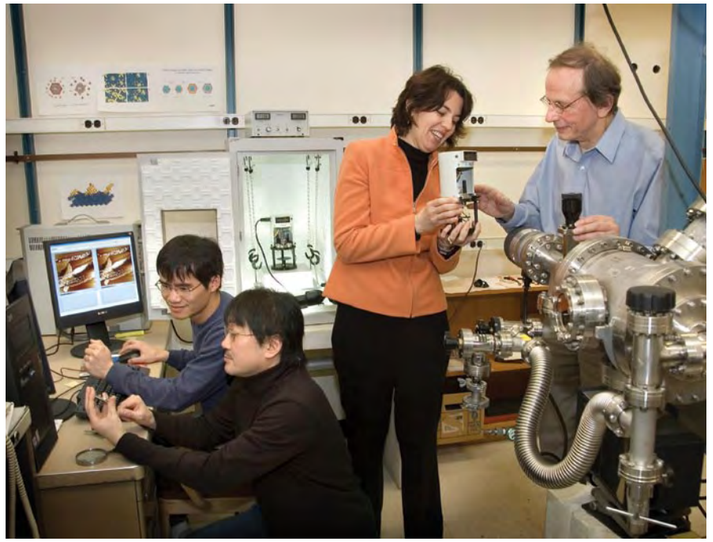
Materials developed in these studies were analyzed using transmission electron microscopy and various x-ray and infrared techniques at NSLS beamlines X7B and U2A.

This research, which has clear connections to improved energy technologies, was funded by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science.

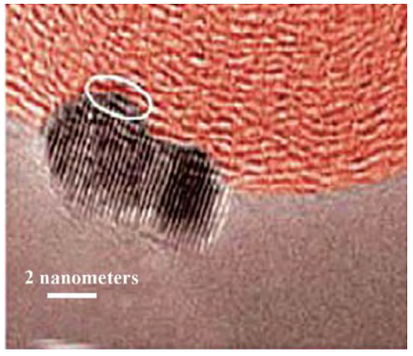
Collaborators on the Advanced Materials paper include Lijun Wu, Robert F. Klie, and Yimei Zhu, all of Brookhaven's CFN. For the Journal of Physical Chemistry paper, collaborators include Brookhaven chemists Wen Wen and Jonathan Hanson; Ding Yi, Mathew Maye, and Oleg Gang of the CFN; Zhenxian Liu of the Carnegie Institution of Washington; and Laura Lewis, formerly at the CFN and now at Northeastern University.

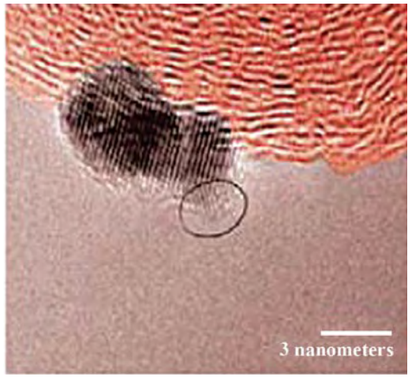
W. Han , W. Wen , Y. Ding, Z. Liu, M. Maye, L. Lewis, J. Hanson, O. Gang, "Fe-Doped Trititanate Nanotubes: Formation, Optical and Magnetic Properties, and Catalytic Applications," *J. Phys. Chem. C,* 111: 14339 (2007).

W. Han, L. Wu, R. Klie, Y. Zhu, "Enhanced Optical Absorption Induced by Dense Nanocavities Inside Titania Nanorods," *Advanced Materials,* 19: 2525-2529 (2007).



Clockwise, researchers Kotaro Sasaki, Junliang Zhang, Eli Sutter, and Radoslav Adzic view gold clusters on a single-crystal platinum surface using a scanning tunneling microscope.





The electron micrographs of a Au/Pt/C catalyst made by displacement of a Cu monolayer by Au. High-resolution images show atomic rows with spacings that are consistent with the Pt(111) single-crystal structure. A different structure in the areas indicated by the arrows is ascribed to the Au clusters.

Using Gold Clusters to Stabilize Platinum Electrocatalysts for Fuel Cells

Platinum is the most efficient electrocatalyst for accelerating chemical reactions in fuel cells for electric vehicles. In reactions during the stop-and-go driving of an electric car, however, the platinum dissolves, which reduces its efficiency as a catalyst. This is a major impediment for vehicle-application of fuel cells.

Now, Brookhaven scientists have overcome this problem. Under lab conditions that imitate the environment of a fuel cell, the researchers added gold clusters to the platinum electrocatalyst, which kept it intact during an accelerated stability test. This test is conducted under conditions similar to those encountered in stop-and-go driving in an electric car. The research is reported in the January 12, 2007 edition of the journal *Science*.

Brookhaven's Chemistry Department researchers Junliang Zhang, Kotaro Sasaki, and Radoslav Adzic, along with Eli Sutter from Brookhaven's Center for Functional Nanomaterials (CFN), authored the research paper.

"Fuel cells are expected to become a major source of clean energy, with particularly important applications in transportation," said coauthor Radoslav Adzic. "Despite many advances, however, existing fuel-cell technology still has drawbacks, including loss of platinum cathode electrocatalysts, which can be as much as 45 percent over five days, as shown in our accelerated stability test under potential cycling conditions. Using a new technique that we developed to deposit gold atoms on platinum, our team was able to show promise in helping to resolve this problem. The next step is to duplicate results in real fuel cells."

A hydrogen-oxygen fuel cell converts hydrogen and oxygen into water and, as part of the process, produces electricity. Platinum electrocatalysts speed up oxidation and reduction reactions. Hydrogen is oxidized when electrons are released and hydrogen ions are formed; the released electrons supply current for an electric motor. Oxygen is reduced by gaining electrons, and in reaction with hydrogen ions, water, the only byproduct of a fuel cell reaction, is produced.

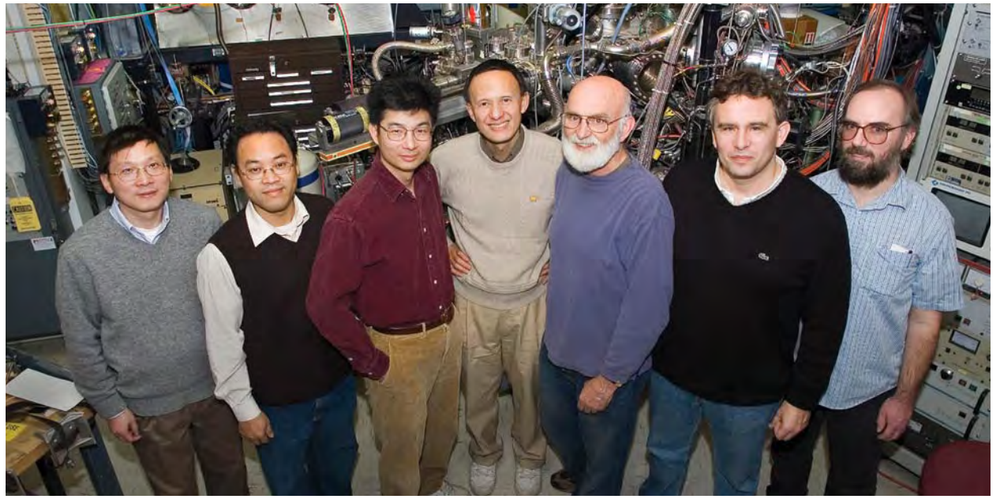
In the unique method developed at Brookhaven, the researchers displaced a single layer of copper with gold on carbon-supported platinum nanoparticles. After being subjected to several sweeps of 1.2 volts, the gold monolayer transformed into three-dimensional clusters. Using x-rays as probes at NSLS beamlines X11B, X18B, and X19A, a scanning transmission microscope at the CFN, and electrochemical techniques in the laboratory, the scientists were able to verify the reduced oxidation of platinum and to determine the structure of the resulting platinum electrocatalyst with gold clusters, which helped them to gain an understanding of the effects of the gold clusters.

In the Brookhaven experiment, the platinum electrocatalyst remained stable with potential cycling between 0.6 and 1.1 volts in over 30,000 oxidation-reduction cycles, imitating the conditions of stop-and-go driving.

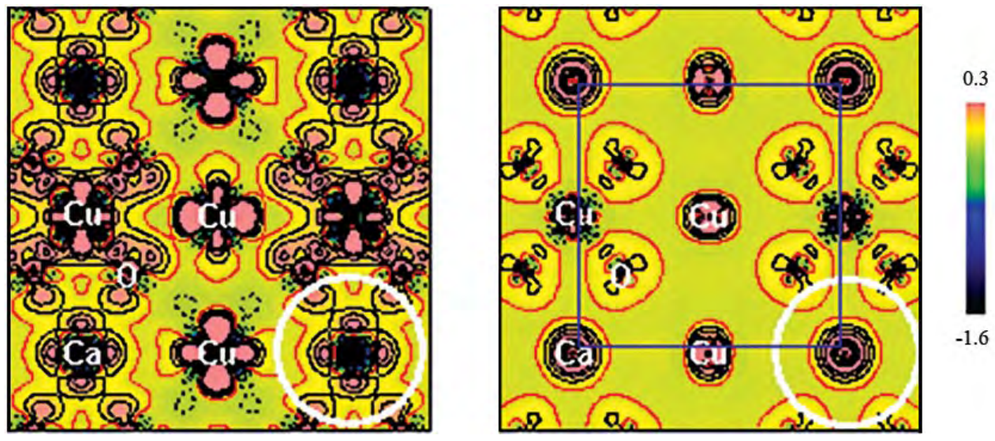
"The gold clusters protected the platinum from being oxidized," Adzic said. "Our team's research raises promising possibilities for synthesizing improved platinum-based catalysts and for stabilizing platinum and platinum-group metals under cycling oxidation/reduction conditions."

This research is funded through the U.S. Department of Energy's Hydrogen Program, which implements the President's Hydrogen Fuel Initiative, a five-year program that began in 2003 to sponsor research, development, and demonstration of hydrogen and fuel cell technologies. Specifically, the funding derived from DOE's Office of Basic Energy Sciences and its Office of Energy Efficiency and Renewable Energy.

J. Zhang, K. Sasaki, E. Sutter, R. Adzic, "Stabilization of Platinum Oxygen-Reduction Electrocatalysts Using Gold Clusters," *Science,* **315**: 220 (2007).



From left, Lijun Wu, Jincheng Zheng, Wei Ku, Yimei Zhu, Jonathan Hanson, Anatoly Frenkel, and Paul Northrup



The bonding-electron distribution of CCTO containing Cu, Ca, and O atoms. (left) Experimental observation extracted from structure factor measurements using combined electron and x-ray diffraction data. (right) DFT calculation based on the ideal crystal structure.

Exploring High-Dielectric Materials Through a New Route

Using integrated techniques at the NSLS and the electron microscopy facility in Brookhaven's Condensed Matter Physics and Materials Science Department, a team of researchers developed a powerful method for probing materials that could help further miniaturize microelectronic components.

As electronic devices continue to shrink, so must components such as capacitors. However, past a certain size, the silicon-based material that's traditionally used to manufacture the critical electronic circuits starts to leak current and no longer performs as it does at larger scales. To prevent this effect, researchers have been searching for materials with high dielectric constants (or high-k), which enable the use of even smaller capacitors within the electronic world. One of the best materials for this purpose is called CCTO, which is made of calcium, copper, titanium, and oxygen.

"CCTO was discovered a few years ago, but only recently it was found to have one of the highest dielectric constants ever measured," said Brookhaven physicist Yimei Zhu.

Materials with a dielectric constant greater than or equal to 7 usually qualify as high-k materials. At 100,000, the dielectric constant of CCTO far surpasses that benchmark. This intriguing property offers promising technological applications, such as the miniaturization of random access memories, as well as resonators and filters for microwave and wireless communications. There's just one problem: No one could figure out why this anomaly existed. In a recent study, Zhu's team set out to find an answer.

First, using quantitative electron diffraction on single crystals of CCTO to measure valence electron distribution, the scientists identified broken symmetry in the electronic density of the material. They then further explored this nanoscale disorder with extended x-ray absorption fine structure (EXAFS) at NSLS beamlines X11A and X15B, as well as with theory calculations.

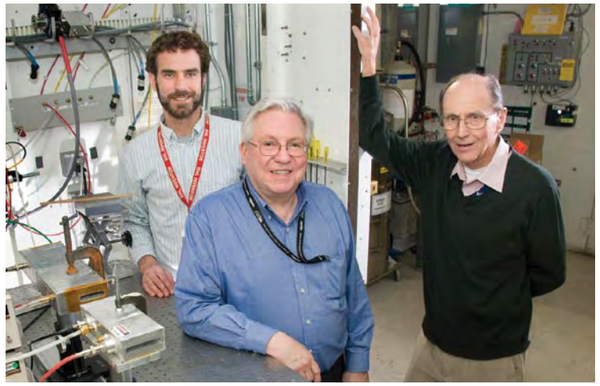
Specifically, the researchers found that a certain number of copper and calcium atoms switched positions within the material, which at least partially results in the high dielectric response. Their results were published in the July 20, 2007 issue of *Physical Review Letters*.

This type of nanoscale disorder was previously ignored by researchers in the field because of the lack of sufficient probing tools, Zhu said.

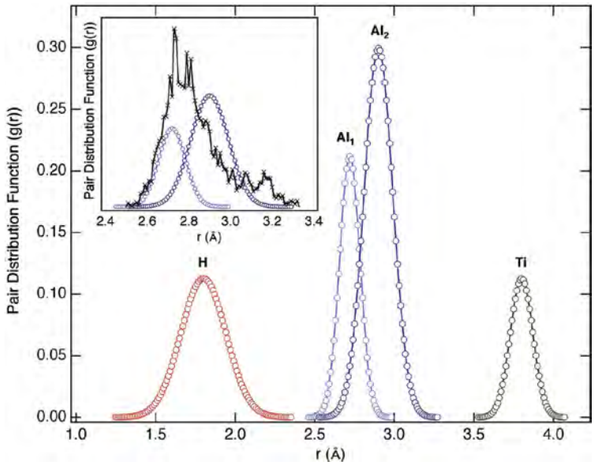
"Our study shows how integrated experiments can work to help solve a puzzling question and also to find a new route in searching for fascinating properties of materials – especially high-dielectric ones – by tailoring local disorder and orbital symmetry," he said.

Other researchers involved were Jincheng Zheng, Lijun Wu, Jonathan Hanson, Paul Northrup, and Wei Ku, all from Brookhaven; and Anatoly Frenkel, from Yeshiva University. The research was funded by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science.

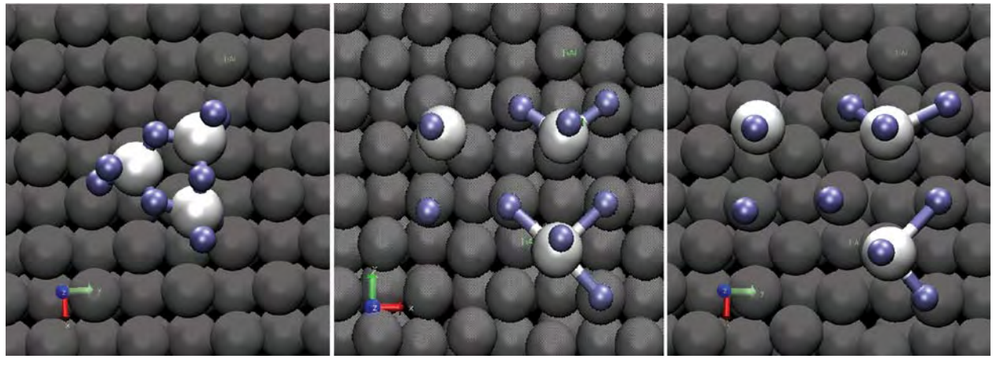
Y. Zhu, J. Zhang, L. Wu, A. Frenkel, J. Hanson, P. Northrup, W. Ku, "Nanoscale Disorder in $CaCu_3Ti_4O_{12}$: A New Route to Enhanced Dielectric Response," *Phys. Rev. Lett.*, **99**: 037602 (2007).



From left, Jason Graetz, James Muckerman, and James Reill



(left) Pair distribution function determined from EXAFS data on 2 mol % Ti-catalyzed $NaAlH_4$ (hydrogenated) and corrected for the phase shift. The inset shows a comparison of predicted and experimental distribution functions for Ti-Al pairs.



(below) Dissociation and diffusion of an Al_5H_9 cluster on an Al(001) surface showing formation of smaller fragments including a stable AlH_4 and AlH_3 cluster.

Science Highlights

Hydrogen-Mediated Metal Transport in Complex Metal Hydrides

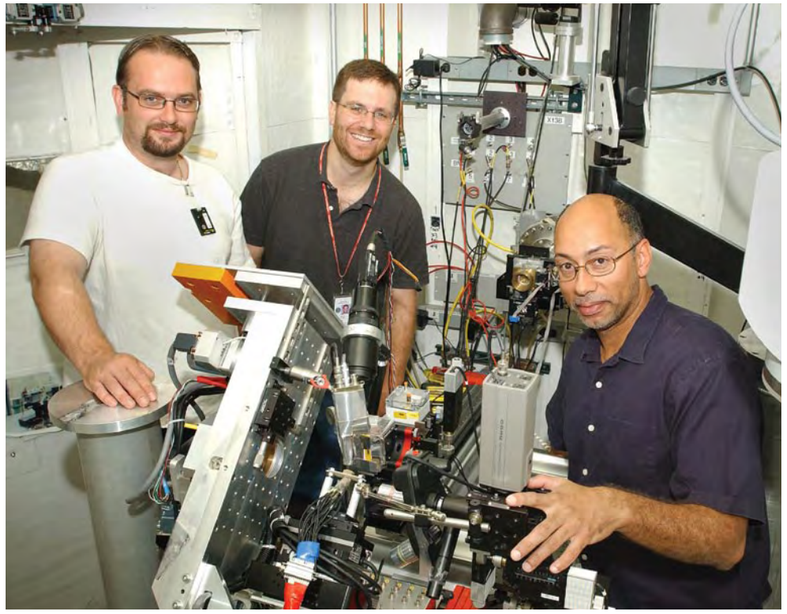
Hydrogen, the most abundant element in the universe, is high on the list of energy carriers that might one day be a replacement for petroleum. A major obstacle, however, is storing it safely and compactly. One promising approach is solid-state storage in the form of a metal hydride. This is often done with the assistance of a catalyst such as titanium. At the NSLS, a team of BNL researchers investigated the titanium environment and the role of metal catalysts in sodium alanate, a hydrogen-storage material composed of sodium and aluminum hydride.

The demonstration of reversible hydrogen cycling in Ti-catalyzed sodium aluminum hydride has generated considerable interest in complex metal hydrides. Since this discovery, a number of studies have looked at improving the catalytic effects and understanding the role of additives in H_2 cycling. However, the mechanism by which sodium aluminum hydride is activated in the presence of a small amount of a transition metal is still not well understood. One of the principle questions is "How do the Al atoms migrate the long distances required during the hydrogenation reaction?" In this study, the researchers investigated the atomistic transport mechanisms of the reversible complex metal hydrides using x-ray absorption spectroscopy at NSLS beamlines X19A and X9B along with first-principles calculations.

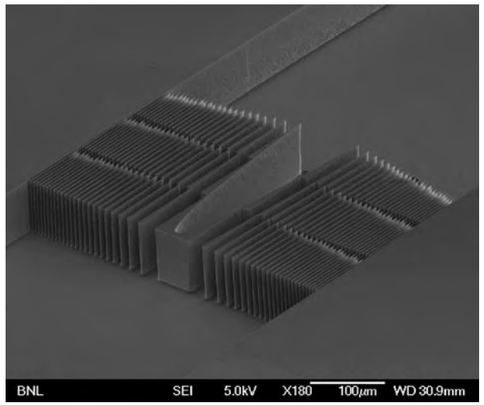
Their results, which were published in the August 10, 2006 edition of the *Journal of the American Chemical Society*, show titanium atoms near the surface, coordinated by a shell of aluminum. Simulations at 450 K revealed a similar local Ti environment. These results suggest that the role of Ti may be linked entirely to active catalytic sites in the metallic Al phase. The formation and migration of transient alane (AlH_x) species is proposed as a plausible mechanism for the long-range aluminum transport that occurs during hydrogen cycling. Therefore, maintaining well-dispersed Ti within the Al phase will be essential to mitigating capacity fade during cycling.

Researchers involved in this work included Jason Graetz, James Reilly, and James Muckerman, all of Brookhaven, Santanu Chaudhuri, now at Washington State University, and Alex Ignatov, now at Louisiana State University. Funding was provided by the U.S. Department of Energy Office of Basic Energy Sciences.

S. Chaudhuri, J. Graetz, A. Ignatov, J. Reilly, J. Muckerman, "Understanding the Role of Ti in Reversible Hydrogen Storage as Sodium Alanate: A Combined Experimental and Density Functional Theoretical Approach," *J. Am. Chem. Soc.*, **128**: 11404-11415 (2006).



The research team at NSLS beamline X13B: from left, James Ablett, Aaron Stein, and Kenneth Evans-Lutterodt.



A kinoform lens

Breaking the Barrier Toward Nanometer X-Ray Resolution

A team of researchers at Brookhaven has overcome a major obstacle for using refractive lenses to focus x-rays. This method will allow the efficient focusing of x-rays down to extremely small spots and is an important breakthrough in the development of a new, world-leading light source facility that promises advances in nanoscience, energy, biology, and materials research.

At the NSLS, the scientists exceeded a limit on the ability to focus "hard," or high-energy, x-rays known as the "critical angle." Their results are described online in the September 28, 2007 edition of *Physical Review Letters.*

The critical angle is the maximum angle that light can be deflected, or bent, by a single surface. Imagine a beam of laser light traveling toward a glass lens. Depending on the characteristics of the lens material and the angle at which the beam is pointed, the light can be refracted, that is, transmitted through the lens but deflected. However, when this light approaches the lens at angles less than the critical angle, the beam does not pass through the lens but is instead reflected. This results in a maximum deflection angle for light that passes through the lens.

The maximum deflection angle determines the minimum spot size to which x-rays can be focused. This poses a problem for researchers who are using x-rays to study molecules, atoms, and advanced materials at the nanoscale – on the order of billionths of a meter. Such small subjects require tightly focused beams.

"One measure of the quality of an x-ray optic is how small a focused spot it can make," said NSLS researcher Ken Evans-Lutterodt. "The problem is that nature does not allow a single lens to deflect the x-rays very much. This limits how small of a spot you can create, and this translates to some fuzziness in the image. To get a sharper image, you need a lens that's more able to deflect the x-rays."

In 2003, a trio of Brookhaven researchers – Evans-Lutterodt, Aaron Stein, and James Ablett – were the first to notice the critical angle limit while investigating the properties of a so-called kinoform lens for focusing hard x-rays. This efficient type of refractive lens is similar to those found in lighthouses. The research team proposed a solution to the critical angle problem of a compound kinoform lens, and both the problem and proposed solution were also suggested later by other researchers in the field.

In the current publication, the researchers implemented their idea by creating a compound lens from a series of four kinoform lenses placed one after the other. Using this setup at NSLS beamline X13B, they showed that the critical angle can be surpassed with hard x-rays, while still focusing like a single lens.

"Thanks to the excellent fabrication resources at Brookhaven's Center for Functional Nanomaterials and at Alcatel-Lucent, we are able to fabricate the lenses to the precision required," Stein said.

This is an important step for the National Synchrotron Light Source II (NSLS-II), a state-of-the-art synchrotron facility that will produce x-rays up to 10,000 times brighter than those generated by the current NSLS and could lead to advances such as alternative-energy technologies and new drugs for fighting disease. One of the major goals of the facility is to probe materials and molecules with just one-nanometer resolution – a capability needed to study the intricate mechanisms of chemical and biological systems.

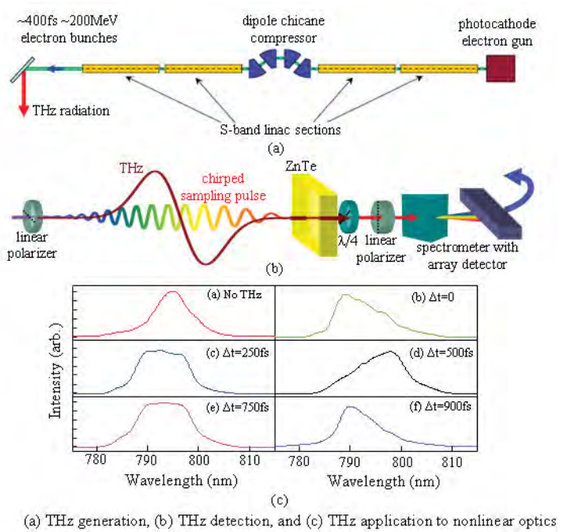
"Without exceeding the critical angle, the refractive lens resolution would be limited to 24 nanometers or more," Ablett said. "Even though in this experiment we just barely exceeded this limit, we've shown that it can be done. This is just the first step."

Next, the researchers will measure the resolution their new lens system produces, and will continue to fabricate and test optics that push further beyond the critical angle, and closer to the one-nanometer benchmark.

Natasha Bozovic, from San Jose State University, also collaborated on this research. Funding was provided by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science.

K. Evans-Lutterodt, A. Stein, J. Ablett, N. Bozovic, A. Taylor, D. Tennant, "Using Compound Kinoform Hard-X-Ray Lenses to Exceed the Critical Angle Limit," *Phys. Rev. Lett.*, **99**: 134801 (2007).

(above) Authors, from left, Dario Arena, Xijie Wang, Yuzhen Shen, Larry Carr, Takahiro Watanabe, Boyzie Singh, James Murphy, and Thomas Tsang at the Source Development Lab.



(a) THz generation, (b) THz detection, and (c) THz application to nonlinear optics

Coherent synchrotron THz generation, detection, and application to nonlinear optics

Researchers Produce Firsts with Bursts of Light

BNL researchers have generated extremely short pulses of light that are the strongest of their type ever produced and could prove invaluable in probing the ultra-fast motion of atoms and electrons. The scientists also made the first observations of a phenomenon called cross-phase modulation with this high-intensity light – a characteristic that could be used in numerous new light source technologies.

The work, which was done at Brookhaven's Source Development Laboratory, an offshoot of the Lab's National Synchrotron Light Source (NSLS), is described online in the July 23, 2007, edition of *Physical Review Letters*.

The light pulses used were in the terahertz (THz) range of the broad electromagnetic spectrum, found between the microwave and infrared range. Scientists send tight bunches of electrons at nearly the speed of light through a magnetic field to produce THz radiation at a trillion cycles per second — the terahertz frequency that gives the light its name and that makes them especially valuable for investigating biological molecules and imaging, ranging from tumor detection to homeland security.

The Brookhaven team is looking to expand the potential uses for this type of light by increasing the strength of individual THz pulses, a longtime goal for scientists in the field. By slamming an electron beam from an accelerator into an aluminum mirror, the researchers produced 100 megajoule (100 megawatt) single-cycle pulses – the highest energy ever achieved to date with THz radiation. For comparison, 100 megawatts is about the output of a utility company's electrical generator.

The combination of this newfound strength with ultra-fast pulses provides researchers with a powerful new tool to study the movement of a material's electrons (which zip around at the femtosecond, or quadrillionth of a second, timescale) or atoms (which move at the picosecond, or trillionth of a second, timescale).

"The goal is really to understand the properties of materials," said NSLS researcher Yuzhen Shen, the lead author of the paper. "One might ask what happens in a solid when light, electricity, or sound goes through it, and it's all related to atoms in a crystal wiggling around or the movement of electrons. So the effort surrounding ultra-fast pulses is going into making tools to probe the real fundamental properties of materials on the scales at which they move."

Using this strong light, researchers can "kick" molecular processes such as catalysis or electronic switching (important for developing data storage media) into action and watch their mechanisms on a very short timescale.

The team also found something surprising: the intensity of their THz pulses is so great that they introduce so-called "nonlinear optical effects," specifically, a phenomenon known as cross-phase modulation.

"When you pull on a spring, if you pull twice as hard, it stretches twice as much," said NSLS researcher Larry Carr. "But there's a limit where if you pull twice as hard, the spring doesn't move anymore. That's when it's called nonlinear. The same thing happens in materials. You let these short pulses pass through a material, and they stress it and pull some of the charges apart so they don't act in a linear manner."

As a result, the researchers can manipulate both the ultra-fast THz pulses and the material they interact with. Some of the simplest examples include changing the color of the light or turning the material into a focusing lens.

This is the first time cross-phase modulation has been observed in single-cycle THz pulses. Learning how to control this characteristic could lead to even more light source technologies.

This research was supported by the Office of Basic Energy Sciences within the U.S. Department of Energy's Office of Science, the Office of Naval Research, and Brookhaven's Laboratory Directed R&D funds.

Y. Shen, T. Watanabe, D. Arena, C. Kao, J. Murphy, T. Tsang, X. Wang, G. Carr, "Nonlinear Cross-Phase Modulation with Intense Single-Cycle Terahertz Pulses," *Phys. Rev. Lett.*, **99**: 043901 (2007).

"The NASA Stardust mission produced one of the most notable highlights in 2007, where tiny particles were probed with four different NSLS microscopes and produced five *Science* papers."

Lisa Miller
Life and Environmental Sciences Division Head

Science Highlights

It's been an extremely productive year for research conducted by NSLS users and staff in the fields of structural biology, biomedical imaging, geological and environmental sciences, and soft condensed matter and biophysics. The community represents almost two-thirds of the NSLS users, and its publications continue to be of high impact in premier scientific journals like *Science, Nature, Physical Review Letters*, and *PNAS*. This year's Activity Report briefly describes some of the 2007 science highlights, while many more highlights can be found on the CD in the back of this book and on the NSLS website.

Undoubtedly, one of the most notable highlights from this year in earth and environmental sciences was the series of five papers published in *Science* on results of the comet 81P/Wild 2 samples collected by the NASA Stardust mission that returned to Earth in early 2006. This large body of work utilized infrared, hard x-ray, and soft x-ray microprobes on four different NSLS beamlines, as well as beamlines at other synchrotrons worldwide. One of the main findings of the study is that the materials from which our solar system were made must have undergone a considerable amount of mixing while the sun and planets were forming. The comet dust was found to contain a wide variety of minerals, as well as organic materials. Some of these minerals and organics look similar to those seen in primitive types of meteorites, but both the minerals and the organics show the presence of some new materials not previously seen in meteorites.

NSLS users also published one of *Discover Magazine's* top 100 science stories of 2007 – a paper by Garai, *et al*. that investigated the extraterrestrial origin of carbonado diamonds using infrared microspectroscopy. In environmental sciences, Huggins *et al*. used x-ray absorption spectroscopy (XAS) to study the speciation of arsenic in fly-ash from coal-burning plants in order to understand its toxicity. Also through XAS, "green chemistry" took a big step forward in 2007 when de Oliveira, *et al*. revealed a structural intermediate for an Fe-TAML catalyst that is responsible for the catalyst's ability to destroy pollutants in water by accelerating cleansing reactions with hydrogen peroxide.

Structural biology researchers had another impressive year. Crystal structures of the catalytic domain of the hepatitis C virus NS2-3 protease (Lorenz, *et al*.) and peptide inhibitors of HIV-1 entry into the cell (Welch, *et al*.) were solved with macromolecular crystallography. Membrane-bound proteins continue to be a challenge for structure determination, but success was achieved by Wang, *et al*. when they solved the structure of a rhomboid family intramembrane protease.

In biomedical imaging, infrared microspectroscopy was used to show that the stiffness of bone lags considerably behind the process of bone mineralization in growing mice (Miller, *et al*.).

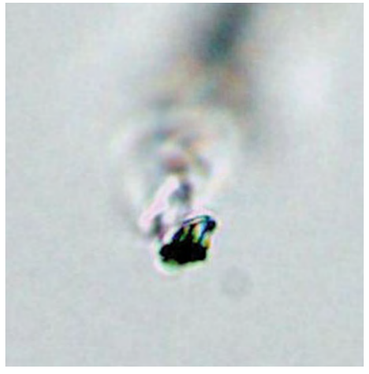
In the field of soft condensed matter and biophysics, the mechanisms behind the ordering and self-assembly of materials were studied in a wide range of systems in 2007. For example, Subburaman, *et al*. used x-ray scattering to demonstrate templated biomineralization of calcium carbonate on self-assembled protein fibers while Douglas *et al.* studied the forces behind self-assembly in organosilane monolayers using XAS. In addition, the unique process of "surface freezing" of thin oil films on water was examined using surface x-ray scattering techniques (Sloutskin, *et al*.).

Finally, dramatic advances in x-ray fluorescence imaging detectors were also demonstrated in 2007 by Ryan, *et al*. who have designed new silicon-based array detectors, electronics, and software that are able to collect data and analyze data 2-3 orders of magnitude faster than current systems. With a finite amount of beam time for all users, this will certainly be a welcomed added capability to x-ray fluorescence microprobe beamlines worldwide.

Life and Environmental Sciences Introduction

From left, George Flynn (SUNY Plattsburgh), Lindsay Keller (NASA), Larry Carr (NSLS), and Randy Smith (NSLS) examine samples from the Stardust mission at beamline U10A.

Composite Stardust image of comet Wild 2 (Photo courtesy of NASA)



Stardust particle embedded in the aerogel collector (Photo courtesy of NASA)

NSLS Scientists Reach for the Stars(dust)

A research project at the NSLS has turned to dust – star dust, that is. After months of studying particles collected from a comet passing inside Jupiter's orbit, a group of NSLS users and scientists has finished its preliminary examination on the dust, revealing details that might help explain the beginning of the solar system.

As part of an international team of more than 175 members, NSLS users and scientists used x-ray, infrared, and ultraviolet light to study the chemical composition and properties of the extremely small dust particles, most less than 15 micrometers in diameter. The diameter of a human hair, in comparison, is about 50 micrometers. Locked within the particles, which were collected from the comet Wild 2 by the NASA Stardust spacecraft, is unique chemical and physical information that provides a record of the formation of the planets and the materials from which they were made.

Working with about a picogram of dust, one trillionth of a gram, the scientists studied the elemental composition, organic materials, and mineralogy and petrology of the particles at four NSLS beamlines – X26A, X1A1, U10A, and U10B. Their findings, combined with those from other synchrotrons and institutions, were published in five of the seven Stardust papers in the December 15, 2006 issue of the journal *Science.*

The first samples from Stardust arrived at the NSLS in February 2006, suspended within "aerogel," a silicon-based, sponge-like material used to capture the particles in space. Once the particles were extracted from the gel, a powerful x-ray imaging device was used to collect detailed images of some of the smallest particles as well as to determine their elemental makeup. In particular, the scientists looked for the element carbon, which can indicate that the particles contain organic compounds — compounds that may have formed at the birth of our solar system.

Infrared light was used to identify specific minerals within the particles, as well as identify any organic compounds that were detected. Unlike x-ray methods, the information collected using these infrared techniques can be compared with the astronomical observations of distant interstellar dust clouds, including those involved with the formation of planetary systems like ours.

One of the main findings of the study is that the materials from which our solar system formed must have undergone a considerable amount of mixing while the sun and planets were forming.

"The common perception is that comets come from these really cold regions of the solar system with lots of ice," said University of Chicago geochemist Tony Lanzirotti, who worked on multiple aspects of the analysis. "But we found minerals like olivine, high-temperature minerals that on Earth are formed in magma. It really says a lot about the violent early history of the solar system, where you have high-temperature phases being mixed with cold regions of outer space in a very rapid manner."

The comet dust was found to contain a wide variety of minerals, as well as organic materials. Some of these minerals and organics look similar to those seen in primitive types of meteorites, but both the minerals and the organics show the presence of some new materials not previously seen in meteorites.

One of the biggest challenges was accounting for the deterioration of the dust particles as they crash-landed into the aerogel at 14,000 miles per hour.

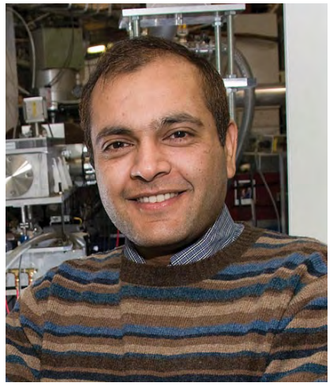
"They're looking at all the little components that were left behind after the particle hit the aerogel, and in the end, they want to get the composition of the original particle," said NSLS physicist Larry Carr. "The particles are a composite with parts getting ripped off and left behind in the form of a debris trail. It would be like finding a dead body in the woods, along with footprints, pieces of cloth, and hair, and trying to figure out who the victim was."

Now that the preliminary examination is completed, the samples will be made available to the general scientific community for more detailed study, possibly at the NSLS, where this small amount of dust has caused a great deal of attention.

D. Brownlee, et al; *Science,* **314**: 1711-1716 (2006); S. Sandford, et al., *Science,* **314**: 1720-1724 (2006); L. Keller, et al., *Science,* **314**: 1728-1731 (2006); G. Flynn, et al, *Science*, **314**: 1731-1735 (2006); M. Zolensky, et al., *Science,* **314**: 1735-1739 (2006).

Stephen Haggerty (left) and Jozsef Garai

Sandeep Rekhi



Mark Chance

A black, or carbonado, diamond

Diamonds from Outer Space: Geologists Discover Origin of Earth's Mysterious Black Diamonds

If indeed "a diamond is forever," the most primitive origins of Earth's so-called black diamonds were in deep, universal time, geologists have discovered. Black diamonds came from none other than interstellar space.

In a paper published on December 20, 2006 in *Astrophysical Journal Letters*, scientists Jozsef Garai and Stephen Haggerty of Florida International University, along with Case Western Reserve University researchers Sandeep Rekhi and Mark Chance, claim an extraterrestrial origin for the unique black diamonds, also called carbonado diamonds.

The researchers used infrared light to analyze the diamonds' elemental makeup at NSLS beamline U2B, which is operated by the Case Center for Synchrotron Biosciences, of Case Western Reserve University.

"Trace elements critical to an 'ET' origin are nitrogen and hydrogen," said Haggerty. The presence of hydrogen in the carbonado diamonds indicates an origin in a hydrogen-rich interstellar space, he and colleagues believe.

The term carbonado was coined by the Portuguese in Brazil in the mid-18th century; it's derived from its visual similarity to porous charcoal. Black diamonds are found only in Brazil and the Central African Republic.

"Conventional diamonds are mined from explosive volcanic rocks [kimberlites] that transport them from depths in excess of 100 kilometers to the Earth's surface in a very short amount of time," said Sonia Esperanca, program director in the National Science Foundation's Division of Earth Sciences, which funded the research. "This process preserves the unique crystal structure that makes diamonds the hardest natural material known."

From Australia to Siberia, from China to India, the geological settings of conventional diamonds are virtually identical, said Haggerty. None of them are compatible with the formation of black diamonds.

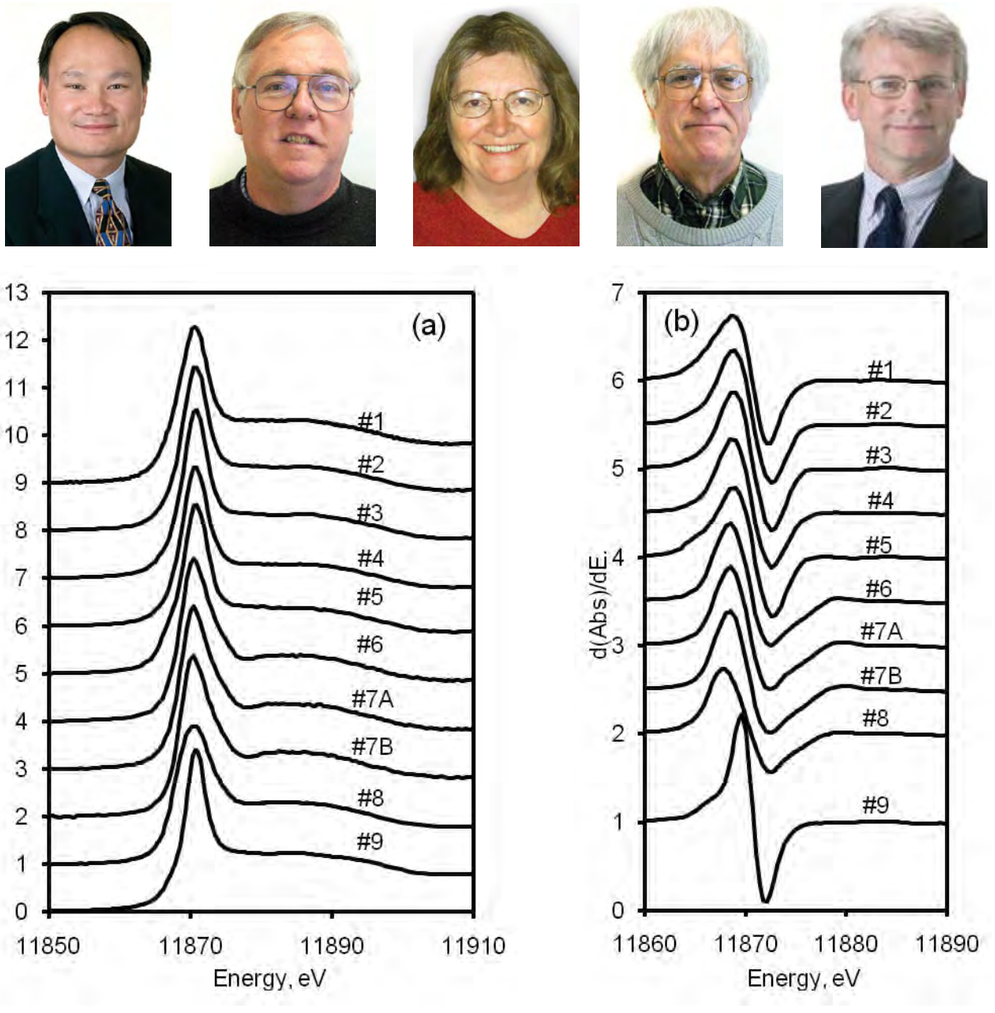
Approximately 600 tons of conventional diamonds have been mined, traded, polished and adorned since 1900, "but not a single black/carbonado diamond has been discovered in the world's mining fields," Haggerty said.

The new data support earlier research by Haggerty showing that carbonado diamonds formed in stellar supernovae explosions. Black diamonds were once the size of asteroids, a kilometer or more in diameter when they first landed on Earth.

J. Garai, S. Haggerty, S. Rekhi, M. Chance, "Infrared Absorption Investigations Confirm the Extraterrestrial Origin of Carbonado Diamonds," *Astrophys. J.*, **653**: L153-L156 (2007).

--Cheryl Dybas, National Science Foundation

From left, Paul Chu, Frank Huggins, Constance Senior, Gerald Huffman, and Ken Ladwig



Arsenic XANES (a) and derivative XANES (b) spectra for 10 fly-ash samples from full-scale pulverized coal combustion plants in North America. Samples 1-5 and 9 were derived from plants burning eastern U.S. bituminous coals that are rich in sulfur and iron. In contrast, samples 6-8 were derived from plants burning western subbituminous coals that are low in sulfur and rich in calcium. The spectra show that the arsenic is present in the fly-ash principally as arsenate species. Also, the XANES spectra for arsenic, as well as for selenium (not shown), exhibit systematic differences that reflect the type of coal. The spectrum for sample #9 was collected using a Si(220) monochromator crystal set and exhibits noticeably better resolution than the other nine spectra, which were collected using a Si(111) monochromator crystal set.

Investigating Arsenic and Selenium in Fly Ash From Coal Plants

Arsenic and selenium – two of the most volatile and potentially hazardous elements to be released from commercial coal-burning plants – are also found in large quantities in the byproduct of the electricity-producing process. This fine residue, known as fly ash, is produced in large quantities as coal is burned and can either be used in "green" applications such as concrete, or disposed of in landfills and tailings ponds. To learn more about how these potentially toxic elements are released when submerged in water, a team of researchers used the NSLS to determine the basic characteristics of selenium and arsenic in fly ash samples collected from several plants in the United States and Canada.

"A huge amount of electricity is generated from coal, which means huge amounts of coal combustion and waste materials," said University of Kentucky researcher Frank Huggins. "About 10-20 percent of the mass of the coal comes out as fly ash, and there are some uses for the material, but most of it ends up in ponds. Ultimately, we want to find out how much of these trace elements leach out and how far they go."

Using x-ray adsorption fine structure spectroscopy (XAFS) at NSLS beamline X18B and at the Stanford Synchrotron Radiation Laboratory, Huggins and his team of researchers determined the oxidation states and speciation of selenium and arsenic in samples from coal-fired utility plants burning a range of coals. These factors primarily determine the elements' toxicity and how easily they escape from the material, Huggins said.

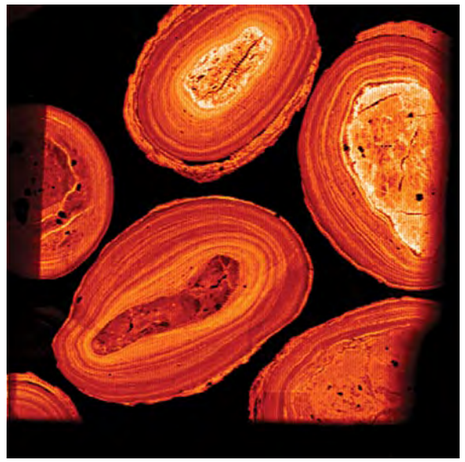
"Generally, the higher the oxidation state of the element, the more soluble it is, and the easier it gets into the water supplies," he said.

After examining 10 samples of two types of coal – eastern United States bituminous (Fe-rich) and western North American sub-bituminous (Ca-rich) – the researchers discovered that selenium is found predominantly as Se(IV) and arsenic is found as As(V). Similar spectral details were observed for both arsenic and selenium in the two different types of fly ash, suggesting that a major component (possibly Fe in bituminous coal or Ca in sub-bituminous coal) controls the capture of these elements by fly ash during combustion. The results were published in the May 1, 2007 issue of *Environmental Science & Technology*.

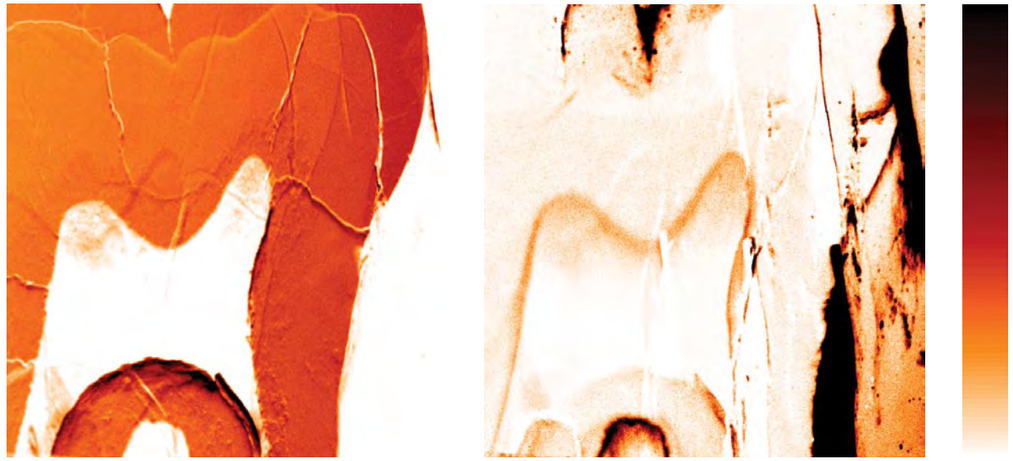
The XAFS work performed by the research team, which also includes Constance Senior (Reaction Engineering International, Salt Lake City, Utah), Gerald Huffman (University of Kentucky), and Paul Chu and Ken Ladwig (Electric Power Research Institute, Palo Alto, CA), is meant to complement another study on arsenic and selenium fly ash leachability.

This work was funded by Electric Power Research Institute.

F. Huggins, C.Senior, P.Chu, K.Ladwig, G.Huffman, "Selenium and Arsenic Speciation in Fly Ash from Full-Scale Coal-Burning Utility Plants," *Environ. Sci. Tech.*, **41**: 3284-3289 (2007).



Iron-oxide nodules imaged using the 32-element prototype detector array at NSLS beamline X27A (12x12 mm^2, 375x375 pixels at 32 ms/pixel).



Calcium (left) and lead in a 14th century tooth imaged using the 32-element prototype detector array at NSLS beamline X27A. The color legend distinguishes high (top) from low concentrations. The tooth was supplied by Rudiger Brenn, University of Freiberg (12x12 mm^2, 2000x2000 pixels at 6 ms/pixel, 6.7 hours acquisition).

Boosting Speed, Precision of X-Ray Fluorescence Microprobes

Combining large, high-resolution detector arrays with an advanced analysis technique, researchers from the NSLS, BNL's Instrumentation Department, and Australia's Commonwealth Scientific and Industrial Research Organization (CSIRO) have developed an x-ray fluorescence microprobe system that will be about 1,000 times faster than previous methods.

X-ray fluorescence is a powerful technique generally used in the environmental and geological sciences for measuring trace element concentrations in a sample. Typically, a very tiny x-ray spot is focused on a sample, which ionizes electrons from the material's atoms. These excited atoms relax, filling the vacancies, and in doing so, emit x-rays at energies characteristic of specific elements. However, scientists can only determine the elements present in the portion of the sample that's exposed to the x-ray spot. To get an idea of the entire sample's chemical composition, the spot must be manually moved from one location to another – a process that can take many hours to produce low-resolution maps of just a few thousand pixels.

"You have to stop and start and it's a pain in the neck," said NSLS physicist Peter Siddons. "So we came up with a method that would allow us to scan the scheme continuously along a line. The exposure time is just a few milliseconds at each point so, like previous 'on-the-fly' scanning systems, it never really stops moving. However, we collect full spectral data as we go. That not only makes it fast, but allows a high-quality quantitative analysis as well."

The new scanning method, which has been tested at NSLS beamline X27A, also includes two other improvements upon the old technique. The first is the use of a multi-element detector, which incorporates many small detectors (32 in the test run) into one device, instead of using one large detector.

"These experiments are limited not only by mechanical speed, but also by how many photons you're detecting from the x-ray spot," Siddons said. "There are always more than enough photons in the synchrotron spectrum to excite these samples and saturate the detector. By making the detectors smaller and making more of them, we can collect more signal at each position in the map, or do it much faster."

The final pieces of the new fluorescence microprobe system are advanced data analysis techniques that can handle the increased data processing speed and map the x-ray energies in real time. Led by physicist Chris Ryan, scientists at CSIRO developed software and hardware to unfold the signals from chemical elements at up to 100 million events per second.

"The combination of large multi-element detectors, fast-scan mapping, and real-time processing means we can tackle challenging new approaches in a realistic timescale," Ryan said. "Techniques such as chemical-state imaging and fluorescence tomography will now become practical tools, made possible with this massive increase in speed and throughput."

In the first demonstration of this technique, which was detailed in the *2006 Goldschmidt Conference Abstracts*, the research team produced a 4 mega pixel image of a 14th century tooth and looked for lead accumulation that might indicate the presence of lead poisoning hundreds of years ago.

"The image was produced in just about six hours, a task that would normally take days," Siddons said. "No one ever did a 4 mega pixel image before because it would just take too long."

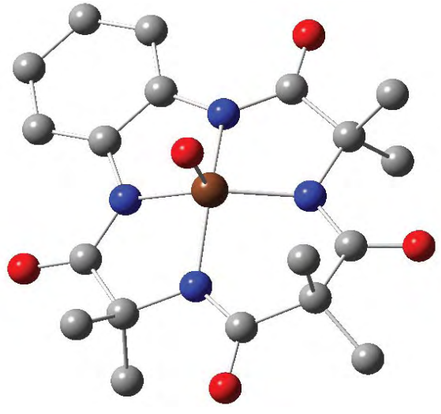
Now, the group is building two 400-element detectors: one for the NSLS and one for the Australian Synchrotron. They are expected to produce scanning images 1,000 times faster than the traditional x-ray fluorescence technique.

Other scientists involved in the research include Gareth Moorhead, Paul Dunn, Robin Kirkham, Robert Hough, and Barbara Etschmann, all of CSIRO; and Angelo Dragone and Gianluigi De Geronimo, from BNL. This research was funded by the U.S. Department of Energy and the CSIRO Emerging Science program.

C. Ryan, D. Siddons, G. Moorhead, P. Dunn, R. Kirkham, A. Dragone, G. De Geronimo, R. Hough, B. Etschmann, "The Next Generation of Synchrotron Fluorescence Imaging for Geological Applications," *Geochim. Cosmochim. Acta,* **70**: A550 (2006).

Terry Collins

Lawrence Que



The structure of the Fe(V)-oxo complex (brown, iron; red, oxygen; blue, nitrogen; and gray, carbon.) For clarity, hydrogen atoms are not shown.

Revealing a Key Intermediate in Green Oxidation Catalysis

As industrial processes release pollutants such as dyes, pesticides and other harmful chemicals into the Earth's water, a team of researchers is turning to a class of "green" catalysts for cleanup. Developed at Carnegie Mellon University in 1995, Fe-TAMLs – short for iron tetra-amido macrocyclic ligand activators – destroy a multitude of pollutants in water by accelerating cleansing reactions with hydrogen peroxide. Since then, researchers have worked to determine exactly how these processes work, and recently, they figured out another piece of that puzzle. Through studies that included extended x-ray absorption fine structure (EXAFS) investigations at the NSLS, researchers from CMU and the University of Minnesota have isolated and characterized a species thought to be a key intermediate in the Fe-TAML reaction with peroxides and oxygen.

The results of the study, published in the February 9, 2007 issue of *Science*, lend weight to the theory that Fe-TAML catalysts work by separating hydrogen peroxide into water and an oxygen atom that is bound to the iron of the Fe-TAML. The resulting Fe-TAML-oxo complex then destroys undesirable molecules, rendering them less toxic and often proceeding to mineralization or near-mineralization. The inventor of these extremely active catalysts, CMU's Terry Collins, describes Fe-TAML reactions as "fire in water," because the activated peroxide carries out chemistry on many (but not all) carbon-containing compounds where the products resemble those of combustion.

Because of this high reactivity, the catalyst's developers had to build ligand systems that allow for the formation of iron-oxo complexes from the Fe-TAML activators without being rapidly destroyed by the highly reactive intermediates they enable.

The stable form of the iron used in the Fe-TAML starting catalyst complex is Fe(III), the state of iron found naturally in rust. However, during the reaction between peroxides and an Fe-TAML activator, the iron's oxidation state has been shown to pass through an Fe(IV) species and then proceed onto an Fe(V) species. This Fe(V)-oxo complex is thought to be a key reactive intermediate in Fe-TAML oxidation catalysis.

"For about the last 10 years, I've been convinced that the Fe(V)-oxo group existed," said Collins, the director of the Institute for Green Oxidation Chemistry at CMU's Mellon College of Science. "Proving it was another matter."

Atoms with high oxidation states, such as Fe(IV) and Fe(V), are difficult to trap because they're so reactive. In order to "catch" the Fe(V)-oxo group, the researchers set up a system in an organic solvent at a very low temperature (about –60 °C) because water's high freezing point prevented its use. They then titrated it in a very reactive peroxide.

Using x-ray absorption spectroscopy at NSLS beamline X3B, University of Minnesota researcher Lawrence Que's group combined with Collins' group and CMU's Mössbauer, EPR, and theory group (led by Eckard Münck) to reveal the long-sought chemical and electronic properties of the intermediate. This is the first characterization of an Fe(V)-oxo complex.

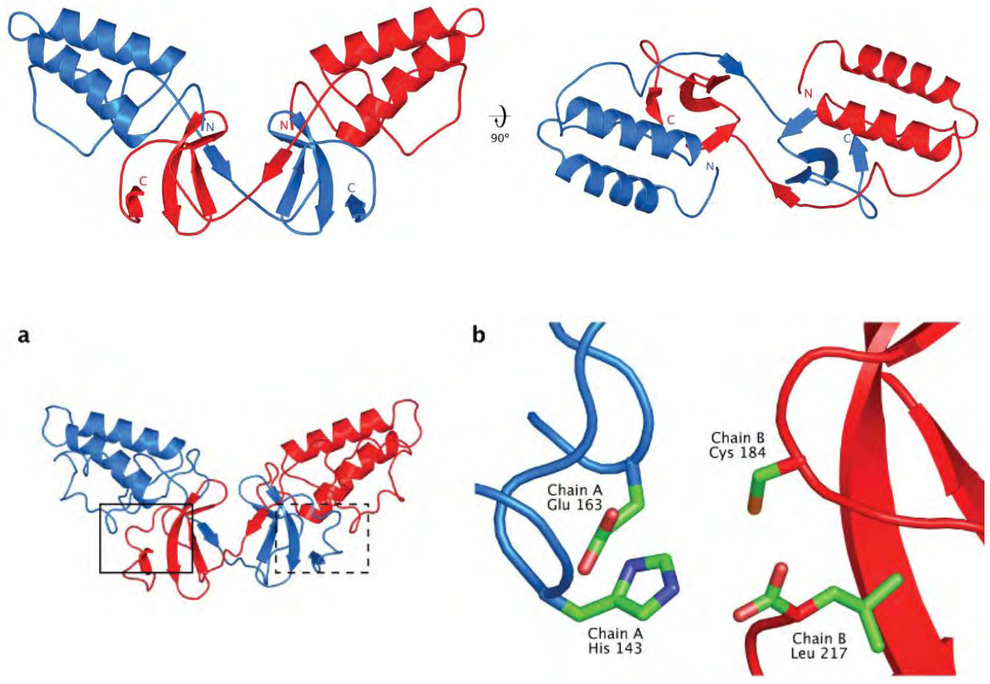
"This was a great challenge, and we've gotten to the top of this mountain," Que said. "Knowing these properties increases our ability to carry out the oxidation of compounds and helps us understand how iron agents might be working in environmental remediation."

That includes an increased knowledge about Collins' Fe-TAML catalysts, which have been demonstrated for a variety of uses including cleaning up water polluted at textile, pulp, and paper mills; removing sulfur from diesel fuel; destroying traces of endocrine-disrupting chemicals in water; killing anthrax spores and other water-borne infectious microbes; and even stopping dark-colored laundry from staining whites and lights in the wash.

This research was supported by the National Institutes of Health, the Heinz Endowments, the Environmental Protection Agency, and the Heinz Family Foundation for the Teresa Heinz Scholars for Environmental Research Award. Other researchers include Filipe Tiago de Oliveira, Arani Chanda, Deboshri Banerjee, Sujit Mondal, and Emile Bominaar, all from Carnegie Mellon University; and Xiaopeng Shan, from the University of Minnesota.

F. de Oliveira, A. Chanda, D. Banerjee, X. Shan, S. Mondal, L. Que, Jr., E. Bominaar, E. Munck, T. Collins, "Chemical and Spectroscopic Evidence for an Fev-Oxo Complex," *Science*, **315**: 835 (2007).

From left, Joe Marcotrigiano, Charles Rice, lab mascot Sadie, Thomas Dentzer, and Ivo Lorenz.



(below) Structure of the NS2 protease domain. a, Ribbon diagram showing the structure of the NS2pro dimer, with one monomer in blue, the other in red. b, Ribbon diagram of the NS2pro dimer rotated 90° around the horizontal axis in a. The amino- and carboxy-termini are labeled.

(above) The active site of NS2. a, Location of the two active sites in the NS2pro dimer, shown within the boxed regions. b, Close-up view of the NS2pro active site in the solid-lined box in a. The amino acid residues His 143, Glu 163, Cys 184, and Leu 217 are shown as ball-and-stick drawings. The active site is composed of His 143 and Glu 163 from one molecule of the dimer (chain A, drawn in blue), and Cys 184 from the other molecule (chain B, drawn in red). The C-terminal residue, Leu 217, originates from the same chain as Cys 184.

Crystal Structure of the Catalytic Domain of the Hepatitis C Virus NS2-3 Protease

An estimated 120 million people are infected worldwide with Hepatitis C virus (HCV), an important human pathogen leading to cirrhosis and liver cancer. A vaccine has not yet been developed, and only 55-60 percent of the patients respond to the currently available therapies. Therefore, approaches to identify novel drug targets are sorely needed. At the NSLS, a team of researchers from The Rockefeller University made a step in that direction by determining the crystal structure of the catalytic domain of the HCV NS2-3 protease, which might aid in the discovery of new antiviral therapies.

The RNA genome of HCV contains one open reading frame that codes for 10 viral proteins. Three proteins are structural components of the virus particle and the remainder is involved in viral replication and host interactions. Synthesis of the HCV proteins occurs by translation of the viral genome into a polyprotein precursor, which is processed into individual proteins by two host cellular and two viral proteases. One of them, the HCV NS2-3 protease, cleaves the viral polyprotein at one position – an essential action for the onset of RNA replication.

Using multiwavelength anomalous diffraction at NSLS beamlines X9A and X29, the research team determined the crystal structure of the catalytic domain of the NS2-3 protease. They found that the protease is a dimer that contains a pair of composite active sites, with both monomers contributing residues to each active site. This unusual feature may enable the virus to regulate the processing of proteins by proteases (an action known as proteolysis) and thus replication initiation.

To demonstrate that NS2 can form dimers with composite active sites in vivo, or within an organism, the team used two mutant NS2-3 polypeptides in tissue culture experiments. Each of these NS2-3 variants expressed individually cannot undergo cleavage at the NS2/3 junction. However, the researchers showed that if a composite active site can form, then mixing of the two NS2-3 mutants leads to the reconstitution of a functional active site, resulting in partial proteolytic processing.

Their results were published in the August 16, 2006 edition of the journal *Nature*.

Proteolysis through formation of a composite active site has never been observed in a serine or cysteine protease (such as HCV NS2-3) before. However, these features are reminiscent of human immunodeficiency virus (HIV), which encodes a dimeric aspartic protease with a single active site at the dimer interface. Thus, HCV and HIV may have evolved similar strategies to control the timing of events during their viral life cycles.

Moreover, knowing details about the protease's structure might help in the search for small-molecule inhibitors directed against the active site.

Researchers involved included Ivo Lorenz, Joe Marcotrigiano, Thomas Dentzer, and Charles Rice, all from The Rockefeller University, Laboratory of Virology and Infectious Disease. This work was funded by the National Institutes of Health, the Greenberg Medical Research Institute, the Swiss National Science Foundation, the Roche Research Foundation, the Swiss Foundation for Medical-Biological Stipends, and the Life Sciences Research Foundation.

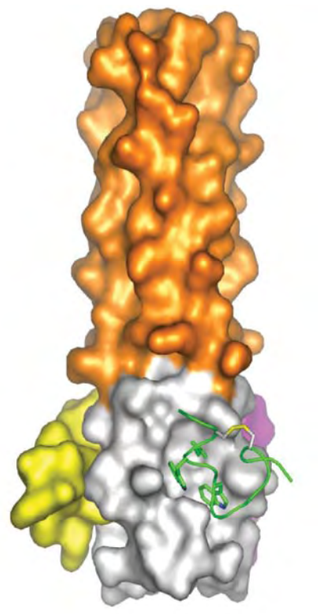
I. Lorenz, J. Marcotrigiano, T. Dentzer, C. Rice, "Structure of the Catalytic Domain of the Hepatitis C Virus NS2-3 Protease," *Nature,* **442**: 831-5 (2006).

Michael Kay (left) and Brett Welch

Chris Hill

Andrew VanDemark

Annie Heroux



Structure of D-peptide inhibitors (green, yellow, and purple) bound to an HIV protein mimic in three "pockets" that are essential to the virus' ability to enter cells. Blocking the pockets thwarts entry and reduces infectivity.

Potent Peptides Inhibit HIV Entry Into Cells

Based in part on protein structures determined at the NSLS, scientists at the University of Utah have developed new peptides that appear to be significantly more effective at blocking HIV's entry into cells than other drugs in their class. In a paper published in the October 23, 2007 edition of the *Proceedings of the National Academy of Sciences*, the researchers say these peptides are sufficiently potent to begin pre-clinical studies as a new class of agents for the prevention and treatment of HIV/AIDS.

"Our 'D-peptides' offer several potential therapeutic advantages over existing peptide entry inhibitors, which are costly, require high dose injections, and suffer from the emergence of drug-resistance," said University of Utah biochemist Michael S. Kay, senior author on the paper. "In contrast, our D-peptides resist degradation, so they have the potential to be administered by mouth and last longer in the bloodstream. Since these inhibitors have a unique inhibitory mechanism, they should work well in combination with existing HIV inhibitors."

The researchers were particularly interested in developing drugs to bind to an essential "pocket" structure found in all HIV strains that was previously identified as a promising drug target using structures determined at the NSLS. Numerous previous attempts to target this pocket failed to produce potent and non-toxic pocket-specific entry inhibitors. In the current work, the researchers used a high-throughput technique to screen a "library" containing hundreds of millions of peptides to identify the rare peptides that would bind to the pocket structure and inhibit HIV entry.

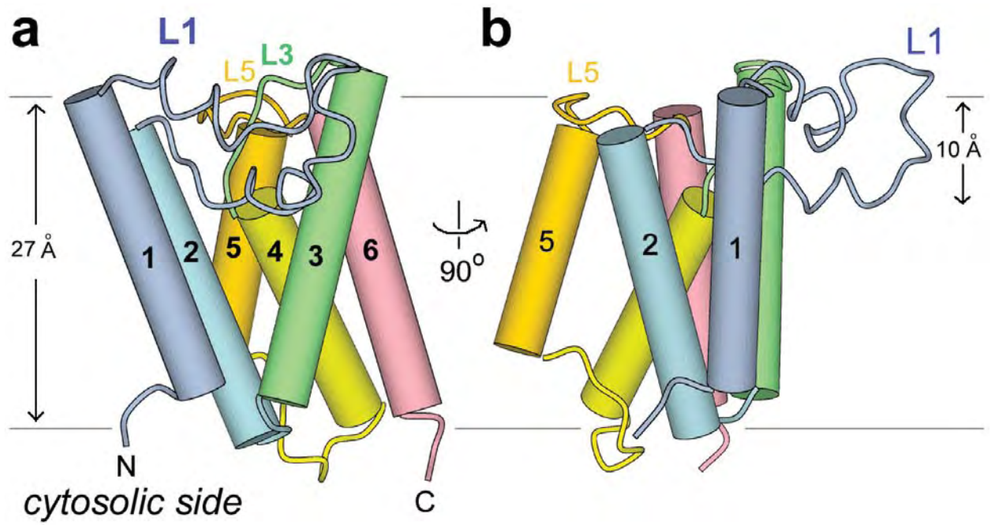
After identifying the most promising candidate peptides, the researchers analyzed the structure of these peptides bound to the target protein using x-ray crystallography at NSLS beamline X29. In this technique, researchers analyze how an extremely bright beam of x-rays bounces off and is refracted by the sample to determine the positions of individual atoms.

"These structures reveal details of how the peptides bind and guide the development of future inhibitors," said paper co-author Annie Heroux, a biologist and crystallography specialist at Brookhaven.

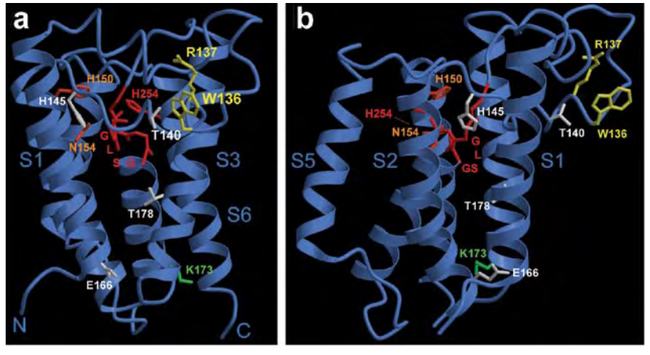
This structure-assisted design led to the discovery of D-peptides with up to a 40,000-fold improved antiviral potency over previously reported D-peptides. The structures also suggest ways to engineer the peptides to reduce the chance of drug resistance.

This research was funded by the National Institutes of Health, the University of Utah Technology Commercialization Project, and the American Cancer Society. Other researchers involved in the study include Brett Welch, Andrew VanDemark, and Christopher Hill, all from the University of Utah.

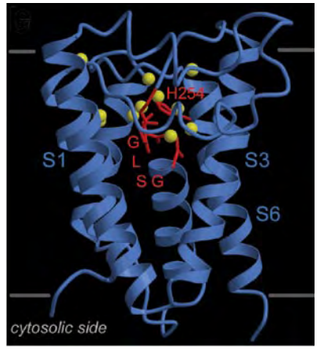
B. Welch, A. VanDemark, A. Heroux, C. Hill, M. Kay, "Potent D-peptide Inhibitors of HIV-1 Entry," *Proc. Natl. Acad. Sci.*, **104**: 16828-16833 (2007).



(above) The overall structure. (a) The front view. The transmembrane helices shown as cylinders are sequentially labeled 1-6. The two horizontal lines mark the boundaries of the membrane. (b) The side view related to (a) by a 90-degree rotation as shown.



Mutagenesis studies on related rhomboid proteases mapped onto GlpG structure. The GLSG (Gly-199, Ser-201) sequence motif and His-254 are shown in red; His-150 and Asn-154 in orange; Trp-136 and Arg-137 in yellow. (a) and (b) represent different views.



The active site is filled with water (yellow spheres).

Crystal Structure of Intramembrane Protease GlpG

Intramembrane proteolysis – a process in which proteases embedded in cellular membranes use water to break apart peptide bonds – was initially a controversial concept because it is not clear where the water comes from in the oily, water-excluding membrane. However, intramembrane proteolysis is now seen as common process in biology, and is responsible for generating amyloid β-peptide that causes Alzheimer's disease. In order to investigate this relationship, a group of Yale School of Medicine researchers used the NSLS to determine the crystal structure of GlpG, one membrane protein that specializes in catalyzing this reaction. Their research illustrates the physical principles that underlie the unique mechanism, and sheds light on the ways proteases regulate crucial biological processes and contribute to disease.

Using x-ray diffraction techniques at NSLS beamlines X6A and X29, the researchers described the crystallized rhomboid structure, GlpG, which provides the first detailed view of the intramembrane protease and correlates with earlier genetic and biochemical studies. Although little is known about their function, rhomboids are found in almost all organisms and they have been paired to processes as varying as mitochondrial function, bacterial communication, and growth factor signaling.

The researchers determined that the structure of GlpG contains six transmembrane helices. Inside the bundle of transmembrane helices is a cavity that opens toward the outside of the cell and is thought to represent the active site of the protease. Their studies also revealed the presence of water molecules in the cavity. Conformational changes cause the cavity to "open," allowing water to diffuse easily into the active site and react with substrate.

Because intramembrane proteolysis usually takes place in aqueous solutions, the researchers' observations solve the long-time puzzle of a water-requiring reaction in the middle of cell membranes. Their results were published in the November 9, 2006 edition of the journal *Nature*.

Researchers included Ya Ha, Yongcheng Wang, and Yingjiu Zhang, all from Yale. Funding was provided by the U.S. Department of Energy, the National Institutes of Health, the Ellison Medical Foundation, and the Neuroscience Education and Research Foundation.

Y. Wang, Y. Zhang, Y. Ha, "Crystal Structure of a Rhomboid Family Intramembrane Protease," *Nature*, **444**: 179-183 (2006).

Accretion of Bone Quantity and Quality in the Developing Mouse Skeleton

An important contributor to bone quality is its chemical makeup. It is well established that the chemical composition and mechanical properties of bone change with age. However, the interdependence between bone's specific chemical makeup and its corresponding mechanical properties is still poorly understood. At NSLS beamline U10B, a team of researchers from Brookhaven and Stony Brook University evaluated the chemical, structural, and mechanical properties of the mouse tibia during the first 40 days of life to find out whether specific compositional properties of bone determine the stiffness of the tissue.

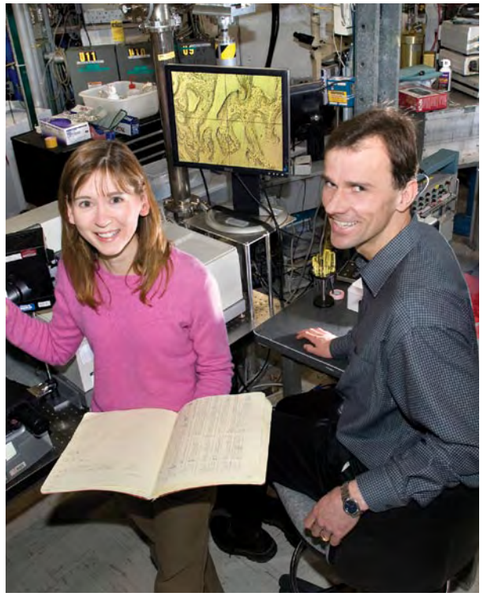
Using synchrotron IR microspectroscopy, microCT, and nanoindentation, the researchers found that bone mineral formation proceeded very rapidly in mice by one day of age, where the degree of mineralization, the tissue mineral density, and the mineral crystallinity reached 36 percent, 51 percent, and 87 percent of the adult values, respectively. However, even though significant mineralization had occurred, the elastic modulus of one-day-old bone was only 14 percent of its adult value and increased to 89 percent after 40 days, indicating that the intrinsic stiffening of the bone lags considerably behind the initial mineral formation.

The researchers believe that this initial lag in elastic modulus may be associated with the specific locations of the mineral crystals within the collagen fibrils. Electron micrographs have shown that, in early mineralization, the overall accumulation of mineral mass is predominant in what are known as collagen "hole zones" compared to "overlap zones." Since the elastic modulus of the mouse bone remained low until the tissue mineral density reached about 65 percent of its adult value, it is possible that mineral accumulation in the hole zones has little effect on bone's intrinsic stiffness. But once the mineral content increased to where the overlap zones become mineralized, the elastic modulus increased rapidly as well.

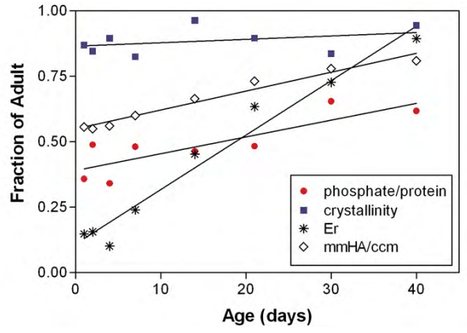
The team's results were published in the July 2007 edition of the *Journal of Bone and Mineral Research.* They indicate that specific chemical and structural properties modulate bone's stiffness during early growth and suggest that changes in bone may be co-regulated by similar genes during this development period. The intrinsic stiffening of the bone, however, falls behind the initial mineral formation, emphasizing the importance of bone mineral quality for optimizing strength. With clear evidence that bone's chemical properties and micro-structure play an important role in defining the micro-mechanical properties of the skeleton during growth, a better mechanistic understanding of the underlying processes may enable the diagnosis, prevention, and treatment of poor bone quality.

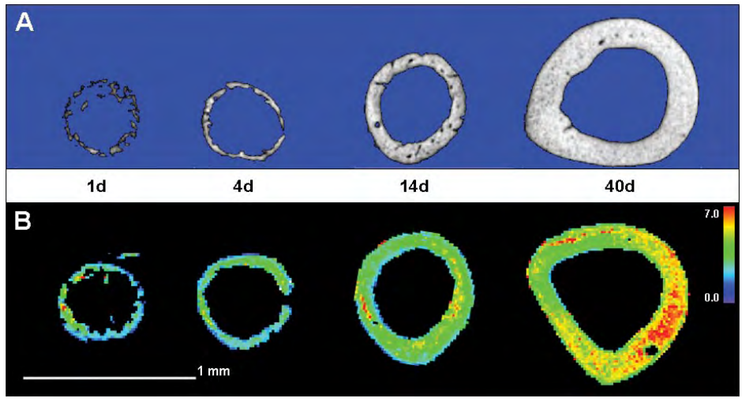
Researchers include Lisa Miller (NSLS), and Anne Schirmer, William Little, Farhan Sheik, Bhavin Busa, and Stefan Judex (Stony Brook University). Funding for the study was provided by the U.S. Department of Energy, NASA, the Whitaker Foundation, and a SUNY-BNL Seed Grant.

L. Miller, W. Little, A. Schirmer, F. Sheik, B. Busa, S. Judex, "Accretion of Bone Quantity and Quality in the Developing Mouse Skeleton," *J. Bone Miner. Res.,* **22**: 1037-1045 (2007).

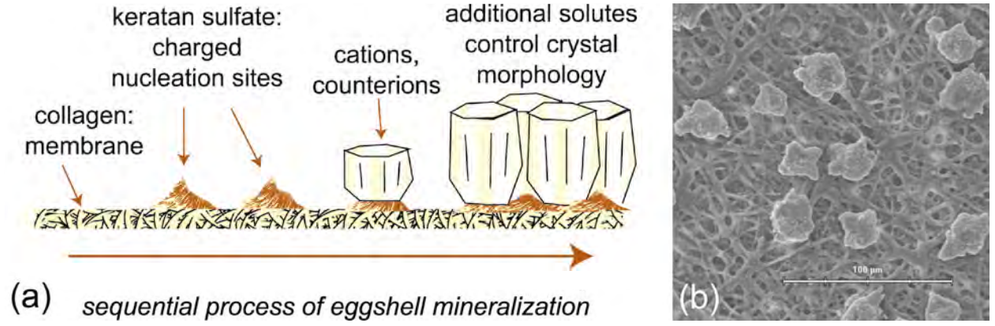


Phosphate/protein, crystallinity, elastic modulus, and TMD as a function of age. In order to compare all parameters on a common scale, the y-axis values represent the fraction of the adult (15mos) value. Results showed that bone mineralization (phosphate/protein ratio, crystallinity, and TMD) proceeded quickly at an early age, whereas bone stiffness (elastic modulus) lagged behind. This initial lag in elastic modulus may be associated with the specific locations of the mineral crystals within the collagen fibrils.

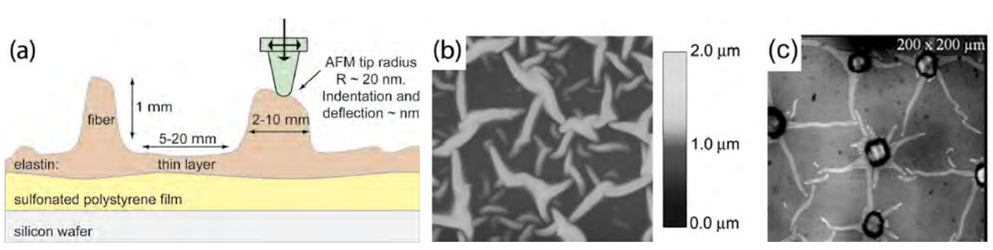




In this study, tibiae of female BALB mice were harvested at 8 time points (n = 4 each) distributed between 1d and 40d of age. Tibiae of 450d old mice served as fully mineralized control specimens. (A) Micro-CT and (B) FTIRM phosphate/protein images of the mouse mid-diaphysis at 1d, 4d, 14d, and 40d of age. All images for each technique are plotted on the same intensity scale for direct comparison. Results showed that the spatial variability in mineralization across the mid-tibia was very high for the early time points and declined over time.



(a) The sequential mineralization stages of an eggshell. Upon the rough collagen membrane, charged sites of nucleating protein are deposited first, then exposed to calcium and carbonate to nucleate crystals. Subsequent chemical species in the fluid control the crystal growth and orientation. (b) Scanning electron micrograph of an eggshell membrane at the early stage, showing the fibers of collagen and the first nucleated crystals. (Courtesy of J. L. Arias, Universidad de Chile, Santiago Chile). Scale bar: 100 microns.



(a) Schematic of the model system showing how thin layer protein and micron fibers are layered upon the silicon wafer/sulfonated polystyrene substrate. (b) AFM image of the network of elastin fibers. Panel 50 microns wide. (c) The eggshell-mimicking result of the experiments shown in an optical micrograph of the elastin network with calcite crystals located at the fiber vertices. Panel 200 microns wide.

Hatching a New Model for Biomineralization

The idea started with an eggshell and ended with a new understanding of how minerals form to build exceptionally strong structures in the bodies of humans and other organisms. Biomineralization, the process by which organisms form materials such as bones, mollusk shells, and other structures, has captured the attention of scientists for years. Finding a way to mimic the properties of these sturdy and naturally made materials could lead to the medical engineering of replacement bone, teeth, and cartilage, as well as the development of new electronic and industrial materials. Most of the research surrounding biomineralization has looked at the multiple processes it involves and the diversity of its products. But at NSLS beamline X6B, using inspiration from an egg, a team of researchers studied the earliest stages of biomineralization to find out what sets the process in motion.

A bird's eggshell is about a half-millimeter of layered calcium carbonate crystals, stabilized by a protein matrix. The shell forms during just about 12 hours of travel time through the bird's oviduct, an amazing natural feat, said NSLS physicist Elaine DiMasi, one of the authors of the biomineralization study that was published in the October 3, 2006 edition of the *Proceedings of the National Academy of Sciences.*

"It starts as a collagen membrane and goes through a series of different fluids with different species in them, and in the end, you have this hard mineral," she said. "We were looking for a system that would mimic some features of that eggshell."

To model extracellular biomineralization, the formation of materials on the outside of the cell wall, such as in the case of egg shell formation, the research team used a self-assembled protein network with both fibronetin and elastin – major connective tissue components in multicellular organisms. These proteins were incubated on negatively charged surfaces in two forms: structurally organized fibers and regions with a thin unorganized layer of protein wedged in between them.

After exposing the system to calcium carbonate for a varying set of times, the researchers used a relatively new technique called shear modulation force microscopy (SMFM) to compare the response of the two sets of protein fibers. SMFM is an atomic force microscopy-based technique in which a cantilever with a superfine tip just 40 nanometers wide is stuck into the soft material being studied. The tip is then vibrated to measure the stiffness of the material, and thus whether or not mineralization occurred on the protein fibers.

The group found that the calcium carbonate stiffened only the organized protein fibers, without affecting the unorganized regions between them. This demonstrates that mineralization requires structural organization of the protein in order to function, DiMasi said.

"It's exciting that there's a demonstration of disorganized and organized proteins side by side in the same exact environment," DiMasi said. "Any other experiment would have just inferred that an organized protein structure was necessary to nucleate, but there's never been a comparison like this between disorganized and organized protein."

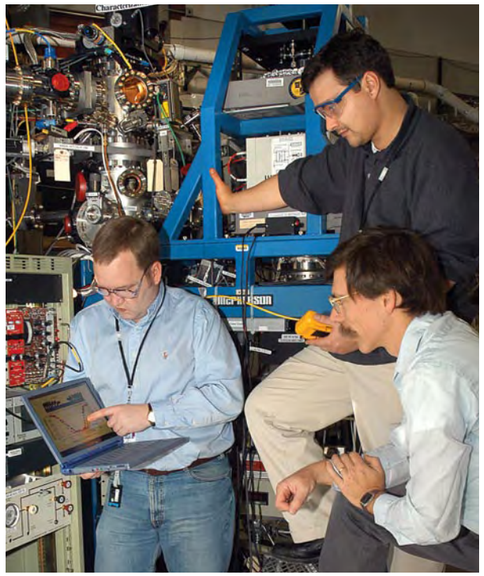
Besides the actual results, the setup of the experiment itself, including the model system and the SMFM technique, provides valuable information for the scientific community – and not just to study eggs.

"This looks like a really good model system," DiMasi said. "Now one could take collagen and calcium phosphate and study bone nucleation or any other number of things. It just looks like a really good platform."

Other scientists involved in the study are Seo-Young Kwak (NSLS); Karthikeyan Subburaman, Nadine Pernodet, Shouren Ge, Vladimir Zaitsev, Xiaolan Ba, and Miriam Rafailovich (Stony Brook University); and Nan-loh Yang (City University of New York).

This research was funded by the National Science Foundation Materials Research and Engineering Centers and the Brookhaven National Laboratory-Stony Brook University Seed Grant Program.

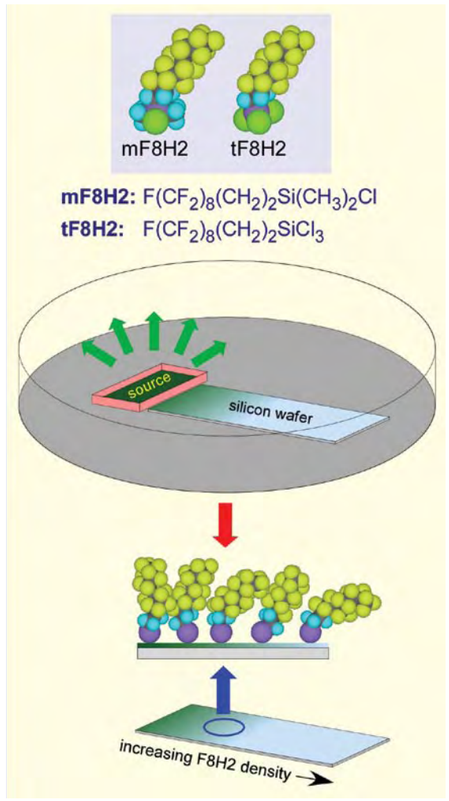
K. Subburaman, N. Pernodet, S. Kwak, E. DiMasi, S. Ge, V. Zaitsev, X. Ba, N. Yang, M. Rafailovich, "Templated Biomineralization on Self-Assembled Protein Fibers," *Proc. Natl. Acad. Sci.*, **103**: 14672-14677 (2006).



Jan Genzer (left), Kirill Efimenko (top), and Dan Fischer

Jack Douglas

Frederick Phelan



Schematic illustration of the formation of self-assembled monolayer (SAM) surface energy gradients. Semifluorinated chlorosilane molecules are mixed with paraffin oil to control the rate of organosilane evaporation and this mixture is placed in a small container positioned at the edge of a silicon wafer subjected to a UV-ozone treatment. The evaporated chlorosilane molecules deposit on the wafer where the ordering process initiates from the edge of the wafer where the concentration is higher. The deposition is performed in a "confined" geometry (covering the OS source and wafer by a Petri dish).

Wave-like Growth of Self-Assembling SAM Layers

By studying the problem of how a layer of molecules self-assembles into surface energy gradients on a silicon wafer, scientists at North Carolina State University working with colleagues from the National Institute of Standards and Technology have gained basic insight through high-resolution x-ray measurements taken at the NSLS. Wavefronts associated with reaction–diffusion and self-assembly processes are ubiquitous in the natural world. Although it is often claimed that self-sustaining or autocatalytic front propagation is well described by models, it has become apparent that fluctuation effects in lower spatial dimensions can lead to appreciable deviations from the classical mean-field theory (MFT) of this type of front propagation. The researchers' work explores these fluctuation effects in a real physical system.

At NSLS beamline U7A, the researchers used high-resolution near-edge x-ray absorption fine structure spectroscopy (NEXAFS) to study the spontaneous frontal self-assembly of organosilane molecules into self-assembled monolayer (SAM) surface-energy gradients on oxidized silicon wafers. They found that upon exposure of the wafer to a volatile source of the organosilane molecules placed to the side of the wafer in a closed container, a carpet-like layer spontaneously organizes from the edge of the wafer, where the organosilane concentration is initially higher. This self-assembly process advances from the wafer edge at a constant velocity, ultimately covering the surface at long times.

By using x-ray measurements in combination with a computer simulation study, the researchers found that simple diffusion is not the process by which these SAM surface energy gradients fronts organize, as people had formally assumed. Instead, the interface region separating the ordered and disordered regions became progressively rougher in time. The position of the wavefront is described by a relatively narrow interfacial region separating the ordered and disordered regions on the substrate that can be determined and tracked by NEXAFS measurements, which provide unique information about the density and ordering of the molecules on the surface.

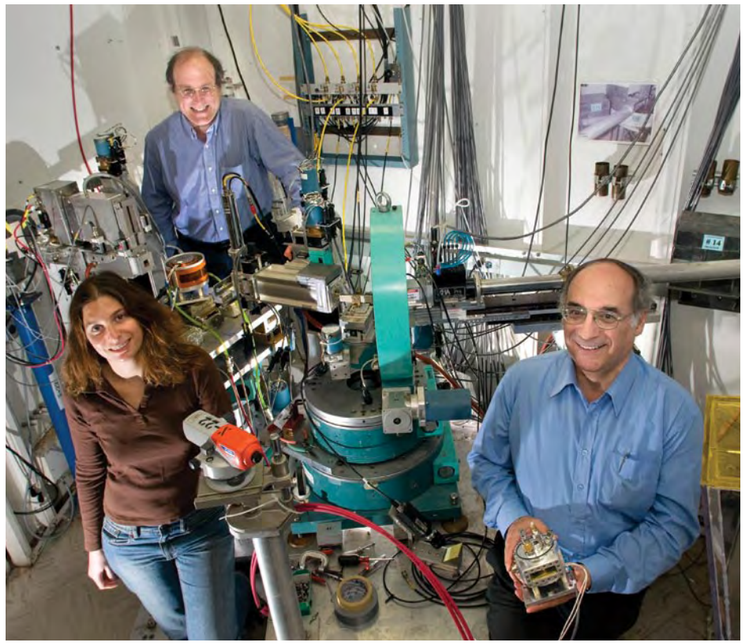
Their results appear in the June 19, 2007 edition of the *Proceedings of the National Academy of Sciences.*

By studying this type of process in detail, as exemplified by the NEXAFS study of growth of SAM surface energy gradients, and by simulation studies, the group hopes to obtain insights into this fundamental growth phenomenon. This project offers the opportunity to compare the growth dynamics of the SAM layers with growth fronts found in numerous other (physical, biological and social dynamics) contexts such as crystallization, the polymerization of actin and the frontal polymerization of synthetic polymers, the spread of diseases, tumor growth, wound healing, and the spread of languages and agricultural practices.

Researchers included Jack Douglas, Frederick Phelan, and Daniel Fischer (National Institute of Standards and Technology), and Kirill Efimenko and Jan Genzer (North Carolina State University). Funding was provided by the National Science Foundation.

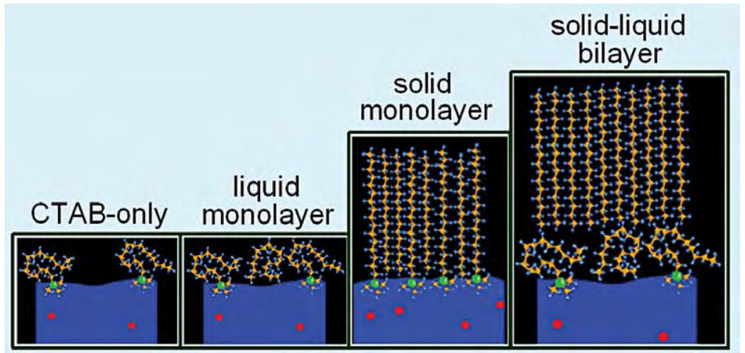
J. Douglas, K. Efimenko, D. Fischer, F. Phelan, J. Genzer, "Propagating Waves of Self-assembly in Organosilane Monolayers," *Proc. Natl. Acad. Sci.,* **104**: 10324-10329 (2007).

From left, Lilach Tamam, Ben Ocko, and Moshe Deutsch



Eli Sloutskin

Colin Bain



Molecular interpretations of the different phases of the alkane film on the CTAB-decorated water surface.

Spreading and Surface Freezing of Nanometer-Thick Oil Films on Water

Most kindergarteners can tell you that no matter how hard you try to mix them, a droplet of oil won't spread on water. However, things are different in the nano world. Recently, a team of researchers from Durham and Oxford universities in the United Kingdom, Bar-Ilan University in Israel, and BNL used x-ray experiments at the NSLS to show that a nanometer-thick layer of oil can be induced to spread on the surface of water by a minute amount of an additive called surfactant. More intriguing, these layers exhibit a very peculiar behavior known as the surface freezing effect.

"Surface freezing is unusual because in most situations, the bulk of a material freezes before the surface," said Bar-Ilan physicist Moshe Deutsch. "You can think of it as a crowd of people standing at a concert. The people in the center are more limited in movement than the people positioned on the outside, and therefore, they'll order first. Yet, if you replace the people with molecules, the surface freezing we've observed in liquids is exactly the opposite: the molecules at the boundary order (or freeze) first while the bulk remains liquid."

Among other applications, understanding surface freezing could be valuable to improving the function of diesel engines, which at cold climates, might be effected by the way oil is injected into the motor.

"It's such an unusual effect, and we still don't fully understand how it happens," said researcher and Durham University chemist Colin Bain.

To gain a better understanding of this phenomenon, the researchers put a drop of a liquid oil called alkane onto the surface of water containing a minute concentration of the surfactant cetyltrimethylammonium bromide (CTAB). This is not so different than making salad dressing by adding oil to vinegar – two liquids that don't naturally mix.

"If you take an oily plate and fill it with water, you'll notice these little droplets of oil on the surface," said Harvard physicist Eli Sloutskin, who took part in the study as a graduate student at Bar-Ilan. "The oil droplets won't spread. But the moment you drop a little bit of soap, which is a surfactant, on the plate, the oil droplets vanish: they spread out on the water's surface and also mix with the bulk."

On a much smaller scale than the kitchen sink, the research group determined that while an alkane won't naturally spread on water, a small amount of CTAB causes it to form a single liquid alkane layer on the surface. After the material is cooled past a certain point, the liquid layer turns crystalline. The type of crystal formed depends on whether the alkane chain is longer or shorter than the CTAB molecule's hydrocarbon tail. Using surface x-ray scattering techniques at NSLS beamline X22B, the researchers determined that if the alkane chain is shorter than the surfactant's tail, the liquid surface monolayer becomes a solid monolayer. If the alkane chain is longer, two layers form: an upper crystalline layer on top of a lower liquid one. Until now, this type of behavior has not been observed in any system.

Their results were published in the September 28, 2007 edition of *Physical Review Letters.*

"In forming these phases, spreading the oil on water and causing it to freeze as a monolayer or a double layer, we are doing nothing but self-assembly: we 'encourage' the molecules to self-organize by creating an environment that favors a particular phase," said Brookhaven physicist Ben Ocko. "We can fine tune the structure through the type of oil and surfactant used, and of course, the temperature. This is an exciting new system that certainly holds more secrets to be discovered and surprises to stumble upon. We are working hard to uncover the former, and keeping our eyes open for the latter."

Other researchers involved included Zvi Sapir and Lilach Tamam (Bar Ilan), and Qunfang Lei and Katharine Wilkinson (Oxford). Funding was provided by the U.S. Department of Energy and the U.S.-Israel Binational Science Foundation.

E. Sloutskin, Z. Sapir, C. Bain, Q. Lei, K. Wilkinson, L. Tamam, M. Deutsch, B. Ocko, "Wetting, Mixing and Phase Transitions in Langmuir-Gibbs Films," *Phys. Rev. Lett.,* **99**: 136102 (2007).

NSLS and NSLS-II staff gather at the Light Sources Directorate Annual Awards Barbeque.

Richard Biscardi, Zhong Zhong Recognized at BNL Award Ceremony

Two NSLS employees – Richard Biscardi and Zhong Zhong – are honored at the Fiscal Year 2007 BNL Employee Recognition Award Ceremony. Biscardi, chief electrical engineer for the NSLS, received one of five Engineering Awards, while Zhong, an NSLS physicist, received one of five Science & Technology Awards.

NSLS Users Receive Prestigious Honors

Numerous users are honored for scientific achievement, including: Noel Clark and Chris Miller, who were among 72 new members elected to the National Academy of Sciences; Cold Spring Harbor researcher Leemor Joshua-Tor, who won the 2007 Dorothy Crowfoot Hodgkin Award, given at the 7th European Symposium of The Protein Society, for her outstanding achievements in protein structure-function relationships; and Yale biophysicist Thomas Steitz, who received one of the five 2007 Gairdner International Awards along with Harry Noller, of the University of California, Santa Cruz, for work that led to the identification of the detailed structure and function of the ribosome.

"This is the best learning experience I've had from start to finish."

- Kelly Jamieson, New York University graduate student and X6A Workbench participant

In Memoriam: Jamshed "Jim" Patel

Advanced Light Source user Jamshed (Jim) Patel, who played a large role in the history of the NSLS, dies at the age of 81. Patel was an initial investigator for one of the NSLS Bell Labs beamlines that were conceived and constructed during the 1980s – X15A.

APS Meeting: Unlocking the Secrets of High-temperature Superconductors

Three Brookhaven physicists, including NSLS user Christopher Homes, discuss their most recent findings about the copper oxide superconductor LBCO at the March meeting of the American Physical Society in Denver, Colorado. Homes discussed what is known as the LBCO 1/8 doping, a state at which the material's superconductivity mysteriously disappears.

NSLS Users Present Energy Research at ACS Meeting

Two NSLS users – Brookhaven chemists Radoslav Adzic and Jose Rodriguez – present their research on fuel cells and energy production at the 233rd National Meeting of the American Chemical Society. Adzic described his research on stabilizing platinum electrocatalysts for use in fuel cells while Rodriguez described how copper can be substituted for gold in reactions that keep fuel cells functioning longer while eliminating unwanted byproducts.

Webconference Brings the Beamline to the Classroom

About 65 students from the Paul J. Gelinas Junior High School and Sayville High School participate in an environmental experiment via webconference at NSLS microprobe beamline X27A. The project analyzed soil samples collected near a deck built with chemically treated wood.

X6A Workbench Returns

After many requests from the structural biology community, the X6A workbench series returns with a four-day course hosted by the NIGMS facility at the NSLS. Six young researchers attended the workshop for hands-on training in synchrotron data collection and analysis.

RapiData 2007 Brings Future Crystallographers to NSLS

For the ninth year in a row, about 50 scientists from around the world gather at the NSLS for RapiData 2007, a crash course designed to introduce participants to the best and latest equipment and techniques for macromolecular x-ray crystallography.

NSLS Sons, Daughters Uncover Mysteries of Diffraction

As part of the national "Take Our Daughters and Sons to Work Day," about 30 children of NSLS users and staff visit the Lab to learn about light sources and the importance of diffraction studies to the science performed on the experimental floor.

COMPRES Beamlines Receive 5-Year Funding Renewal →

Funding for the Consortium for Materials Properties Research in Earth Sciences is renewed through the National Science Foundation with a five-year, $11.5 million cooperative agreement that includes more than $4 million for the operation of NSLS beamlines X17B2, X17B3, X17C, and U2A.

2007 NSLS-CFN Joint Users' Meeting

The second joint meeting of the NSLS and Center for Functional Nanomaterials user communities stressed the importance of NSLS-II to the Lab's research synergy. Plenary session speakers included BNL Director Sam Aronson, U.S. Department of Energy's Director of Basic Energy Sciences Pat Dehmer, Associate Laboratory Director for Light Sources Steve Dierker, CFN Director Emilio Mendez, and NSLS Chair Chi-Chang Kao.

← Heese Receives 2007 UEC Community Service Award

Richard Heese, a senior scientist at BNL in the Operations Division of the NSLS, receives the 2007 NSLS Users' Executive Committee Community Service Award for service, innovation, and dedication to NSLS users.

Inaugural Julian Baumert Ph.D. Thesis Award Goes to Benjamin Hornberger

NSLS user and Stony Brook University graduate, Benjamin Hornberger, receives the first Julian Baumert Ph.D. Thesis Award, which was established in memory of Julian David Baumert, a young Brookhaven physicist and NSLS user who died in June 2006.

Crystallization Workshop: Five Years and Growing ↓

More than 40 researchers travel across the country and the world to learn tricks of the trade from protein crystallography leaders at the "Crystallization: Focus on Membrane Proteins" workshop, the fifth annual course of its type.

BioCD-2007: Circular Dichroism Spectroscopy of Proteins and Nucleic Acids

A weeklong workshop on ultraviolet (UV) circular dichroism (CD) brings 14 students to the NSLS. UV CD is particularly useful for studying the structure of proteins, DNA, RNA and other biopolymers in solution, and this was a major focus of the workshop.

NSLS Hydrogen Research Projects Receive DOE Funding

The U.S. Department of Energy's Office of Basic Energy Sciences awards $11.2 million for 13 hydrogen research proposals, including two projects to take place, in part, at the NSLS. One study seeks to better understand and improve the performance of nanocatalysts for a process known as the "water-gas shift reaction" and the other tackles the hydrogen storage issue through investigating ammonia borane.

NIST Receives $5 Million Funding Increase for Synchrotron Research

As part of the American Competitiveness Initiative, the National Institute of Standards and Technology (NIST) announces that it will expand upon its synchrotron research capabilities, primarily at the NSLS, with a $5 million funding increase.

← Over 700 Visitors Drawn to NSLS Summer Sunday for Demos, Tours

More than 700 community members flock to the NSLS Summer Sunday to learn about everything from spectroscopy to space dust. Visitors participated in 14 hands-on displays, listened to introductory science lectures, and had the opportunity to win a tour of the experimental floor.

CD-1 Status Awarded to NSLS-II Project at First Users Workshop

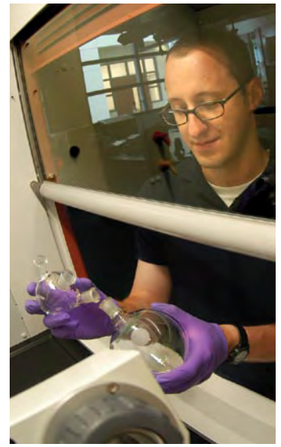
More than 450 participants in the Users Workshop for the National Synchrotron Light Source II (NSLS-II) are among the first to hear that the project had been awarded "Critical Decision One." Pat Dehmer, Director of the Department of Energy's Office of Basic Energy Sciences, made the dramatic announcement during the plenary session of the workshop, which included project updates, talks, and technique- and science-based breakout sessions.

NSLS Hosts Historically Black Colleges and Universities Professors' Workshop

A Historically Black Colleges and Universities (HBCU) Professors' Workshop is held at the NSLS, linking 11 professors from nine institutions with the tools to become successful synchrotron users. The workshop resulted in the formation of an HBCU User Consortium that will encourage further involvement at the NSLS and Brookhaven.

ACS Meeting: Using Life's Building Blocks to Control Nanoparticle Assembly →

NSLS user Mathew Maye, a chemist in Brookhaven's Center for Functional Nanomaterials, presents the latest findings in DNA self-assembly at the 234th National Meeting of the American Chemical Society. Using DNA, Maye and fellow researchers are studying how to control the speed of nanoparticle assembly and the structure of its resulting nanoclusters.



NSLS Hosts 17 Summer Students ↑

Seventeen high school and college students performed summer research projects at the NSLS, working with scientists and engineers from the department in research fields ranging from medical sciences to electrical and mechanical engineering. Students also had the opportunity to attend scientific lectures, tour Brookhaven's research facilities, and participate in social activities.

"There are opportunities that come to only a very few people in the right place at the right time. The place is here, the time is now."

- Pat Dehmer, DOE's Director of Basic Energy Sciences and NSLS-II Users' Meeting Workshop speaker

NSF Awards $550,000 for NSLS X-Ray Spectroscopy Detector

The National Science Foundation awards $550,000 to develop a new silicon detector for x-ray spectroscopy at the NSLS. Proposed by researchers from the New Jersey Institute of Technology, the NSLS, and the University of Tennessee, the detector system will produce at least a 50-fold increase in signal processing capability while maintaining high-energy resolution.

Staff Reflects on Successful Year at Light Source Directorate Barbeque

With sunny skies and tables full of food, employees gather around BNL's picnic area for the Light Sources Directorate Annual Awards Barbeque. NSLS Chair Chi-Chang Kao and Steve Dierker, Associate Laboratory Director for Light Sources, hosted the end-of-year celebration to honor NSLS and NSLS-II employees for another year of smooth and productive operation.

↑ A Course on Quantitative Hard X-Ray Micro-Fluorescence Imaging

Twenty-one graduate, postdoctoral, and research scientists come to the NSLS for a series of hands-on tutorials and research-based talks outlining the necessary techniques and analysis tools for unraveling and quantifying micro-fluorescence data from synchrotron facilities.

← 428th Brookhaven Lecture: Kenneth Evans-Lutterodt

NSLS physicist Kenneth Evans-Lutterodt gives the 428th Brookhaven Lecture, titled "Lighthouses, Lightsources and Kinoform Hard X-Ray Optics," about an innovative method that uses a type of refractive lens called a kinoform lens to overcome a major x-ray focusing obstacle.

NSLS Receives NIH Grant for a New Infrared Imaging Microscope

The NSLS receives a Shared Instrumentation Grant from National Institutes of Health for $296,000 to purchase a state-of-the-art infrared imaging microscope system – a new endstation that will allow for faster data collection and improved spatial resolution for numerous studies.

Short Course: Applications of XAFS to Nanocatalysis Science

A short course in x-ray absorption fine-structure analysis is offered at the NSLS, tailored to Synchrotron Catalysis Consortium users who have recently collected data in systems of relevance for catalysis applications: nanoparticles, mono- and bimetallic, supported on different surfaces, studied ex situ or in situ.

NSLS Web Conference Connects Cross-Country Schools for Bivalve Study

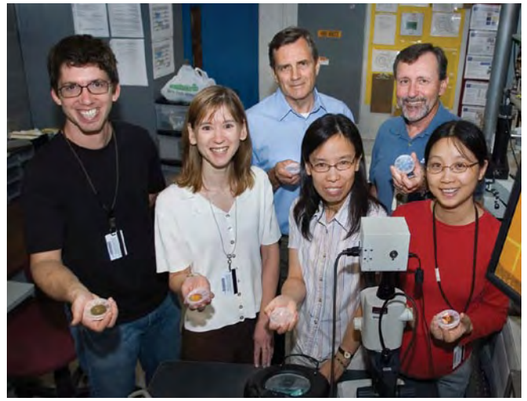
After collecting sediment, mussels, and oysters from their local waterways, about 200 students and teachers from 10 local and cross-country schools participate in a web conference linking scientists at beamlines X27A and X26A with the individual classrooms.

Sep

Oct

Microgrid Allows Simultaneous Study of Multiple Variables →

At the NSLS, Brookhaven scientists develop a method for correlating the results of microscopic imaging techniques in a way that could lead to improved understanding, diagnosis, and possibly treatment of a variety of disease conditions, including Alzheimer's disease. The Laboratory filed a U.S. provisional patent application for the invention.



Physicists Vivian Stojanoff, Timur Shaftan Awarded Tenure ↓

Light Source Directorate researchers Timur Shaftan and Vivian Stojanoff are two of six Brookhaven scientists granted tenure in 2007 by Brookhaven Science Associates. Shaftan was awarded for his research on electron beams in accelerators for synchrotron radiation and free-electron laser sources, and Stojanoff was appointed based on her development of new approaches to characterize the quality of macromolecular crystals and mitigate their radiation damage.

↑ Department of Energy Grants CD-2 Status to NSLS-II Project

The U.S. Department of Energy grants "Critical Decision 2" (CD-2) status to the National Synchrotron Light Source II (NSLS-II). This decision approves the facility's performance baseline based on preliminary design, signifying a major step forward in the process to make the state-of-the-art research complex a reality.

Nov

Dec

"This is a great way for students to experience big science from their classroom."

- Scott Bronson, Brookhaven's Office of Educational Programs administrator and NSLS web conference coordinator

"Everyone is asked to help by continuing to do great science while assuring the needed emphasis on personnel safety and on protection of the environment."

Andrew Ackerman
Environment, Safety, Health, and Quality Division Head

Facility Report

Everyone was busy in 2007 and there was considerable Environment, Safety, Health, and Quality (ESH&Q) activity throughout the department. A summary of some of those activities follows:

- An audit of the department's Integrated Safety Management (ISM) program was completed by the Department of Energy's Environmental, Safety, Security, and Health team. The NSLS scores from that audit were excellent and the department program was recognized as the best at the Laboratory. The audit report also included some helpful findings and corrective actions have begun.

- The NSLS maintained independent registration with the Environmental Management System (ISO 14001) and the Occupational Health and Safety Assessment Series (OHSAS) – an important goal for the Laboratory.

- Nanoscience work was characterized and the risks presented were evaluated, resulting in a set of control requirements for work involving nanoscale materials at the facility. New HEPA-filtered, ventilated enclosures were installed for work with nanoscale particulate and detailed review of this work was added to the experiment review process and documentation.

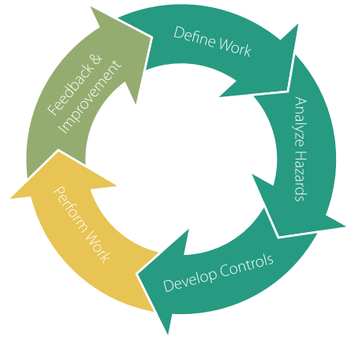
- Inspection of electrical equipment for compliance with Nationally Recognized Testing Laboratory (NRTL) requirements was advanced for facility and user equipment. All "high-hazard" equipment was inspected. Engineered corrections for equipment that did not meet requirements were developed and implemented.

- A gap analysis between department programs and the requirements of 10 CFR Part 851 was completed and a corrective action plan was established and is in progress.

- The NSLS wet chemistry laboratory steward program was evaluated and a detailed set of roles, responsibilities, authorities, and accountabilities (R2A2s) was defined.

Several performance measures are tracked to follow the effectiveness of the ESH&Q programs. In 2007: training compliance was excellent and improved from last year; routine workplace inspections yielded fewer findings per inspection and correction time was short; numerous internal and external audits resulted in few findings and the department response was timely and organized; waste generation continued to be low and well managed; personnel exposure to chemical and physical hazard was well controlled and well below allowable limits; radiation exposure remained very low with a measured collective dose of approximately 50 mRem for the year for some 3,000 personnel monitored; there was one "Days Away, Restricted, Transferred" (DART) case – a minor elbow injury that resulted in lost days and temporary restriction from heavy lifting; and there was only one incident that met the BNL criteria for reporting within the Occurrence Reporting and Processing System (ORPS). It involved a small fire caused by an exhaust fan. There was no injury, no significant equipment damage, and no significant environmental insult, and it yielded some valuable lessons that were disseminated throughout the community.

There is more to report, but the summaries above provide a good overall picture of the department activity and performance in ESH&Q. There is much reason for pride in staff and user attention to keeping the facility a safe place to work. Good performance indicators and well-managed programs are important, but what is most important is that we completed an enormous amount of science last year without significant injury or environmental insult.



NSLS Integrated Safety Management Audit Scores

Safety

"High reliability can only be accomplished through the hard work and dedication of our staff members, who need to be thanked for this outstanding achievement."

James B. Murphy
Deputy Chairman for Accelerators and Operations

Facility Report

This year has been a very busy one for the NSLS staff. A lot of improvements have been made in the injection system, which reduces the amount of time spent injecting beam while maximizing the beam time available to our users. Progress has also been made on the safety front, with the finalizing of personal protective equipment (PPE) requirements and large steps made in the Electrical Equipment Inspection (EEI) program. Finally, the installation of the X9 insertion device and beamline, dedicated to studies in the field of nanotechnology at Brookhaven's Center for Functional Nanomaterials (CFN), takes center stage as our top priority for 2007 and 2008.

For the 2007 calendar year, the Vacuum Ultraviolet (VUV) reliability was 98.5% with 5,682 hours of beam time delivered to its users, while X-Ray reliability came in at 96.1% and 4,961 hours delivered to the users. High reliability can only be accomplished through the hard work and dedication of our staff members, who need to be thanked for this outstanding achievement.

Overall Facility Progress

The Department of Energy (DOE)-mandated EEI program ensures that electrical equipment is free from reasonably foreseeable risks due to electrical hazards through inspections conducted by Laboratory Electrical Safety Committee-approved personnel who work for the NSLS. Priority is given to equipment built in-house or built according to BNL specifications (categorized as high hazard). Twenty-six NSLS staff members are trained EEIs. By the end of September 2007, more than 96% of the listed high-hazard equipment was inspected. This is an ongoing effort and will continue through 2009.

Also regarding safety, an outside expert performed an arc-flash study, and calculations for the PPE required for each panel installation were completed. Since we posted the equipment conservatively prior to having the completed calculations, many of our electrical installations were posted with relaxed PPE requirements. Since this will ease requirements for electricians to reset some breakers and switches, facility and beamline downtime will be reduced to some degree. PPE requirements were also relaxed with the installation of five remote motor control starters on utilities pumps. This effort will minimize arc flash risk and eliminate the need for "bee-keeper" PPE when actuating high-power breakers and switches because these switches may now be actuated at a safe distance.

A project began to build an automated liquid nitrogen (LN_2) fill system, which will more safely deliver LN_2 to beamlines while significantly reducing staff time needed for filling dewars. The automated system will fill dewars with LN_2 and shut off automatically when full. The necessary hardware needed to upgrade the existing LN_2 fill system to the new automated system was identified and ordered and a phase separator system was ordered to upgrade the LN_2 delivery system. This phase separator system will decrease gaseous nitrogen pressure fluctuations and more efficiently and safely provide nitrogen to NSLS users. Installation will continue into 2008.

The last item to mention regarding overall facility improvement is the Automated Process and Control Software (APACS). This software was installed on a dedicated utilities systems computer and will provide accurate control and faster troubleshooting for NSLS (high pressure copper, low pressure copper, experimental, and aluminum) cooling water systems. This software collects input from numerous sensors so that corrective actions can be determined remotely.

Injection System

We continued to improve the injection system diagnostics this year. After the wall current monitor (WCM) was installed at the entrance of the Booster-to-X-Ray transport

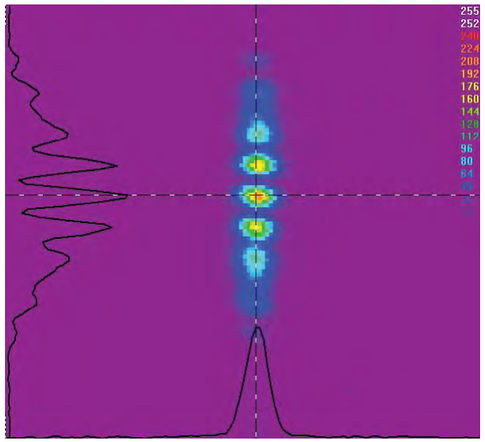


Figure 1
U3B double-slit interferogram (skew quad off). Relative depth of interference fringes and imaging optics parameters result in the vertical beam size of 110 um.

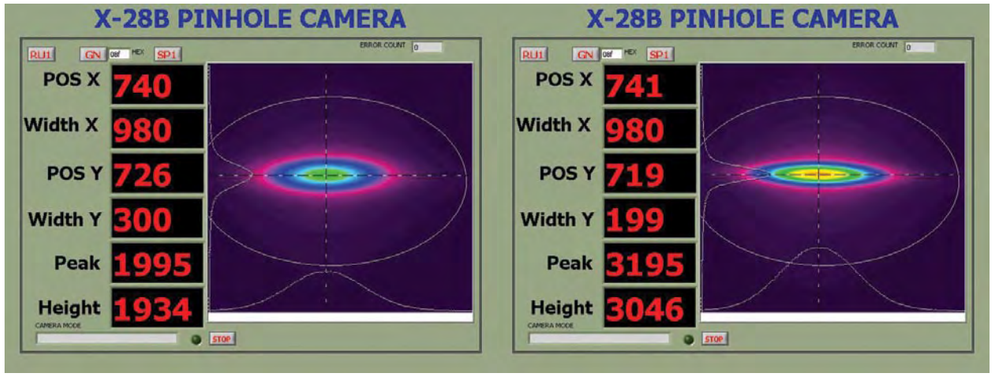


Figure 2 (above)
X28 pinhole camera image before (left) and after coupling correction.

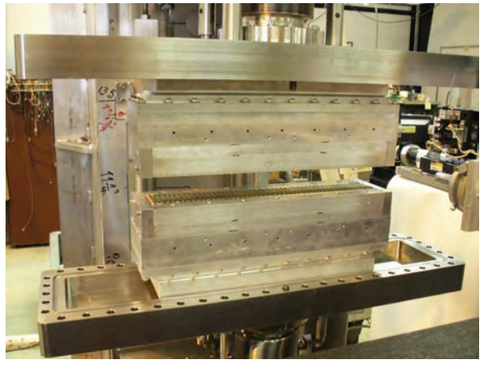


Figure 3 (left)
X9 undulator mechanical assembly in progress.

line, the strong noise signal from the neighboring septa was largely removed by a calibrated high-pass filter so that in conjunction with the WCM at the end of the transport line, the efficiency from Booster to the transport line and the efficiency of the transport line can be monitored and tuned up separately. We made significant progress in developing diagnostics software, improving the frame grabber and Booster performance monitor programs. The MatLab program to capture and process video images of any beam flag in transfer lines was completed. The program provides a function to chart the beam parameters against any of the control devices used to tune the beam in transfer lines, and it was compiled into stand-alone executables readily available for the operators.

The beam-position monitor commissioned last year called "Orbtrak" had a new PC interface-based analog-to-digital converter card installed to replace the oscilloscope and a MatLAB routine was written to read the digitizer in real time for post processing. The synchrotron radiation monitor image acquisition and processing system were routinely used to obtain the beam intensity, size, and position through the Booster ramp.

Two horizontal pick-up electrodes, one on the X-Ray extraction section and the other on the UV extraction section, were revived so that we can measure the beam orbit at the X-Ray extraction section and UV extraction section simultaneously. We developed a multiwavelength anomalous diffraction model including the backleg windings, which are used to distort the beam orbit very close to the extraction septum. The model agrees with the experiment within 5%, and it's in the process of being used to optimize the extraction process.

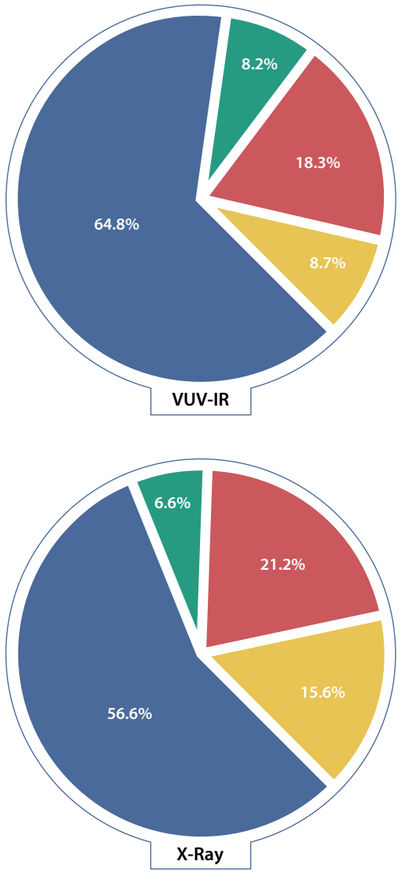
Based on the input of the newly developed model, an active study program of the injection system was conducted. Extraction parameters have been optimized to increase injection efficiency into the X-Ray Ring, which reduces the time spent injecting into the storage rings and allows for more operation time.

A project to upgrade the kicker drivers for BXESH1, the Linac-to-Booster Injection Septum (LBISH), and two Booster-to-VUV Extraction septa (BUESH1 and 2) also is in progress. These magnets require thousands of amps in pulses of several tens of microseconds in length to generate the fields of a few kilogauss to deflect the electron bunches into the booster synchrotron and out of the booster into the extraction transport lines. The new design will provide improved operation as well as improved serviceability, which will reduce downtime due to component failures. BXESH1 and LBISH construction is on schedule for installation during the spring 2008 machine shutdown.

Further safety improvements include the completion of the Safety System Power Supply Interface project, which turns off the booster main magnet power supplies when ring security is broken. A system also was installed in the booster security system to indicate when all magnet power supplies are in an off condition. The system will not permit access to the area if all supplies are not reading off.

VUV Ring

A double slit interferometric setup to measure vertical beam size in the U3B diagnostic beamline has been implemented and tested (**Figure 1**). This has an advantage over the conventional synchrotron light imaging from a bend magnet (such as the Spiricon system at U5) in that it can work at low beam energy and measure very small beam sizes. This system will serve as a prototype for a future vertical beam size measurement during X-Ray Ring injection, which we don't have at present. Several repairs also were made in the VUV Ring, including an emergency repair to the U13 safety shutter and a VUV beam scraper vacuum leak.



X-Ray Ring

MATLAB and Middle Layer software packages used for measurements and control of beam properties in both rings have been upgraded for improved functionality. A new lattice with reduced sextupole strength and a lower emittance coupling ratio was established and put into regular operations in spring 2007 (**Figure 2**).

During the winter 2006-07 shutdown, all X9 front-end components and the dipole chamber were removed, and the new dipole vacuum chamber with the 0° port for an X9 undulator beamline was installed. A new front end was designed for the higher undulator heat loads and unique needs (e.g. no Be window) of the X9 undulator and beamline. The water-cooled aperture, water-cooled mask, fast valve, front end vacuum valve, two ion pumps, the safety shutter and all related stands were installed during the winter 2007-08 shutdown in preparation for the final installation during the spring 2008 shutdown.

As part of the overall X9 mini-gap undulator (MGU) installation, the vacuum chamber was vented and an upstream pick-up-electrode (PUE) was welded in place during the winter 2007-08 shutdown. (The downstream PUE was installed in an earlier shutdown). These PUEs feed horizontal and vertical position data to the X9 Active Interlock system. An Active Interlock system will protect the vacuum pipe and is critical for operation of the MGU, which produces energies capable of damaging the vacuum pipe. This PUE installation ended in a carefully orchestrated bake-out of 1.5 superperiods, which included four delicate ceramic vacuum chamber pieces. Thanks to this careful planning, the ceramic pieces suffered no damage.

Several major components of the X17 cryogenic system were rebuilt, repaired, or replaced. The components include the refrigerator/liquifier (rebuilt), one helium compressor (repaired), and an oil cooler (replaced). Leaks in several locations were identified and repairs were made as necessary.

Insertion Devices

The new in-vacuum MGU for the planned CFN beamline at X9 is under construction. The X9 MGU, like its predecessor at X29, has a magnetic array about 36 cm long, and a magnetic period of 14.5 mm (compared to 12.5 mm for X29). The flange-to-flange length is also 100 cm and it will fit between the two radio frequency accelerating cavities in the X9 straight. This device is designed to produce a peak field of 1.25 Tesla at the minimum gap of 3.3 mm, giving a tuning range of 2.2–4.5 keV at the fundamental. With a significant 2nd harmonic and the usual odd harmonics (up to the 9th), this device will provide full-spectrum coverage from 2.2 to 20 keV and beyond. The mechanical design of the predecessor X29 MGU was revised and upgraded to accommodate the higher magnetic field and the resultant doubling of magnetic forces. Fabrication was done in-house (**Figure 3**).

The magnetic design was optimized for a high-field, high-temperature grade of permanent magnets (NEOMAX 42AH) developed for hybrid car motors. Using two magnet arrays, populated with sacrificial 42AH samples, we determined that a maximum bake temperature of 90° C is adequate. Then, the remaining magnets were pre-baked at 90° C, re-measured, and the data was put into the sorting code that was developed in-house. Based on the code output, the magnets were swapped to minimize the root mean square (rms) trajectory error. The magnets were then installed in the arrays according to the resulting final sequence and subsequent magnetic measurements have shown that the final magnet assembly is well within the specifications for dipole error as well as the rms phase error, so no further adjustments (shimming) will be required.

Final steps of mechanical assembly, preparation for the bakes, and final magnetic measurements are in preparation or underway. The device is on schedule for installation in the X-Ray Ring during the spring 2008 machine shutdown.

Source Development Laboratory

The Source Development Laboratory (SDL) is a linear accelerator and laser-based R&D facility dedicated to the exploration of new radiation sources and high-brightness electron beams. In 2007, the scientific program emphasized three key themes:

- High-Gain Laser-Seeded Free Electron Laser (FEL) Amplifiers
- High-Intensity Terahertz (THz) Radiation
- Ultrafast Electron Diffraction

The high-gain laser-seeded FEL amplifier program, at a wavelength of $\lambda = 800$ nm, is funded by the Office of Naval Research. Researchers developed two schemes to increase the efficiency of conversion of electron kinetic energy to FEL light: electron energy detuning and undulator tapering. The two schemes provided a doubling and tripling, respectively, of the FEL efficiency. (X. Wang *et al.*, "Efficiency Enhancement Using Electron Energy Detuning in a Laser Seeded Free Electron Laser Amplifier," *Appl. Phys. Lett.*, **91**: 181115 (2007).)

Record-high intensity THz radiation (100 μJ/pulse) was generated from transition radiation by colliding the intense electron bunches (500 pC in 1 psec) at the SDL with an aluminum mirror. The THz radiation properties were explored using electro-optical techniques and exciting new nonlinear cross phase modulation of the titanium-sapphire laser probe pulse was observed. *(See page 32 for the science highlight about this work).*

The laser-seeded radio frequency (RF) photoinjector developed at the NSLS is serving as the nucleus of a new ultrafast electron diffraction source under development in 2007. This novel source will provide ultrashort electron bunches (~100 femtoseconds) containing 105 electrons at energies of 1-4 MeV to make time-resolved diffraction studies of gases and thin films possible.

"2007 was a busy and productive first year for the newly formed Experimental Systems Division"

Steven Hulbert
Experimental Systems Division Head

The major activities, projects, and accomplishments of the Experimental Systems Division in 2007 are summarized below:

Beamline R&D

The Beamline R&D Section, led by Lonny Berman, was created as part of the April 2007 NSLS reorganization, evolving from its predecessor in the previous organization. Probably the most visible examples of work involving members of the Beamline R&D Section are beamline and endstation construction and upgrade projects. One such project, completed in 2007, was the upgrade of beamline X25, whose radiation source and beamline optics were replaced to optimize them for X25's macromolecular crystallography program. To exploit the radiation properties of its new in-vacuum mini-gap undulator, the beamline optics were replaced in early 2007. The new optics consist of a double silicon crystal monochromator with cryogenic cooling of the first crystal and sagittal bending of the second crystal to focus the beam horizontally, followed by a bendable mirror to focus the beam vertically. A comparison of the measured spectrum from the undulator, with theory, is shown in **Figure 1**. At a wavelength of 1.1 Å, the beam intensity delivered through a 100 μm square aperture at the focal position in the X25 hutch increased by a factor of five compared to the old wiggler beamline.

A second major project in 2007 was the construction of the new X9 undulator beamline for small angle x-ray scattering (SAXS). Jointly developed by the NSLS and the Center for Functional Nanomaterials (CFN), X9 is capable of carrying out SAXS measurements in both transmission and grazing incidence geometry within a wide x-ray energy range from 2 to 20 keV and spot sizes as small as 10 microns in the sample. Upon completion, X9 will host the SAXS program that currently exists at beamline X21 and will provide much-needed beam time for the life sciences, soft condensed matter physics, and nanoscience communities.

Several other projects involving members of the Beamline R&D Section were underway in 2007 and will continue in 2008. These projects, all of which are included in the NSLS 5-Year Plan, include: replacement of the X7B monochromator with one that is optimized for high x-ray energies and will support the new pair distribution function program at that beamline; replacement of the X18A monochromator with one that will permit expansion of that beamline's experimental program to include quick extended x-ray absorption fine structure (QEXAFS) and diffraction; and a new endstation for beamline X13A that seeks to broaden the capabilities of the soft x-ray scattering program by providing a switchable high-field magnet to surround the sample and a large scattering angle range for the detector, among other things. Many of these projects are led by members of the science divisions, and involve participation by members of the Beamline R&D Section.

Optics R&D is an important aspect of the Beamline R&D Section's mission. Two R&D efforts pursued by members of the section are of importance for NSLS-II as well as for NSLS. One of these is led by NSLS physicist Ken Evans-Lutterodt and addresses the design and development of kinoform refractive x-ray lenses for nanofocusing. This R&D has mainly been pursued at beamline X13B, where it was proven that resolution and aperture restrictions, resulting from the critical angle associated with the lens material, can be overcome through implementation of compound lenses, as shown in **Figure 2**. More recently, further optimization of these lenses is being conducted and verified through measurements undertaken at Argonne National Laboratory's Advanced Photon Source (APS). The long-term goal of this effort is to be able to develop an optic that can focus an x-ray beam to a dimension of 1 nm, which is one of the major goals for NSLS-II. *(See page 30 for the science highlight about this work).*

A second R&D effort, led by NSLS physicist Zhong Zhong, addresses the further development of back-reflection

Figure 1
Measured brightness of the new X25 undulator, compared with simulation. From J.M. Ablett and L.E. Berman, *Nucl. Instrum. Meth. A*, **582**: 37-39 (2007).

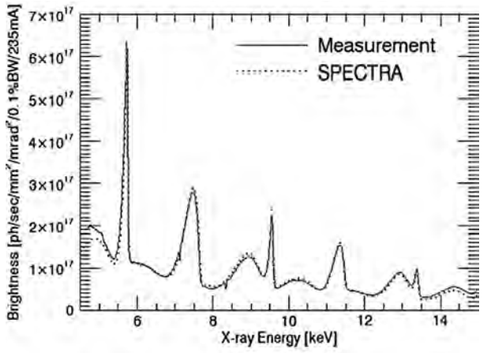


Figure 2
Compound kinoform lenses etched in silicon, which were used to demonstrate that the critical-angle-imposed restriction on the resolution and aperture of refractive lenses can be overcome. From K. Evans-Lutterodt, A. Stein, J. M. Ablett, N. Bozovic, A. Taylor, and D. M. Tennant, *Phys. Rev. Lett.*, **99**:134801 (2007).

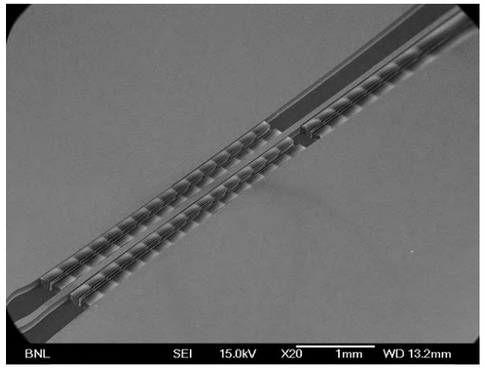


Figure 3
80mm-long silicon strip detector consisting of 640 strips 0.125 x 4 mm².

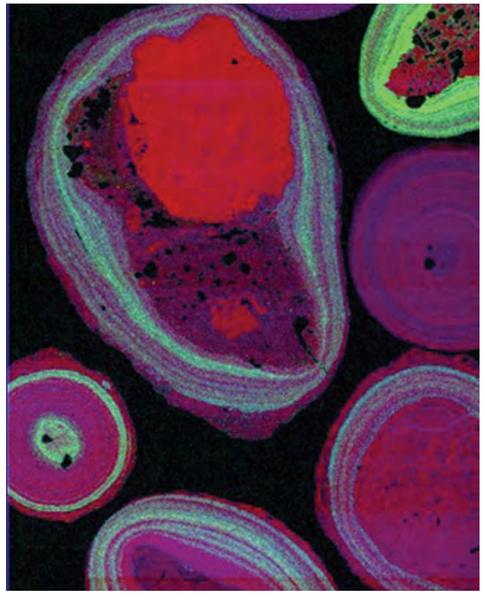


Figure 4
Megapixel elemental map of iron-rich mineral nodules from Washington state, measured in a few hours using the new integrated detector and scanning system for x-ray microprobe applications.

wavelength-dispersing analyzer crystals that can be incorporated in optical systems designed to deliver high-energy resolution, ultimately, as fine as 0.1 meV. This also is a major goal for NSLS-II. The first set of measurements, which were done at NSLS beamline X12A in late 2007, resulted in a prototype that demonstrated the concept of wavelength dispersion on which the performance of this device depends. The results corroborate with the findings of the pioneer of this concept, Yuri Shvyd'ko, who made earlier measurements at the APS. Further R&D work will be carried out in 2008 to push the limits of the capabilities of such devices, in collaboration with members of the NSLS-II Project.

Finally, an important, and rather new, focus of the Beamline R&D Section concerns measuring and ensuring the performance of NSLS beamlines. This effort is being coordinated by the newest member of the section, Joe Dvorak. Joe will focus his attention on vetting the performances of our beamlines and, by working with beamline and accelerator staff, will suggest measures to improve them. This is important for keeping NSLS beamlines at state-of-the-art performance, for maintaining the NSLS facility metrics, and also for judging and ensuring the suitability of beamlines for possible transfer to NSLS-II.

Detectors

The mission of the Detectors Section, headed by Peter Siddons, is threefold: to develop and implement new detectors for synchrotron applications, to support these systems for beamline use at the NSLS, and to support those systems provided to other labs as necessary.

In 2007, the Detectors Section delivered three 80 mm silicon strip detectors, one to Argonne, one to Brookhaven's CFN, and one as a pool instrument for general use (**Figure 3**). These detectors are based on an Application-Specific Integrated Circuit (ASIC) designed by Gianluigi de Geronimo from BNL's Instrumentation Division. The strip detector uses 20 of these ASICs to instrument 640 detectors arranged as a row of strips, each 0.125 x 4 mm², thus forming a one-dimensional position-sensitive detector. Each strip can handle up to 105 photons per second, and has an energy resolution of 350 eV at 6 keV. Our project to build a large array of such strip detectors for time-resolved powder diffraction is mostly designed, but is currently on hold due to budgetary considerations.

We have also continued work on the fast imaging detector for the LCLS Ultrafast Science Instruments (LUSI) project. LUSI is the beamline portion of Stanford Linear Accelerator Center's x-ray free-electron laser project, and these detectors are required to record full 1k x 1k images at 120 Hz, with a readout noise level less than one 8 keV photon, and a full-well of 104 photons. We are working on two versions of the sensor, one fabricated in-house based on junction gate field-effect transistor technology, and the other in collaboration with IBM based on metal-oxide semiconductor technology.

We have also made successful tests of a new, very fast, integrated detector and scanning system for x-ray microprobe applications. This system makes the collection of megapixel elemental maps quite straightforward, offering 100 times faster acquisition than conventional systems, and real-time spectral analysis (**Figure 4**). This work is done in collaboration with the Commonwealth Scientific and Industrial Research Organization (CSIRO) in Australia. The CSIRO members are responsible for the high-speed computing and the NSLS is responsible for the detector and readout electronics. The system is closely integrated with the X-Y scanning stage to allow continuous scanning, and the data analysis is based on CSIRO physicist Chris Ryan's Dynamic Analysis principle, which essentially eliminates errors arising from peak overlap in complex spectra. *(See page 42 for the science highlight about this work).*

The developers of the fast spectroscopy system: From left, Robin Kirkham, Gareth Moorhead, John Kuczewski, Chris Ryan, Tony Kuczewski, and Pete Siddons.

Mechanical Systems

The Mechanical Systems Section, led by Ed Haas, provides mechanical engineering, design, and technical support to all NSLS divisions, both for operations and for projects, large and small. Operationally, the Mechanical Systems section continued its preventative maintenance and emergency repair efforts on all systems and components in the Vacuum Ultraviolet (VUV), Booster, and X-Ray rings with the goal of better than or equal to 95 percent reliability of the storage rings. As part of this effort, numerous components subject to wear and degradation were replaced (hoses, fittings, valves, and connectors), rebuilt (e.g. deionizers), and calibrated (e.g. gauges and instruments) during 2007.

NSLS projects for which the Mechanical Systems Section provided effort during 2007 are detailed below:

Accelerator

Injection System: We initiated a project to replace four failing ion pumps in the booster ring, and development of a plan to add additional shielding (lead and borated polyethylene).

VUV Ring: Repairs were performed on a leaking beam scraper, the U13 safety shutter, and the U15 mask.

X-Ray Ring: A number of activities were performed, including the preparation and installation of a new ring vacuum chamber for the new X9 mini-gap undulator (MGU), removal of the old X9 front end components followed by engineering and design of the new front end, and engineering and design of the new X9 MGU and beamline; decommissioning, disassembly, and removal of the X5 Laser Electron Gamma Source equipment, both inside and outside the X-Ray Ring shield wall; two projects related to improvement of the vacuum measurement systems in the X-Ray Ring and front ends; in response to a 2006 Machine Operations and Readiness Evaluation recommendation, three spare X-Ray Ring ceramic assemblies were fabricated in 2007 for ready replacement in case of vacuum failure.

Insertion Devices: Engineering, detailed design, and construction of the X9 mini-gap undulator (MGU) began in-house in 2007. This effort required a large new engineering and design effort owing to the significantly higher magnetic load forces in addition to the difference in period length (14.5 mm for X9 compared to 12.5 mm for X29 and X13). The X9 MGU development differed significantly from past projects because of the temperature-sensitive magnetic material, which required much more precise, accurate, and reliable control of the magnet temperature during bake-out. This project will extend into 2008, with installation in the X-Ray Ring planned for the May 2008 maintenance period. The downstream pick-up electrode (PUE) needed for this project was installed during 2006, and preparations for installing the upstream PUE occurred in 2007, with installation planned for the December 2007 maintenance period.

Owing to the age and condition of the X17 cryogenic system, two possible plans for upgrade of this system were developed and investigated by the NSLS with assistance from Brookhaven's Superconducting Magnet Division and the Collider-Accelerator Department. These two minimal-risk, minimal-cost cryogenic system upgrade plans were presented to an X17 Cryogenic System Review committee, which included several outside-NSLS reviewers and led to recommendation of one of the plans and concurrence with the capital and operating costs estimated by the NSLS/BNL group. Alternative plans to obtain a new cryo-cooled superconducting wiggler are being investigated, all of which would obviate the need for the present X17 cryogenics plant. In the meantime, the existing X17 cryogenic system continues to be maintained. In 2007, several major components of the X17 cryogenic system were rebuilt, repaired, or replaced.

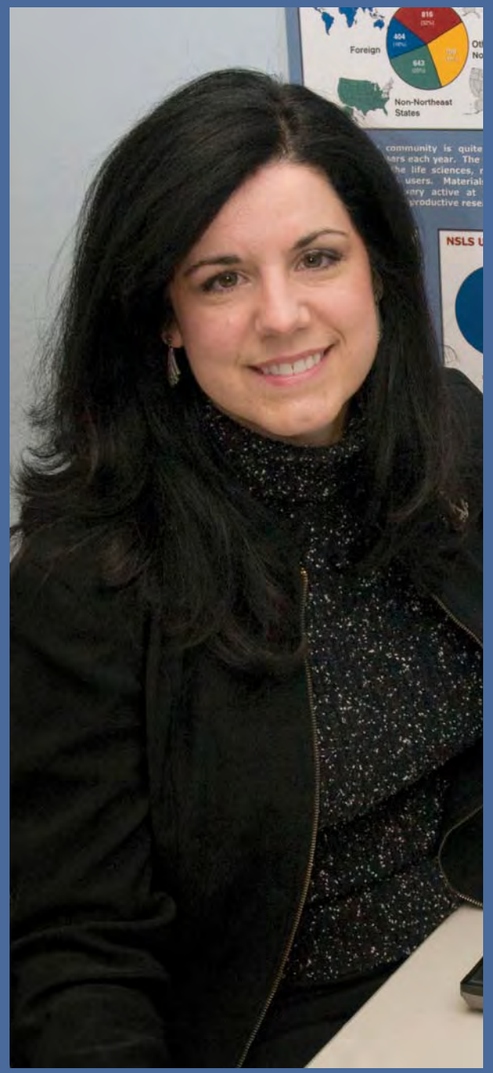
Utilities: Automated Process and Control Software was acquired and installed on a dedicated utilities systems computer to provide accurate control and faster troubleshooting for NSLS cooling water systems. This industry-standard software collects inputs from numerous temperature, pressure, valve position, and fluid flow sensors so that troubleshooting information can be analyzed and corrective actions determined remotely.

Beamlines

In addition to the mechanical engineering, design, and technical development and support for the beamline activities described earlier in this report, an effort was led by the Mechanical Section in 2007 to assess all NSLS beamline vacuum systems for compliance with the DOE 10CFR851 pressure safety requirements. This effort included participation in inter-laboratory communication with DOE and the submission of an action plan for establishing guidelines consistent with these requirements – work which will extend into 2008.

Safety Improvements

The Mechanical Section remains proactively involved in efforts to improve the safety of mechanical systems at NSLS. During 2007, these efforts included: installation of five remote motor control starters on utilities pumps to effectively remove arc flash risk to personnel; initiation of an automated liquid nitrogen fill system project to fill users' dewars more safely and efficiently; initiation of a project to develop and implement an efficient gaseous nitrogen utility for users, which will decrease pressure fluctuations.



"2007 was a record-breaking year with 985 user and staff publications, nine conferences and workshops, 151 seminars, and 80 tours of the facility."

Kathleen Nasta
NSLS User Administrator

Facility Facts and Figures

As a national user facility, the NSLS operates seven days per week, 24 hours per day, except during maintenance and studies periods, and does not charge for beam time if the research results are published in open literature. Work that is proprietary in nature is charged on a full-cost recovery basis. Researchers, called "users," primarily obtain beam time by submitting a "General User" (GU) proposal through the peer-reviewed proposal system. The proposal is rated based on various scientific criteria, and can remain active for up to six cycles of operations (or about two years). In fiscal year 2007, nearly 1,000 requests for beam time were submitted, well over one-third of the requests on new proposals.

The NSLS currently has 51 x-ray and 14 vacuum ultraviolet-infrared operational beamlines available for a wide range of experiments utilizing many different techniques. There are two types of beamlines at the NSLS: Facility Beamlines (FBs), of which there are 18; and Participating Research Beamlines (PRTs), currently totaling 47. FBs are operated by the NSLS staff and reserve a minimum of 50 percent of their beam time for GUs. Some FBs also host Contributing Users (CUs), who enhance endstation capabilities and provide generalized support to users. PRT beamlines are operated by user groups with related interests from one or more institutions. PRT beamlines reserve 25 percent of their beam time for GUs, although they can grant additional time at their own discretion. Membership in a PRT or CU program is open to all members of the scientific community who can contribute significantly to the program of the beamline. For example, this can include funding, contribution of equipment, scientific program, design and engineering, or operations staffing. The following pages detail operational beamlines at the NSLS and their unique characteristics.

Throughout calendar year 2007, many visitors attended special workshops, short courses, seminars, and tours at the NSLS. In all, nine workshops and conferences were held, 151 seminars were hosted, and 80 tours of the facility were given. In fiscal year 2007, more than 2,200 users representing almost 400 different institutions came to the NSLS to conduct their research. Of this number, about 700 were new users – evidence that the NSLS user community continues to grow. About 7 percent of users were from corporate organizations, while the majority continues to come from academic institutions.

These users can be characterized by their field of research in different ways. The greatest number of users works in the life sciences field, while materials sciences studies follow closely. When considering the number of days spent on experiments – based on running times gathered from experimental Safety Approval Forms – materials sciences studies makes up the greatest portion of beam time used.

About half of our users are U.S. citizens, and more than 25 percent are women. One-third of our users are from within New York State, and almost another third come from locations nearby in the northeastern states.

In 2007, students in undergraduate or graduate studies made up more than 40 percent of the researchers working at the NSLS. Thirteen percent were post-doctoral researchers, while the remaining researchers were faculty members, professional staff, or research scientists.

Breaking last year's record, NSLS users and staff had 985 publications in fiscal year (FY) 2007, which ran from October 1, 2006 through September 30, 2007. Of these, about 20 percent were published in premier journals. A publication is considered premier if the journal has an impact factor of 6 or greater (from Journal Citation Report 2005, Thomson Institute for Scientific Information). The table shown in the following pages summarizes publications during the past year. The first column in the table lists the number of publications reported to the NSLS during FY07 and published between 2004 and 2007. Although some of these publications were published earlier than FY07, they were not reported to the NSLS until this fiscal year. Thus, they have not been counted in prior years' reports. The second column in the table lists the number of papers published in the 2007 calendar year and reported to the NSLS as of February 29, 2008. These numbers are slightly lower than the fiscal year values because they contain only publications from 2007, and it often takes many months or years to account for user and staff publications.

X-Ray and VUV-IR Parameters

	X-Ray	VUV-IR
Energy	2.800 GeV	0.808 GeV
Maximum Current	300 mA	1.0 A
Photon Critical Wavelength (Energy) for Dipole	1.75 Å (7.1 keV)	19.9 Å (622 eV)
Photon Critical Wavelength (Energy) for X17 wiggler at 4.2T	0.57 Å (22 keV)	
Horizontal Emittance	62 nm-rad	160 nm-rad
Vertical Emittance	0.34 nm-rad	≥ 0.35 nm-rad (4 nm-rad in normal ops.)
Electron Orbital Period	567.2 nanoseconds	170.2 nanoseconds
Number of RF Buckets	30	9
Typical Bunch Mode	25	7
Natural RMS Energy Spread	9.2×10^{-4}	5.0×10^{-4} ($I_b < 20$ mA)
RMS Bunch Length	44 mm	5 cm ($I_b < 20$ mA)

2007 Publications

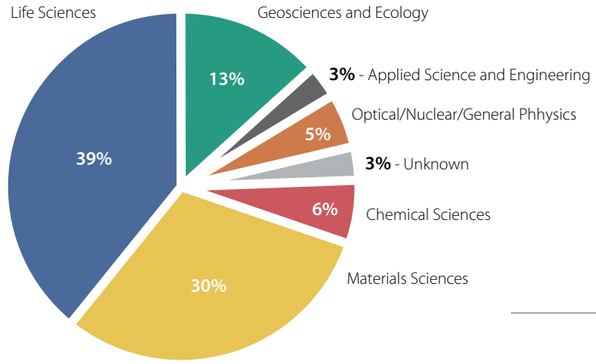
	Reported in FY07*	Published in CY07**
Journals, peer-reviewed, premier	189	195
Journals, other peer-reviewed	646	595
Journals, non peer-reviewed	36	24
Books/Chapters in Books	6	10
Published Conference Proceedings	74	41
Reports: Technical, Formal, Informal	3	5
Theses/Dissertations	25	23
Patents	6	1
Total Publications	**985**	**894**
NSLS VUV-IR User Publications	102	97
NSLS X-Ray User Publications	758	669
NSLS Staff Publications	125	128
Total Publications	**985**	**894**

* Publications reported to the NSLS from Oct. 1, 2006 - Sept. 30, 2007 and published between 2004 - 2007.

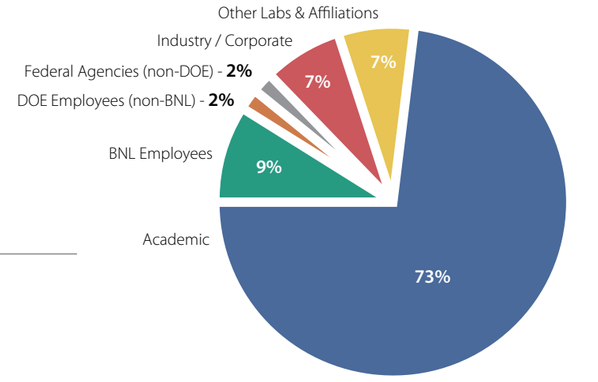
** Publications published in 2007 as reported to the NSLS by Feb. 29, 2008.

Facility Facts and Figures

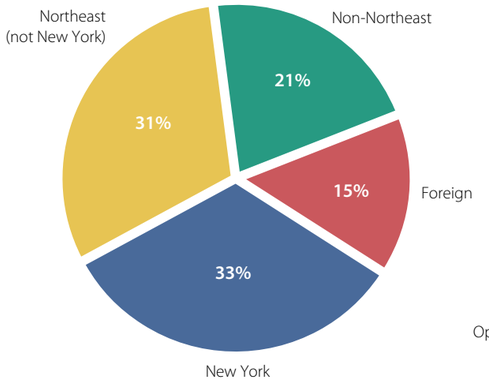
Users by Field of Research
Fiscal Year 2007



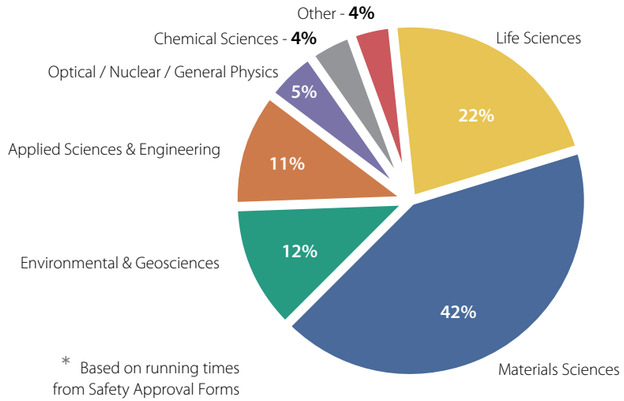
Users by Affiliation
Fiscal Year 2007



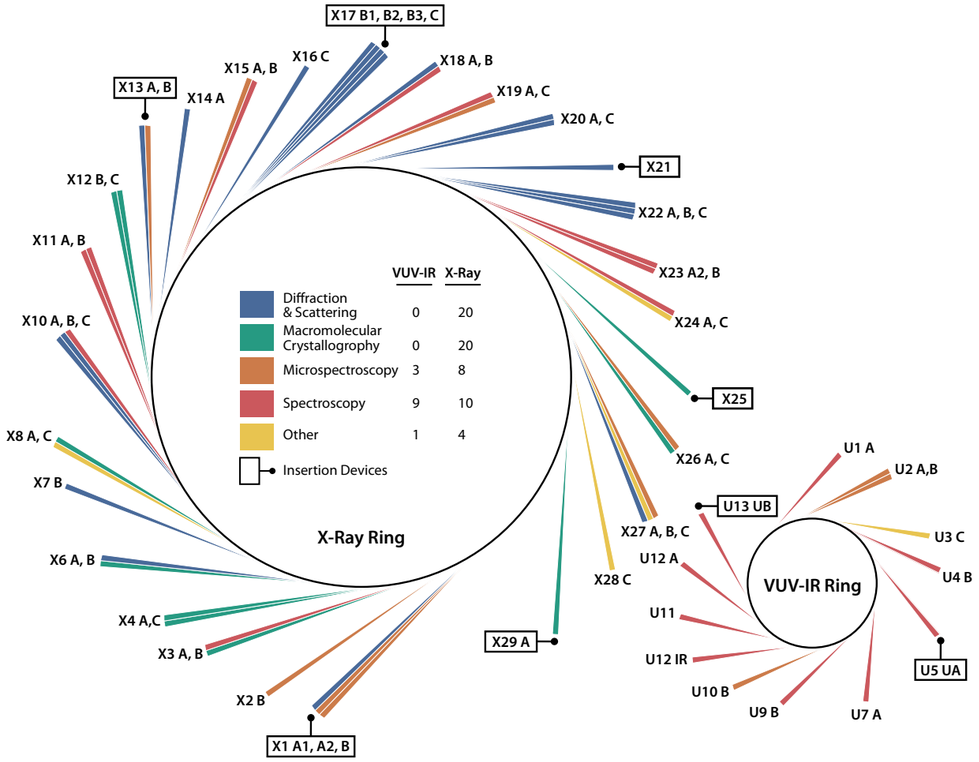
Geographical User Distribution
Fiscal Year 2007



Beamtime Used*
Fiscal Year 2007



* Based on running times from Safety Approval Forms



Beamline Guide Abbreviations

ARPES	UV Photoelectron Spectroscopy, Angle-Resolved	**SAXS**	Small Angle X-Ray Scattering
DAFS	X-Ray Diffraction Anomalous Fine Structure	**STXM**	Scanning Transmission X-Ray Microscopy
DEI	Diffraction-Enhanced Imaging	**UPS**	UV Potoelectron Spectroscopy
EXAFS	X-Ray Absorption Spectroscopy, Extended Fine Structure	**UV-CD**	Ultraviolet Circular Dichroism
HARMST	High Aspect Ratio Microsystems Technology	**WAXD**	Wide-Angle X-Ray Diffraction
IRMS	Infrared Microspectroscopy	**WAXS**	Wide-Angle X-Ray Scattering
MAD	Mulit-Wavelength Anomolous Dispersion	**XAS**	X-Ray Absorption Spectroscopy
MCD	Magnetic Circular Dichroism	**XPS**	X-Ray Photoelectron Spectroscopy
NEXAFS	Near Edge X-Ray Absorption Spectroscopy	**XRD**	X-Ray Diffraction
PEEM	Photo Emission Electron Microscopy	**XSW**	X-Ray Diffraction, Standing Waves

Beamline	Source	Type of Research	Energy Range	Type	Organization
VUV-IR Beamlines					
U1A	Bend	XAS EXAFS NEXAFS	270-900 eV	PRT	ExxonMobil Research and Engineering Co.
U2A	Bend	IRMS High pressure research IR spectroscopy	30-8000 cm^{-1}	FB	BNL-NSLS Carnegie Institution of Washington COMPRES
U2B	Bend	IRMS IR spectroscopy	50-4000 cm^{-1}	PRT	Case Western Reserve University
U3C	Bend	Metrology	50-1000 eV	PRT	Lawrence Livermore National Laboratory Los Alamos National Laboratory National Security Technologies Sandia National Laboratory
U4B	Bend	X-ray scattering, resonant MCD UPS X-ray fluorescence spectroscopy XPS	20-1200 eV	FB	BNL-NSLS Montana State University
U5UA	Insertion Device	ARPES UPS, spin-resolved PEEM	15-150 eV	FB	BNL-NSLS BNL-CFN
U7A	Bend	NEXAFS XPS	180-1200 eV	PRT	BNL-Chemistry Dow Chemical Company NIST University of Michigan
U9B	Bend	Time-resolved spectroscopy UV-CD UV florescence spectroscopy	0.8 - 8 eV	PRT	BNL-Biology
U10B	Bend	IRMS	500-4000 cm^{-1}	FB	BNL-NSLS
U11	Bend	UV-CD	3-10 eV	PRT	BNL-Biology
U12A	Bend	XAS XPS	100-800 eV	PRT	Oak Ridge National Laboratory
U12IR	Bend	IR spectroscopy Magnetospectroscopy THz / millimeter wave spectroscopy Time-resolved spectroscopy	8-600 cm^{-1}	FB	BNL-NSLS
U13UB	Insertion Device	UPS ARPES	3-30 eV	PRT	Boston College Boston University BNL-Physics Columbia University

Beamline	Source	Type of Research	Energy Range	Type	Organization
X-Ray Beamlines					
X1A1	Insertion Device	STXM NEXAFS	0.25-0.50 keV	PRT	BNL-Environmental Sciences ExxonMobil Research and Engineering Co. SUNY @ Plattsburgh Stony Brook University University of Texas @ Houston
X1A2	Insertion Device	STXM	0.25-1 keV	PRT	Stony Brook University
X1B	Insertion Device	X-ray scattering, coherent XAS X-ray fluorescence spectroscopy XPS	0.2-1.6 keV	PRT	Boston University Thomas Jefferson National Accelerator Facility University of Illinois
X2B	Bend	X-ray microtomography	8-35 keV	PRT	ExxonMobil Research and Engineering Co.
X3A	Bend	MAD Macromolecular crystallography	5-15 keV	PRT	Case Western Reserve University Rockefeller University Sloan-Kettering Institute
X3B	Bend	XAS EXAFS	5-15 keV	PRT	Case Western Reserve University
X4A	Bend	MAD Macromolecular crystallography	3.5-20 keV	PRT	Albert Einstein College of Medicine City University of New York (CUNY) Columbia University Cornell University Mount Sinai School of Medicine New York Structural Biology Center New York University SUNY @ Buffalo Sloan-Kettering Institute Wadsworth Center
X4C	Bend	MAD Macromolecular crystallography	7-20 keV	PRT	Albert Einstein College of Medicine City University of New York (CUNY) Columbia University Cornell University Mount Sinai School of Medicine New York Structural Biology Center New York University Rockefeller University SUNY @ Buffalo Sloan-Kettering Institute Wadsworth Center
X6A	Bend	MAD Macromolecular crystallography	6-23 keV	FB	BNL-NSLS
X6B	Bend	XRD, surface WAXD X-ray reflectivity	6.5-19 keV	FB	BNL-NSLS BNL-CFN
X7B	Bend	XRD, single crystal XRD, time resolved WAXD WAXS	5-21 keV	PRT	BNL-Chemistry General Electric
X8A	Bend	Metrology	1-5.9 keV	PRT	Bechtel Nevada Lawrence Livermore National Laboratory Los Alamos National Laboratory National Security Technologies Sandia National Laboratory
X8C	Bend	MAD Macromolecular crystallography	5-19 keV	PRT	Biogen Incorporated Biotechnology Research Institute Hoffmann-La Roche National Institutes of Health
X10A	Bend	XRD, powder WAXD SAXS WAXS	8-11 keV	PRT	ExxonMobil Research and Engineering Co.
X10B	Bend	XRD, powder XRD, surface WAXD X-ray reflectivity X-ray scattering, surface WAXS	14 keV	PRT	ExxonMobil Research and Engineering Co.
X10C	Bend	XAS EXAFS NEXAFS	4-24 keV	PRT	ExxonMobil Research and Engineering Co.
X11A	Bend	DAFS XAS EXAFS NEXAFS	4.5-35 keV	PRT	BNL-Material Sciences BNL-Environmental Sciences Canadian Light Source ETH Labs - Zuerich Natural Resources Canada Naval Research Laboratory (NRL) Naval Surface Warfare Center New Jersey Institute of Technology North Carolina State University Stony Brook University Sarah Lawrence College
X11B	Bend	XAS EXAFS NEXAFS	5-23 keV	PRT	BNL-Environmental Sciences BNL-Material Sciences Canadian Light Source ETH Labs - Zuerich Natural Resources Canada Naval Research Laboratory (NRL) Naval Surface Warfare Center New Jersey Institute of Technology North Carolina State University Stony Brook University Sarah Lawrence College
X12B	Bend	MAD Macromolecular crystallography	5-20 keV	PRT	BNL-Biology
X12C	Bend	MAD Macromolecular crystallography	5.5-20 keV	PRT	BNL-Biology
X13A	Insertion Device	X-ray scattering, magnetic X-ray scattering, resonant MCD	0.2-1.6 keV	FB	BNL-NSLS
X13B	Insertion Device	Microdiffraction Imaging	4-16 keV	FB	BNL-NSLS BNL-CFN Columbia University IBM
X14A	Bend	MAD XRD, powder XRD, single crystal XRD, time resolved WAXD X-ray reflectivity	5-26 keV	PRT	New York State College of Ceramics Oak Ridge National Laboratory Tennessee Technological University University of Tennessee
X15A	Bend	XSW DEI	3-25 keV XSW 10-60 keV DEI	FB	BNL-NSLS

Beamline	Source	Type of Research	Energy Range	Type	Organization
X15B	Bend	XAS EXAFS NEXAFS	0.8-15 keV	PRT	BNL-Environmental Sciences Lucent Technologies, Inc. Stony Brook University Temple University University of Texas @ Austin
X16C	Bend	XRD, powder	6.5-25 keV	PRT	Stony Brook University
X17B1	Insertion Device	XRD, powder	55-80 keV mono 20-150 keV white	FB	BNL-NSLS Rutgers University
X17B2	Insertion Device	XRD, powder XRD, time resolved High pressure research	20-130 keV	FB	BNL-NSLS COMPRES Stony Brook University
X17B3	Insertion Device	XRD, powder XRD, single crystal High pressure research	5-80 keV	FB	BNL-NSLS COMPRES University of Chicago
X17C	Insertion Device	XRD, powder XRD, single crystal High pressure research	5-80 keV	FB	BNL-NSLS COMPRES University of Chicago
X18A	Bend	XRD, powder XRD, single crystal XRD, surface WAXD X-ray reflectivity X-ray scattering, surface WAXS	4-19 keV	PRT	BNL-Chemistry Indiana University @ Bloomington Pennsylvania State University Purdue University Stony Brook University University of Missouri @ Columbia
X18B	Bend	XAS EXAFS NEXAFS	4.8-40 keV	FB	BNL-NSLS BNL-Chemistry BNL-Electrochemistry ORNL University of Delaware UOP LLC Yeshiva University
X19A	Bend	X-ray scattering, resonant XAS EXAFS NEXAFS	2.1-17 keV	FB	BNL-NSLS BNL-Chemistry BNL-Electrochemistry ORNL University of Delaware UOP LLC Yeshiva University
X19C	Bend	XRD, surface X-ray topography X-ray reflectivity X-ray scattering, liquid X-ray scattering, surface	6-17 keV	PRT	Arizona State University Fairfield Crystal Technology, LLC Kansas State University Kyushu University SUNY @ Albany Stony Brook University University of Illinois @ Chicago
X20A	Bend	XRD, single crystal WAXD Microdiffraction Imaging X-ray reflectivity X-ray scattering, surface	4.5-13 keV	PRT	IBM Research Division
X20C	Bend	XRD, single crystal XRD, surface XRD, time resolved X-ray reflectivity X-ray scattering, surface	4-11 keV	PRT	IBM Research Division
X21	Insertion Device	XRD, single crystal X-ray scattering, magnetic X-ray scattering, resonant SAXS	5-15 keV	FB	BNL-NSLS Boston University University of Vermont

Beamline	Source	Type of Research	Energy Range	Type	Organization
X22A	Bend	XRD, single crystal XRD, surface WAXD X-ray reflectivity X-ray scattering, surface WAXS	10.7 keV 32 keV	PRT	BNL-Chemistry BNL-CMPMSD
X22B	Bend	X-ray scattering, liquid	6.5-10 keV	PRT	BNL-CMPMSD BNL-CFN
X22C	Bend	XRD, single crystal XRD, surface X-ray reflectivity X-ray scattering, magnetic X-ray scattering, surface	3-12 keV	PRT	BNL-CMPMSD Massachusetts Institute of Technology Rutgers University
X23A2	Bend	DAFS XAS EXAFS NEXAFS	4.7-30 keV	PRT	NIST
X23B	Bend	XRD, powder XAS EXAFS NEXAFS	4-10.5 keV	PRT	Hunter College NIST Naval Research Laboratory (NRL) Naval Surface Warfare Center New Jersey Institute of Technology Northeastern University Sarah Lawrence College
X24A	Bend	XSW Auger spectroscopy EXAFS X-ray fluorescence spectroscopy XPS	1.8-5 keV	PRT	NIST
X24C	Bend	X-ray reflectivity UV absorption spectroscopy XAS	0.006-1.8 keV	PRT	Naval Research Laboratory (NRL) Universities Space Research Association
X25	Insertion Device	MAD Macromolecular crystallography	5-20 keV	FB	BNL-NSLS BNL-Biology
X26A	Bend	Microdiffraction Imaging X-ray microprobe	3-30 keV	PRT	BNL-Enironmental Sciences University of Chicago University of Georgia
X26C	Bend	MAD Macromolecular crystallography	5-20 keV	PRT	BNL-Biology Cold Spring Harbor Laboratory Stony Brook University
X27A	Bend	X-ray microprobe	4.5-32 keV	FB	BNL-NSLS BNL-Enironmental Sciences Stony Brook University
X27B	Bend	HARMST	8-40 keV	PRT	BNL-Nonproliferation & National Security
X27C	Bend	XRD, time resolved WAXD SAXS WAXS	9 keV	PRT	Air Force Research Laboratory Dow Chemical Company National Institutes of Health Naval Surface Warfare Center Stony Brook University
X28C	Bend	X-ray Footprinting	White Beam	PRT	Case Western Reserve University
X29A	Insertion Device	MAD Macromolecular crystallography	6-15 keV	PRT	BNL-Biology Case Western Reserve University

Users' Executive Committee

The NSLS consults on a regularly scheduled basis with the Users' Executive Committee (UEC). This Committee provides a forum for ongoing, organized discussions between representatives from the NSLS user community in various research disciplines and the NSLS management, administration, and Brookhaven National Laboratory. The purpose is to communicate current and future needs, concerns, trends, and to disseminate information about NSLS and BNL future plans.

Chair	Daniel Fischer, *National Institute of Standards and Technology*
Vice Chair	John Parise, *Stony Brook University*
Past Chair	Chris Jacobsen, *Stony Brook University*
General Member	Peter Abbamonte, *University of Illinois @ Urbana-Champaign*
General Member	Jean Jordan-Sweet, *IBM*
General Member	Joe Dvorak, *Montana State University*
General Member	Howard Robinson, *BNL*
General Member	Peter Stephens, *Stony Brook University*
Ex-Officio	Chi-Chang Kao, *NSLS Chair, BNL*
Ex-Officio	Lisa Miller, *NSLS Information and Outreach Coordinator, BNL*
Ex-Officio	Kathleen Nasta, *NSLS User Administrator, BNL*

Special Interest Group Representatives

Special Interest Group (SpIG) Representatives are listed below; these groups in areas of common concern communicate with NSLS management through the UEC.

Bio. Crystallography & Diffraction	Vivian Stojanoff, *BNL/NSLS*
High Pressure	Baosheng Li, *Stony Brook University*
Imaging	Zhong Zhong, *BNL/NSLS*
Industrial Users	Simon Bare, *UOP LLC*
Infrared Users	Liping Wang, *Stony Brook University*
Magnetism	Cecilia Sanchez-Hanke, *BNL/NSLS*
NSLS-II	John Parise, *Stony Brook University*
Nuclear Physics	Mahbub Khandaker, *Thomas Jefferson National Laboratory*
Students & Post Docs	Abdel Isakovic, *BNL/NSLS*
Time Resolved Spectroscopy	John Sutherland, *BNL/Biology*
Topography	Michael Dudley, *Stony Brook University*
UV Photoemission & Surface Science	Jeff Keister, *SFA, Inc.*
XAFS	Paul Northrup, *BNL/Environmental Sciences*
X-ray Scattering & Crystallography	Peter Stephens, *Stony Brook University*

Scientific Advisory Committee

The NSLS Scientific Advisory Committee (SAC) advises the NSLS Chair and the Associate Laboratory Director for Light Sources on scientific, technical, and policy issues related to the optimization of the scientific productivity of the NSLS.

Mario Amzel, *Johns Hopkins University*
Simon Bare, *UOP, LLC*
Joel Brock, *Cornell University*
Tony Heinz, *Columbia University*
Robert Hettel, *Stanford Synchrotron Radiation Laboratory/ Stanford Linear Accelerator Center*
Eric Isaacs, *Argonne National Laboratory*
Leemor Joshua-Tor, *Cold Spring Harbor Laboratory*
James Kaduk, *INEOS Technologies*
Edward Kramer, *University of California, Santa Barbara*
Simon Mochrie, *Yale University*
James Penner-Hahn (Chair), *University of Michigan*
William Thomlinson, *Canadian Light Source*
Daniel Fischer (UEC Chair), *National Institute of Standards and Technology*

Proposal Review Panels

As part of the peer-review process of GU proposals, the Proposal Review Panels (PRPs) at the NSLS review and rate the proposals within their scientific area. Members are drawn from the scientific community and usually serve a two-year term. Each PRP has an appointed Chair, who is part of the Proposal Oversight Panel (POP), established to review any proposals that might need special attention due to use of multiple techniques or conflicts in scores. The POP has additional appointed members, listed below after the PRP membership.

* = Chair of PRP

Imaging and Microprobes: Biological and Medical

Leroy Chapman, *University of Saskatchewan*
Max Diem, *City University of New York*
Paul Dumas, *Centre National de La Recherche Scientifique*
Kathleen Gough, *University of Manitoba*
Barry Lai*, *Argonne National Laboratory*
Carolyn Larabell, *Lawrence Berkeley National Laboratory*
Irit Sagi, *Weizmann Institute of Science*
Stefan Vogt, *Argonne National Laboratory*

Imaging and Microprobes: Chemical and Materials Sciences

Harald Ade*, *North Carolina State University*
David Black, *National Institute of Standards and Technology*
Paul Evans, *University of Wisconsin*
Steve Heald, *Argonne National Laboratory*
Gene Ice, *Oak Ridge National Laboratory*
Barry Lai, *Argonne National Laboratory*
Ismail Noyan, *Columbia University*
Qun Shen, *Argonne National Laboratory*
Nobumichi Tamura, *Lawrence Berkeley National Laboratory*

Imaging and Microprobes: Environmental and Geosciences

Don Baker, *McGill University*
David Black, *National Institute of Standards and Technology*
Jeffrey Fitts, *BNL*
George Flynn, *SUNY at Plattsburgh*
Matthew Ginder-Vogel, *University of Delaware*
Keith Jones*, *BNL*
Lindsay Keller, *NASA Johnson Space Center*
Kenneth Kemner, *Argonne National Laboratory*
Mark Rivers, *University of Chicago*
Donald Ross, *University of Vermont*
David Wetzel, *Kansas State University*

IR/UV/Soft X-ray Spectroscopy: Chemical Sciences/Soft Matter/Biophysics

Jingguang Chen, *University of Delaware*
Daniel Fischer*, *National Institute of Standards and Technology*
Jan Genzer, *North Carolina State University*
Robert Lodder, *University of Kentucky*
Gary Mitchell, *The Dow Chemical Company*
David Mullins, *Oak Ridge National Laboratory*
Michael White, *BNL*

IR/UV/Soft X-ray Spectroscopy: Magnetism/Strongly Correlated Electrons/Surface

Robert Bartynski*, *Rutgers University*
Hong Ding, *Boston College*
Di-Jing Huang, *Synchroton Radiation Research Center*
Jeffrey Keister, *SFA, Inc.*
Alexander Moewes, *University of Saskatchewan*
David Mullins, *Oak Ridge National Laboratory*
Boris Sinkovic, *University of Connecticut*
Jiufeng Tu, *City University of New York*
Tonica Valla, *BNL*
Barrett Wells, *University of Connecticut*

Methods and Instrumentation

Leroy Chapman, *University of Saskatchewan*
Kenneth Finkelstein, *Cornell University*
Jeffrey Keister, *SFA, Inc.*
Albert Macrander, *Argonne National Laboratory*
Ralf-Hendrik Menk, *Sincrotrone Trieste*
Peter Takacs*, *BNL*

Macromolecular Crystallography

Alex Bohm*, *Tufts University*
Brian Crane, *Cornell University*
Dax Fu, *BNL*
Xiangpeng Kong, *New York University*
Daniel Leahy, *John Hopkins University*
Nicolas Nassar, *Stony Brook University*
Brenda Schulman, *St. Jude Children's Research*
Peter Stephens, *Stony Brook University*
DaNeng Wang, *New York School of Medicine*
Joshua Warren, *Duke University*
Hao Wu, *Cornell University*

Powder/Single Crystal Crystallography

Simon Billinge, *Columbia University*
Andrew Campbell, *University of Maryland*
Thomas Duffy*, *Princeton University*
Andrew Fitch, *European Synchrotron Radiation Facility*
Joseph Hriljac, *University of Birmingham*
Stefan Kycia, *University of Guelph*
Peter Lee, *Argonne National Laboratory*
James Martin, *North Carolina State University*
Wendy Panero, *Ohio State University*
Patrick Woodward, *Ohio State University*

X-Ray Scattering: Magnetism/Strongly Correlated Electrons/Surface

Sean Brennan, *Stanford Linear Accelerator Center*
Kenneth Finkelstein*, *Cornell University*
Peter Hatton, *University of Durham*
Valery Kiryukhin, *Rutgers University*
Karl Ludwig, *Boston University*
George Srajer, *Argonne National Laboratory*
Trevor Tyson, *New Jersey Institute of Technology*

X-Ray Scattering: Soft Matter and Biophysics

Masafumi Fukuto, *BNL*
Randall Headrick, *University of Vermont*
Paul Heiney, *University of Pennsylvania*
Ben Hsiao*, *Stony Brook University*
Huey Huang, *Rice University*
Robert Leheny, *John Hopkins University*
H. Miriam Rafailovich, *Stony Brook University*
Thomas Russell, *University of Massachusetts*
Detlef Smilgies, *Cornell University*
Helmet Strey, *Stony Brook University*
Lei Zhu, *University of Connecticut*

X-Ray Spectroscopy: Biological, Environmental and Geosciences

Suzanne Beauchemin, *Natural Resources Canada*
Martine Duff, *Westinghouse Savannah River Company*
Dean Hesterberg, *North Carolina State University*
Douglas Hunter, *University of Georgia*
Kenneth Kemner, *Argonne National Laboratory*
Satish Myneni*, *Princeton University*
Matthew Newville, *University of Chicago*
James Penner-Hahn, *University of Michigan*

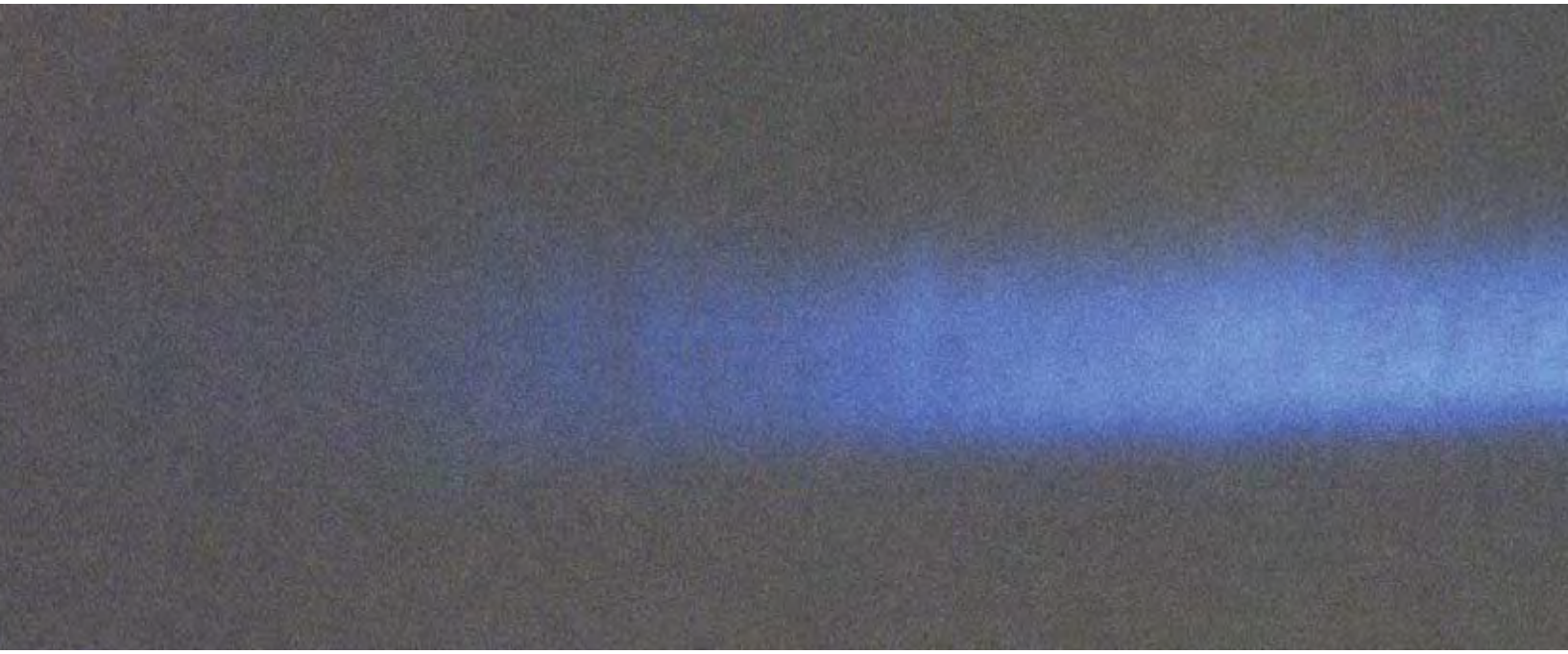
X-Ray Spectroscopy: Chemical and Material Sciences

Simon Bare, *UOP LLC*
Uwe Bergmann, *Stanford Linear Accelerator Center*
Scott Calvin, *Sarah Lawrence College*
Anatoly Frenkel, *Yeshiva University*
Steven Heald, *Argonne National Laboratory*
Bruce Ravel, *Argonne National Laboratory*
Jean-Pascal Rueff, *Université Paris VI*
Tsun Sham, *University of Western Ontario*
Trevor Tyson*, *New Jersey Institute of Technology*

Proposal Oversight Panel Members

Antonio Lanzirotti, *University of Chicago*
Jonathan Hanson, *BNL*
Stephen Shapiro, *BNL*
Peter Stephens, *Stony Brook University*
Subramanyam Swaminathan, *BNL*

In addition to a PDF version of the 2007 Activity Report, this CD contains almost 60 science highlights from the year and a complete listing of staff and user publications.



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