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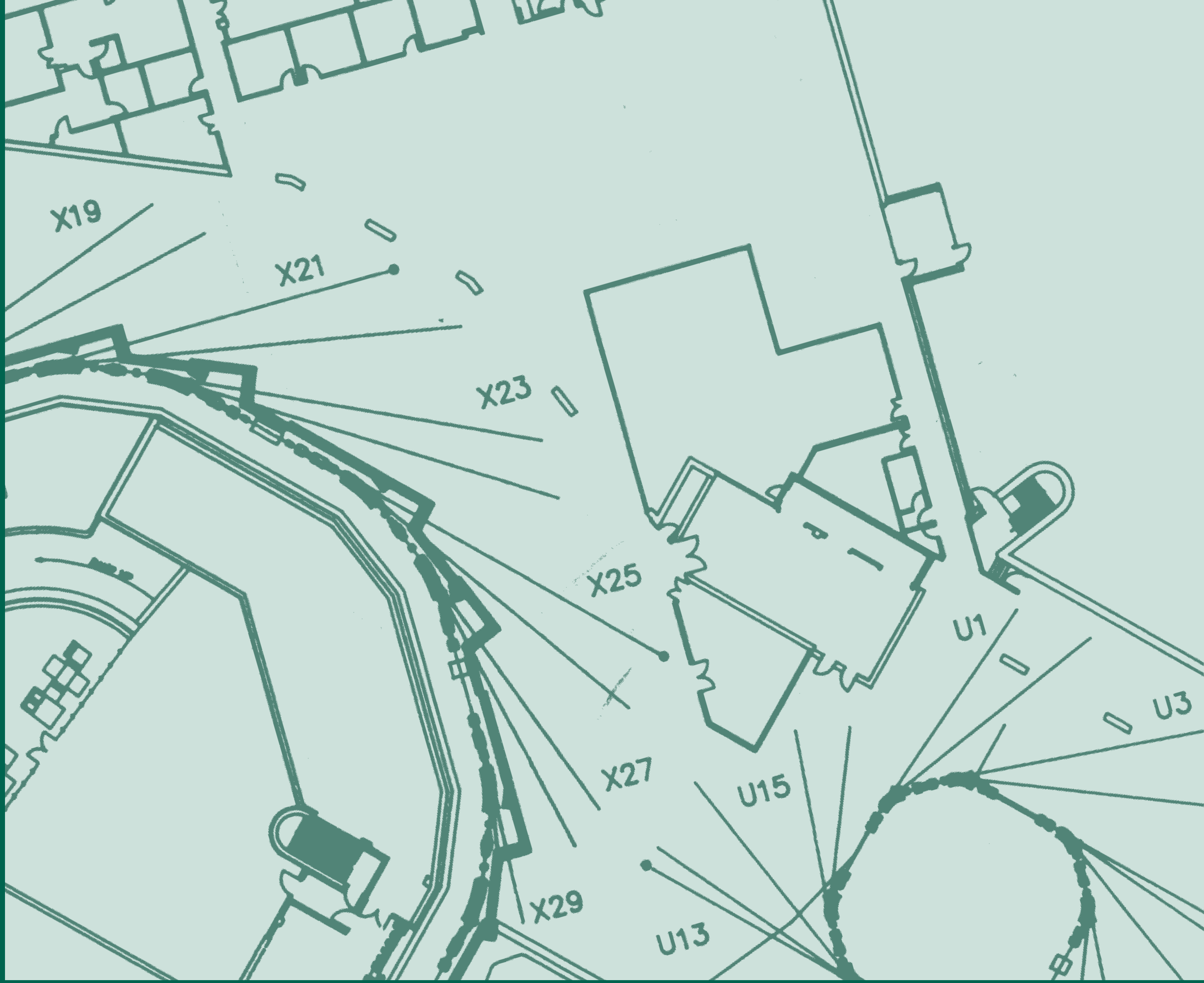
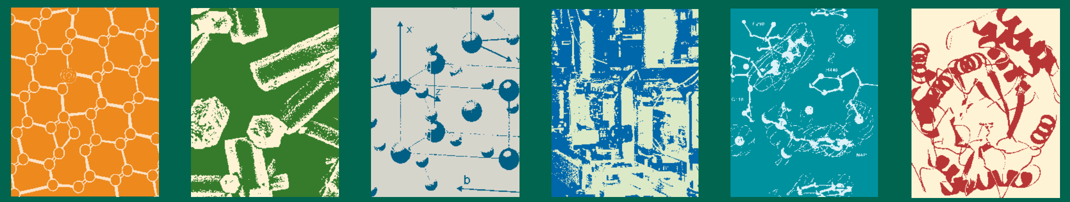
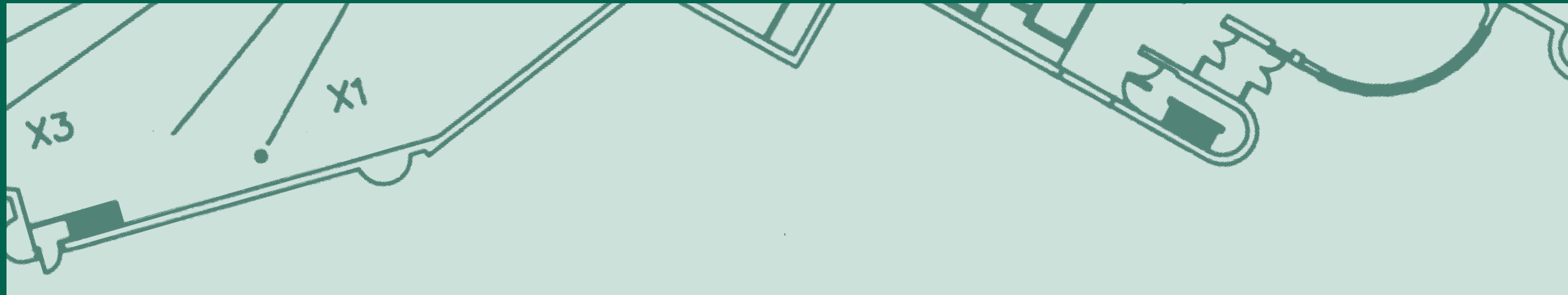
BNL 71045 National Synchrotron Light Source Activity Report 2002





National Synchrotron Light Source
Activity Report 2002



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National Synchrotron Light Source

Activity Report

For the period October 1, 2001 through September 30, 2002

May 2003

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published on the web at http://nslsweb.nsls.bnl.gov/nsls/pubs/actrpt/2002/Abstracts/

Introduction

Chairman's Introduction

Steven Dierker

Chairman, National Synchrotron Light Source

The year 2002 has been another highly productive year at the NSLS and an impressive array of highlights from this scientific activity is included in this Activity Report. We have taken significant steps this past year toward better supporting beamlines and users. The number of user science support staff has been increased by about ten positions. We have also worked with our users, DOE, and the other DOE synchrotron facilities to develop a new, more flexible user access policy. Doing things safely remains a top priority, and we are reviewing our training and safety requirements to ensure they are thorough and everyone fully understands the necessity of abiding by them.

A major development this past year was approval from DOE for BNL to begin the conceptual design of the Center for Functional Nanomaterials (CFN). The CFN will have a dramatic impact on nanoscience in the Northeast, facilitating the synthesis, characterization and scientific exploration of new classes of novel nanostructured materials. It will be located adjacent to the NSLS and a number of NSLS beamlines will be optimized to serve the needs of the nanoscience community. The NSLS and CFN user programs will be coordinated to facilitate easy access to both in a single visit.

The VUV and X-Ray rings operated with excellent reliability as a result of continued attention to aging critical systems. I congratulate the staff in the Operations and Engineering Division and extend them special thanks for their rapid response to a few potentially disruptive equipment failures.

The DUV-FEL achieved several important milestones this year, including production of Self-Amplified Spontaneous Emission (SASE) laser light at 400 nm and 266 nm, laser seeded saturation at 266 nm, and the first observation of High Gain Harmonic Generation (HGHG) light at 266 nm, with a third harmonic at 89 nm. Light from the DUV-FEL is now enabling user science experiments in ion pair imaging and we look forward to an expanding user program and a continued series of pioneering accelerator physics studies.

In 2002, we continued to work with our user community to develop a plan to upgrade the capabilities of the NSLS. In the short term, we have a number of projects ongoing to upgrade insertion devices and beamlines to provide new and enhanced capabilities to our users. In the long term, recognizing the importance of meeting the future needs of our user community, we are working with DOE to formulate a plan for construction of a new state-of-the-art third-generation storage ring at NSLS to provide world class capabilities to our large and outstandingly productive user community.

Overall, it has been a very positive year at the NSLS and we look forward to an exciting year ahead.

Users' Executive Committee and NSLS Town Meetings

Leemor Joshua-Tor

Users' Executive Committee Chair, Cold Spring Harbor Laboratory

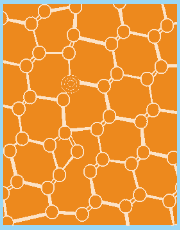
It was an exciting time at the NSLS during 2002. The announcement of the new Center for Functional Nanomaterials (CFN), which will be built adjacent to the NSLS, will undoubtedly invigorate the science at the facility. But even more exciting is the proposal for a major NSLS upgrade in the not too distant future. The Users' Executive Committee (UEC), as well as many leaders of the user community, has been providing comments and suggestions for what we would like to see in what would be a valuable resource for many different and new areas of science. This proposed upgrade would play an important and critical role, especially in the very strong scientific communities of the Northeast and Mid-Atlantic regions of the United States.

However, it is also a challenging time for the NSLS. Security regulations have been tightening up, as I'm sure most of you have felt. The directors of the four Department of Energy (DOE) synchrotron facilities, in coordination with the Office of Basic Energy Sciences (BES), have drafted a new user access policy statement that will change the structure of some Participating Research Teams (PRTs) and beamline support. A much more detailed policy for the NSLS is currently under development. In addition, a Proposal and Safety System (PASS), is under development by the User Administration office, which will replace the current systems for general user proposals and safety reviews and will reduce the lead-time between proposal submission and scheduling cycles significantly. My colleagues on the UEC, along with very conscientious members of the user community, have been actively involved in all of these projects to make sure they evolve with you, the user, as the main focal point. I'd like to acknowledge them for their very hard work and enthusiasm. As always, we welcome your thoughts on any of these issues and we will convey them accordingly.

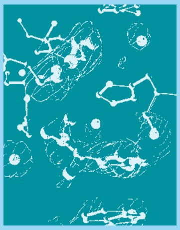
These are also challenging times in terms of the budgets allocated to science in the United States. The UEC, along with the users organizations of the other DOE-run synchrotron facilities, is vigorously continuing its lobbying efforts to increase the Office of Science's budget, which funds the operations of these synchrotrons, and thus would increase funding for the light sources. With the current problems in the economy and the increasing uncertainties for the future, it is vital that we keep up our mission and impress upon our lawmakers how crucial our research is for the future of science, technology and the economy.

Most importantly, the UEC, along with NSLS management, is reviewing ways to improve the overall safety record at the NSLS. The increased number of inexperienced users, who may only spend a few days at the facility, together with perceived lack of attention to safety guidelines by users, calls for a change in the overall culture and working habits at the NSLS. We, as users, have always appreciated the efforts of NSLS staff to place our experiments as the first priority and make the safety procedures as efficient as possible. However, this requires us, the users, to have respect for possible hazards to ourselves as well as to our fellow experimentalists on the floor and be less cynical towards these safety procedures.

Lastly, the UEC has a new website: www.nslsuec.org. Please visit our website for detailed information on our activities. Finally, the UEC wishes you a SAFE and FRUITFUL year!





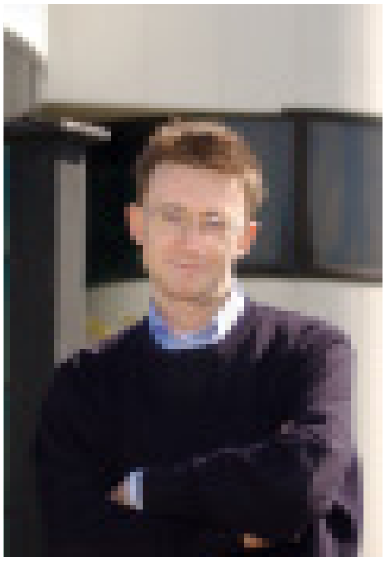



Science Highlights

Science at the NSLS

Patrice Pages

Science Writer, NSLS



The following pages contain a representation of the outstanding science published by NSLS users in 2002. Due to space limitations, we have chosen to highlight only a portion of the scientific accomplishments from last year, but we think that these articles illustrate the quality and breadth of NSLS research.

The following stories contain a wealth of information in a wide range of scientific fields and provide an update on important scientific topics. For example, you will discover how bacteria resist antibiotics (page 2-71) or how a certain type of protein is involved in cancer (page 2-62). You will read about new ways to immobilize contaminants in soil (page 2-47), and you will learn how scientists have created "light tweezers" to study the electronic properties of solids (page 2-28).

The stories are organized into two categories: "feature" stories, which are geared not only to the NSLS user community but also to a more general audience, and "highlights", which are more specifically aimed at the scientific community. I have particularly enjoyed working with the NSLS users on these stories, and I would like to thank the scientists whom I interviewed to write the feature stories and those who authored the highlight stories and patiently allowed me to edit them.

I hope that you will share with me the excitement generated by the following stories, and I will be delighted to hear your impressions about them.

Science Highlights

Accelerator Physics

Chemistry

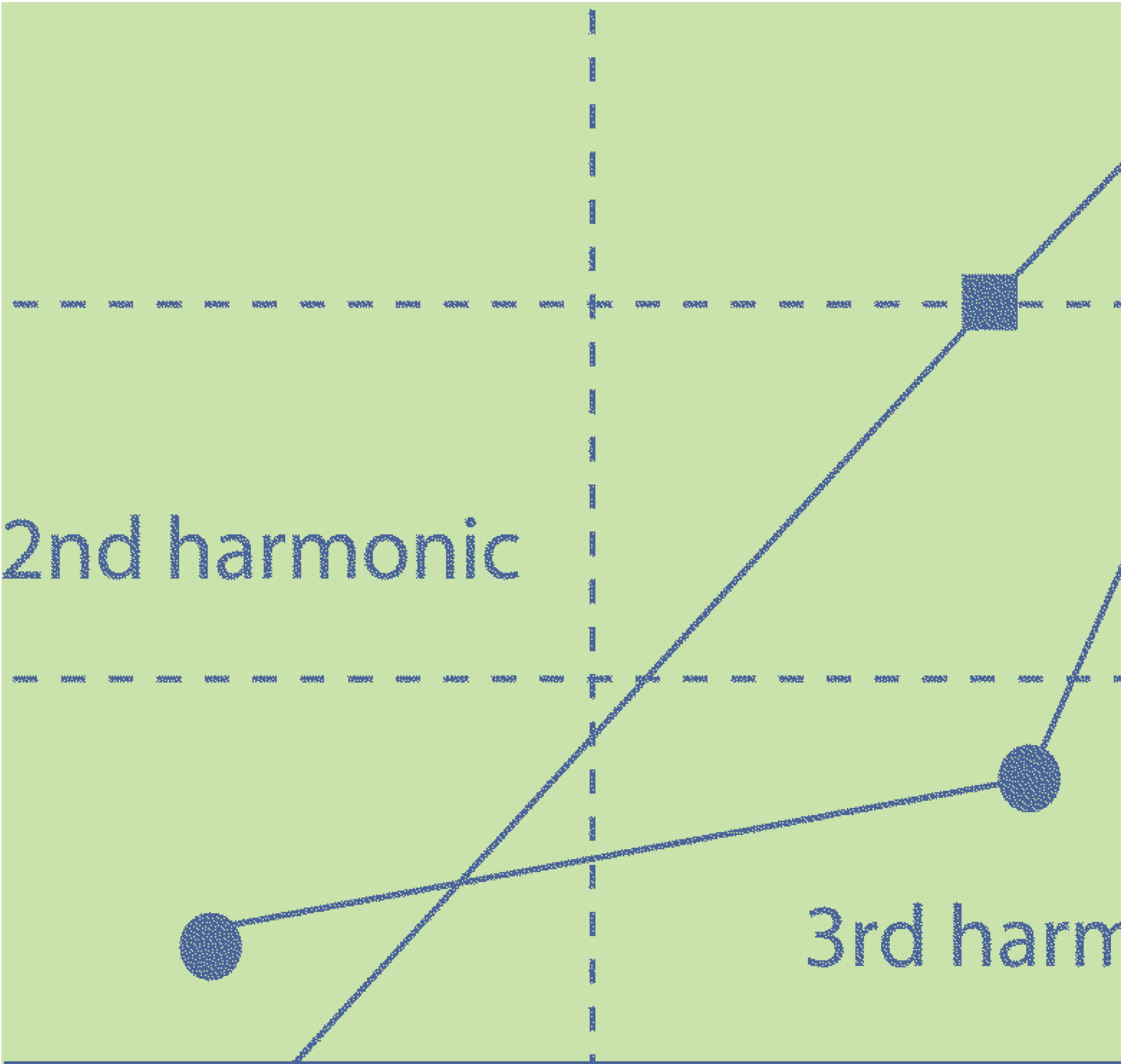
Condensed Matter Physics

Condensed Matter Physics (cont.)

Geology and Environmental Sciences

Life Sciences

Surfaces, Interfaces, and Nanomaterials



Accelerator Physics

Blue Light Special at the DUV-FEL

February 13, 2002 - A specially intense blue light shone for the first time at BNL's Deep Ultra-Violet Free Electron Laser (DUV-FEL). On hand to see this 400-nanometer (billionths of a meter) wavelength light were NSLS scientists Adnan Doyuran, Bill Graves, Henrik Loos, Timur Shaftan, Brian Sheehy, and Li-Hua Yu, with technical specialist Phil Marino. It was a satisfying and visible reward for the hard work of many.

"Achieving this 400-nanometer-wavelength light is an important milestone in the development of the DUV-FEL," says Erik Johnson, the NSLS's DUV-FEL Project Manager. "It paves the way to producing even shorter and more intense 100-nanometer-wavelength laser light."

Visible light's wavelength ranges from 400 to 700 nanometers (nm). When it produces the more energetic 100 nm light, the DUV-FEL will become an important tool for studying chemical reactions.

The commissioning experiment, led by Yu, achieved the visible 400-nm light by Self Amplified Spontaneous Emission (SASE).

The experiment demonstrated that the components of the machine work successfully and have been appropriately tuned. The scientists also found that the light intensity was about 600 times higher than anticipated.

"We were pleasantly surprised by how intense the output light was," says Johnson. At least two factors contributed to this high output and its comparatively rapid attainment, he explains: The high quality of the beam generated by the linear accelerator, and the beam alignment, which allows rapid and precise correction of the beam trajectory.

"However, light produced by the SASE process is just the starting point for the DUV-FEL program," Johnson says.

The project has its roots in earlier work. In 1999, in what was an important milestone for the field, the High Gain Harmonic Generation (HGHG) process in an FEL was tested for the first time by a team of scientists led by Yu.

This experiment at BNL's Accelerator Test Facility used a 10 micrometer-wavelength infrared laser to provide a coherent starting signal for the lasing process. Some of the same scientists involved in the successful HGHG experiment are now planning to extend that work to wavelengths down to 100 nanometers or fewer at the DUV-FEL.

BNL's DUV-FEL will complement the NSLS in many ways. The DUV-FEL's pulses of light are up to 1,000 times shorter than those produced at the NSLS, enabling the study of time dynamics of molecules and chemical reactions. DUV-FEL light is also coherent, meaning that the particles of light, or photons, move like soldiers following marching orders, in contrast to incoherent light, in which the photons follow their own pace.

The FEL's coherent light will have a peak intensity more than a billion times higher than that of the NSLS.

"The high power and short timescale of FEL light is expected to open entirely new fields of research in much the same way that the NSLS rings did 20 years ago," says NSLS physicist William Graves, who led the electron beam development effort at the DUV-FEL. "The NSLS will continue to be invaluable to a broad user community, while the FEL will provide en-

While many people were involved in the development and installation of the DUV-FEL machine, a smaller core of individuals carried out the commissioning and initial SASE experiment. Among them: (from left) Richard Heese, Jim Rose, Phil Marino, Timur Shaftan, Li-Hua Yu, (seated) Boyzie Singh, William Graves, Adnan Doyuran, Henrik Loos, and Brian Sheehy. (Not in photo, Joe Greco, Erik Johnson.)

hanced capabilities for some specialized experiments."

The DUV-FEL is one of just a handful of FEL's in the world designed to operate in the ultraviolet, and it is the only facility designed to generate light through the HGHG process.

"It is also an important resource for the FEL research community right now," Graves says, "because most of the other similar FEL programs are shut down for upgrades or are under construction. For a period of about a year, we are the only game in town for doing this kind of work, which will increase our collaboration with scientists from DESY [German Synchrotron Laboratory] and SLAC [Stanford Linear Accelerator Center]."

According to Johnson, the prospects for the facility are bright. "Production of light at wavelengths below 400 nm is planned in the coming year, experimental use of the laser for chemical physics experiments should begin, and it will continue to provide a platform for exploring upgrade paths for the NSLS scientific program.

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - April 12, 2002.]

Learn About the SASE and HGHG Processes

BNL's Deep Ultra-Violet Free Electron Laser uses fast-moving electrons traveling at close to the speed of light. They are produced in a photo-injector and brought to their high energy by a linear accelerator. They then pass through an undulating magnetic field in a "wiggler" device that forces them to wiggle, and thus to emit light.

SASE

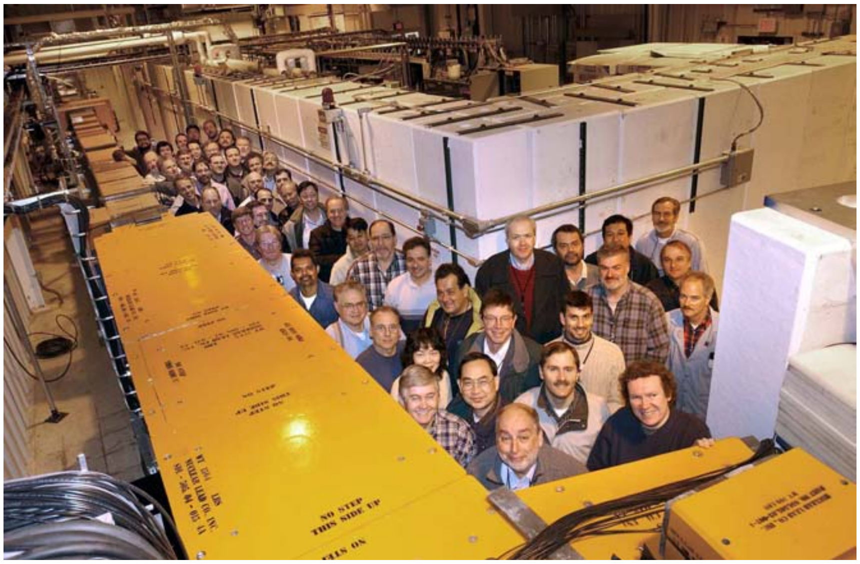
In the Self Amplified Spontaneous Emission (SASE) process, the FEL's output light gets its start from "noise," or random signals, that naturally happen in the wiggler.

HGHG

In the High Gain Harmonic Generation (HGHG) process, the FEL's output light starts from the fast-moving electrons interacting with a laser called the "seeding" laser. The conditions are set so that the output light has a frequency that is a harmonic, or a multiple, of the frequency of the input laser light.

Similarly to the way one can strike a key on a piano and set the strings of higher octaves in motion, the HGHG process shifts the wavelength of the seeding laser to a higher frequency, generating a wavelength for the output light, the HGHG light, that is beyond the range of the original laser.

In addition, the coherence, or orderliness, of the seeding laser is imparted to the output DUV-FEL light, providing a light beam that is significantly more coherent than light produced from SASE sources.



Many of the National Synchrotron Light Source's Deep Ultra-Violet Free Electron Laser team are gathered in the Source Development Laboratory, celebrating the successful effort to achieve a 400-nanometer wavelength beam of light.

Deep Ultraviolet Free Electron Laser Reaches New Milestone Using BNL's Unique HGHG Process

November 1, 2002 - After three years of intense effort in developing a new source of laser light called a deep ultra-violet free electron laser (DUV-FEL), BNL scientists generated very intense ultraviolet light by a process called high gain harmonic generation (HGHG). This process, unique to BNL, was developed under the direction of Li-Hua Yu, a physicist at the NSLS. HGHG will ultimately generate deep ultraviolet laser light with unsurpassed brightness.

"Such intense light," Yu says," will be a powerful new tool that will reveal fine details of atomic interactions inside materials and the very fast motions of molecules in chemical reactions all this with a precision unequaled so far."

By using the HGHG process, the DUV-FEL seeks to achieve very short wavelengths of light. While visible light's wavelengths range from 400 nanometers (nm) for blue light to 700 nm for red light - a nanometer being a billionth of a meter - the wavelength of the light produced at the DUV-FEL last week was 266 nm. The HGHG process also generated significant deep ultraviolet light at 88 nm.

The advantages of two current light sources will be combined in the DUV-FEL. By generating very intense deep ultraviolet light, the DUV-FEL will act like a synchrotron, which delivers short wavelengths but less intense light, and a laser, which produces very intense light, but only at longer wavelengths.

Over the last three years, the DUV-FEL has been advancing through many steps, each step representing a new technological challenge. The many NSLS scientists, engineers, and technicians who made key contributions to overcome these challenges include physicists William Graves, Erik Johnson, Samuel Krinsky, Timur Shaftan, and Brian Sheehy; postdoctoral research associates Adnan Doyuran and Henrik Loos, mechanical engineer John Skaritka, and radio frequency engineer Jim Rose. The experiment also benefited greatly from the scientific contributions of Ilan Ben-Zvi, Richard Heese, George Rakowsky, Xijie Wang, and Zilu Wu; the computer control of Kate Berman; and the technical assistance of Joseph Greco, Dave Harder, Michael Lehecka, Phil Marino and Boyzie Singh.

"In addition," says Yu, "valuable contributions were made by other scientists in past stages of the DUV-FEL. The team spirit of all who have worked on the project helped in achieving the present milestone."

Yu explains that the DUV-FEL can produce such intense light because of a property called "coherence." In both a synchrotron and an FEL, electrons are accelerated to near the speed of light, and then are forced to emit very intense light by going through magnets that bend their trajectories. But while light is emitted at random, or "incoherently," in a synchrotron, it is produced "coherently," in the DUV-FEL, leading to a dramatic increase in intensity, which can be up to 10 million times as intense as current synchrotron light.

"It is as if, instead of having a group of people singing the same song at different times, they sang in unison," says Shaftan.

Interim Lab Director Peter Paul (right) celebrates the latest milestone at BNL's DUV-FEL with National Synchrotron Light Source (NSLS) Department members (foreground, from left) Li Hua Yu, James Murphy, Adnan Doyuran, NSLS Chair Steve Dierker; (back, from left) Timur Shaftan, John Skaritka, Boyzie Singh, Erik Johnson, Henrik Loos, and Brian Sheehy.

In the DUV-FEL, the electrons can "sing in unison" in two different ways, called self-amplified spontaneous emission (SASE) and HGHG. In the first process, electrons interact with light emitted by their fellow electrons, creating small groups of electrons. "Inside each group, all the electrons sing in unison, but the songs between two groups are out of sync," Shaftan says.

In the HGHG process, the electrons interact with light provided by a laser at the entrance of the DUV-FEL, a process leading to groups of electrons, as before, but "this time, not only do you have a united choir in each group, but all groups sing together," Loos says. "This 'super-choir' of electrons generates light that is many times more intense than the one generated by the SASE process."

While many FELs are currently under development throughout the world, they are all based on the SASE process. Brookhaven's DUV-FEL is the only FEL using the HGHG process, which will allow it to generate the brightest deep ultraviolet light so far. Unlike SASE, HGHG also generates light at wavelengths that are fractions (one-half, one-third, one-quarter, etc.) of the input wavelength.

"Such harmonics can be generated by conventional lasers when they cross a given crystal or gas," Doyuran says. "But the intensity of these harmonics is much weaker than the input light. The big advantage of HGHG is that the harmonics are much more intense than those produced with a conventional laser."

To benefit from this very intense light, three BNL scientists from the Chemistry Department have already submitted proposals to use the DUV-FEL.

Arthur Suits is interested in using light-induced dissociation of atoms in hydrocarbons to reveal details of their molecular structure and bonding. "The DUV-FEL is really ideal for our experiments," he says. "The information we hope to gain using it would be impossible to obtain by other means."

Louis DiMauro plans to use the DUV-FEL's short and highly intense light pulses to study the interaction of matter with intense, short-wavelength light. "This tool will open up many new areas of research in fundamental and applied science," he says.

Michael White intends to use the DUV-FEL to study chemical reactions that occur on the surfaces of materials, for which the product yields are low or where the gaseous products are particularly difficult to detect.

Says White, "In the long term, the DUV-FEL will produce very short pulses that will provide new information on surface processes in real time."

For Yu and his collaborators, these proposals are a clear sign that the efforts of the past three years are coming to fruition.

"The scientific use of the DUV-FEL will be the best reward for all of us and the crowning of a lot of hard work and dedication from the scientists, engineers and technicians who worked on this project," says James Murphy, NSLS Associate Chairman for Accelerators.

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - November 8, 2002.]

The National Synchrotron Light Source Department team working on BNL's deep ultraviolet free electron laser include: (front, from left) Timur Shaftan, Richard Heese, Boyzie Singh, Adnan Doyuran, Phil Marino, and Li Hua Yu; (middle, from left) Joe Greco, James Murphy, Xijie Wang, Bill Bambina, Sorin Pop, Brian Sheehy, Zilu Wu, and James Rose; (back, from left) Henrik Loos, Erik Johnson, and John Skaritka.

Very Intense, Terahertz Light Created for the First Time

Scientists report in the November 14, 2002 issue of *Nature* that they have created a beam of radiation that has never been produced before. The beam, which contains a broad spectrum of frequencies up to about one terahertz - or a trillion of cycles per second - and a brightness 20,000 times higher than that of previous terahertz beams, was produced at the U.S. Department of Energy's Thomas Jefferson National Accelerator Facility in Newport News, Virginia.

"One of the anticipated applications for this terahertz radiation is imaging," says Larry Carr, a physicist at the NSLS, and lead author of the study. "Terahertz radiation can penetrate many centimeters inside materials, allowing one to 'see' what's inside."

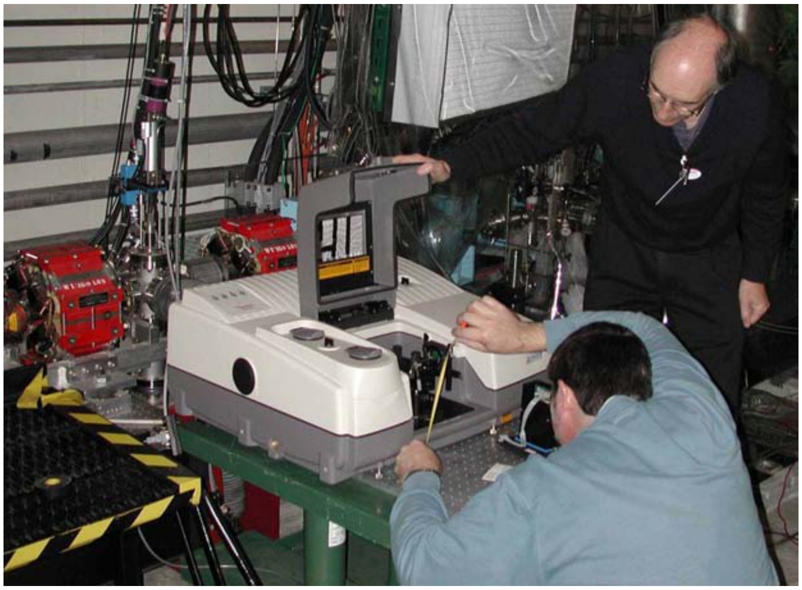
Other potential applications of the newly created terahertz radiation include better detection of concealed weapons, hidden explosives and land mines; "fingerprinting" of chemical and biological terror materials in envelopes, packages or air; and widening the frequency bands available for wireless communication.

The powerful terahertz radiation was produced in an experiment conceived and led by Gwyn Williams, physicist at Jefferson Lab. He and his collaborators, from Brookhaven and Lawrence Berkeley National Laboratories, sent a beam of electrons at nearly the speed of light inside an accelerator, in which they were deflected by a magnetic field, causing them to emit radiation. Inside the accelerator, the electrons were squeezed into small bunches (less than 1 millimeter long), inside which they emitted radiation in unison.

"Producing intense light pulses by this method dates back to more than 10 years ago," Carr says, "but the pulse rate was limited to just a few times each second. Now, we can accelerate 75 million electron bunches each second, which creates a stream of terahertz pulses delivering the unprecedented average power of 20 watts."

As with any new technology, the most important applications of terahertz radiation are not known so far, but the authors of the study are confident that it can potentially extend and add to the wave-based technologies that have defined the last century and a half. "The growing awareness of the usefulness of terahertz radiation is like what happened a century ago with X-rays - only terahertz radiation will have a much wider range of applications," Williams says.

-Patrice Pages



NSLS Physicist Larry Carr (left) and Jefferson Lab Physicist Gwyn Williams working on the experiment that recently produced very powerful terahertz radiation.

BEAMLINE
ATF3

PUBLICATION
A. Tremaine *et al.* "Experimental Characterization of Nonlinear Harmonic Radiation from a Visible SASE FEL at Saturation", *Phys. Rev. Lett.*, **88**, 204801 (2002).

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Nonlinear Harmonic Radiation from a Visible Self-Amplified Spontaneous Emission Free Electron Laser

A. Tremaine[1], A. Murokh[1], C. Pellegrini[1], S. Reiche[1], J. Rosenzweig[1], M. Babzien[2], I. Ben-Zvi[2], R. Malone[2], G. Rakowsky[2], J. Skaritka[2], X. Wang[2], V. Yakamenko[2], R. Carr[3], M. Cornacchia[3], H.-D. Nuhn[3], R. Ruland[3], L. Bertolini[4], M. Libkind[4], A. Toor[4], and K. Van Bibber[4]

[1]University of California, Los Angeles; [2]Brookhaven National Laboratory; [3]Stanford Linear Accelerator Center; [4]Lawrence Livermore National Laboratory

Scientists from the University of California, Los Angeles, Brookhaven National Laboratory, Stanford Linear Accelerator Center and Lawrence Livermore National Laboratory have demonstrated for the first time the feasibility of using nonlinear harmonic self-amplified spontaneous emission (SASE) free electron laser (FEL) radiation to produce coherent, femtosecond x-rays. Nonlinear harmonic radiation (NHR) was observed using the visible-to-infrared SASE amplifier (VISA) FEL at saturation.

The growth of synchrotron light source applications in the last few decades is due largely to the improved brightness of the x-ray beams they produce. During this same period, high power, femtosecond-laser technologies have opened new frontiers in spectroscopy and dynamics system studies. The free electron laser (FEL), which produces coherent, femtosecond x-rays, can bridge these technologies. Due to the lack of x-ray seeds and mirrors, all proposed x-ray FELs are based on self-amplified spontaneous emission (SASE), which is a high-gain, single-pass FEL amplifier.

We report here the results from a light source called visible-to-infrared SASE amplifier (VISA) FEL, which served as research and development for the linear accelerator (linac) coherent light source (LCLS), a 1-angstrom (Å) FEL to be built at the Stanford Linear Accelerator Center (SLAC) in California. VISA exemplifies that future facilities can use SASE nonlinear harmonic radiation (NHR) to produce narrower bandwidth and harder x-rays compared to the fundamental radiation.

A major challenge for the VISA collaboration was to implement the technologies necessary to reduce the size and cost of FELs, which could be considerable for future devices. Using the high-brightness beam generated by beamline 3 of Brookhaven National Laboratory's Accelerator Test Facility, and a novel magnetic undulator built by SLAC, VISA demonstrated the shortest visible gain length to date with fundamental saturation after a distance of only 3.8 meters.


Aaron Tremaine

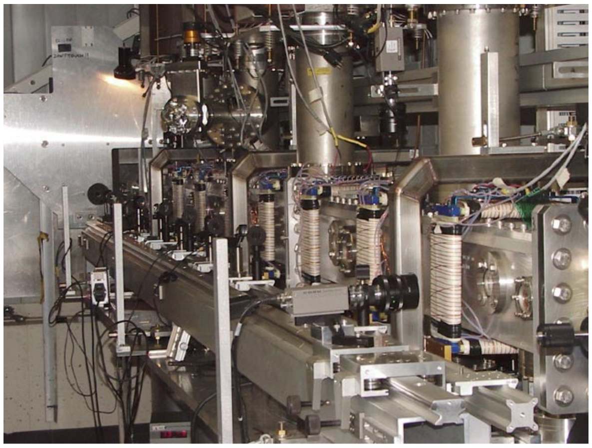
NHR accompanies the fundamental radiation only in the SASE high gain regime. **Figure 1** shows the superimposed VISA spectrum at the end of the undulator for the three lowest FEL modes. The fundamental spectrum is centered at 845 nanometers (nm), and, as expected, the second and third nonlinear harmonics are centered at 422 and 280 nm, respectively.

Gain lengths for each mode are calculated from the data shown in the log-linear plot of **Figure 2**. A fundamental gain length of L_g =19 centimeters (cm) is measured, and NHR gain lengths of 9.8 cm and 6 cm are obtained for the second and third NHR, respectively, using only the data in the nonlinear regime. The NHR grows faster than the fundamental by $L_{g,n} = L_{g,1} / n$, verifying theoretical predictions.

By the undulator exit, the energies of the second and third NHRs are two percent and one percent of the fundamental energy, respectively, confirming theoretical predictions.

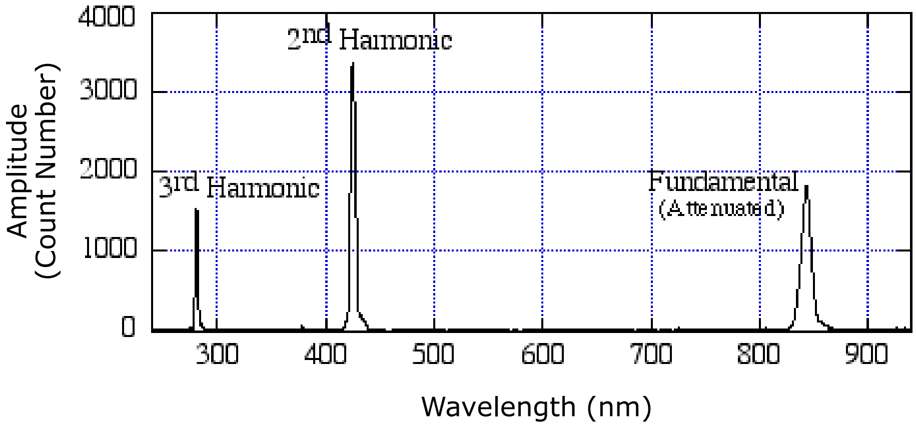
Our results show that high-gain SASE FELs generate substantial power and narrow spectra for the

NHR. We measured about five megawatts of 280-nm (third harmonic) NHR, an impressive power considering our relatively small system. Extending these results, the third NHR for the LCLS will be peaked narrowly around 0.33 Å with power several orders of magnitude larger than current third-generation synchrotron light sources.



Magnetic undulator which helped to demonstrate that coherent, femtosecond x-rays can be produced by using nonlinear harmonic SASE-FEL radiation.

__Figure 1__. SASE FEL spectra at saturation, showing fundamental, second and third harmonics. The fundamental is highly attenuated to be on the same scale as the other modes. The resolution of the spectrometer is about 1 nm.



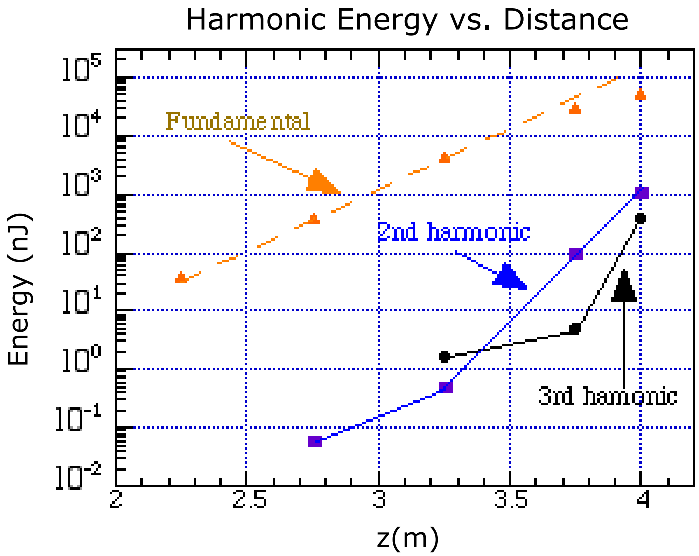
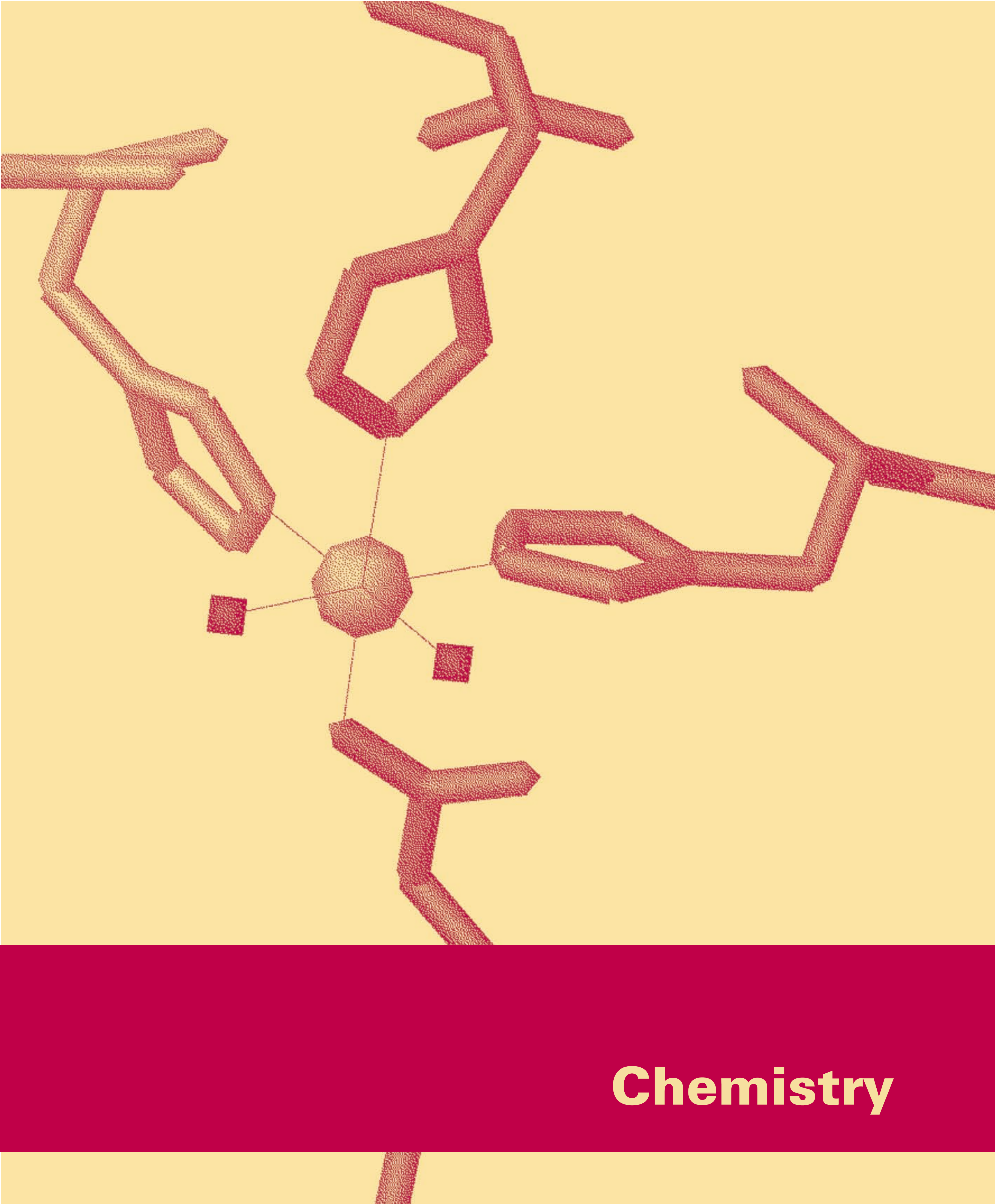


__Figure 2__. Measured Energy vs. distance for the fundamental, second and third nonlinear harmonics along the second half of the 4-meter undulator. The gain lengths are 19, 9.8, and 6.0 cm respectively. The energies at the undulator exit are 52, 0.93, and 0.40 microjoules, respectively.



Chemistry

At the National Synchrotron Light Source at Brookhaven NIST, Dow Scientists Reveal Catalysts' Private Lives

What do car interiors, adhesives, and sterilizing agents have in common? They are all produced from propylene oxide, a volatile, colorless liquid with a $10 billion-annual market value.

At the NSLS, scientists who work for the National Institute of Standards & Technology (NIST) in Gaithersburg, Maryland, and Dow Chemical in Midland, Michigan, are studying how to produce propylene oxide in a single-step, environmentally friendly way.

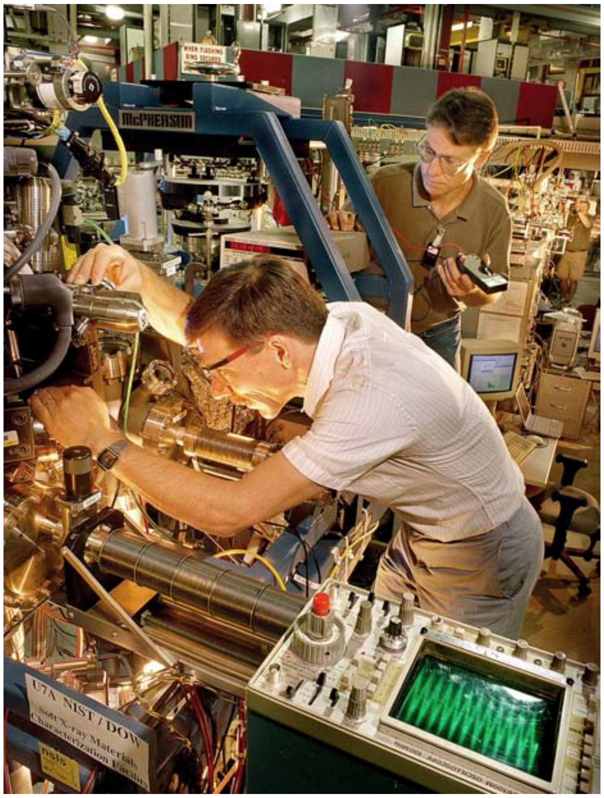
To help in their investigations, they have designed and built a state-of-the-art instrument that is now producing very exciting results.

Propylene oxide is produced by combining propylene, a flammable gas obtained by breaking up petroleum molecules, with oxygen. The process is accelerated by chemicals which act as catalysts.

"Selecting the best catalysts is usually done by testing different chemicals and choosing the one with the best results," says Daniel Fischer, NIST physicist and team leader. "Instead, we are developing a new method that reveals the complex transformations that occur during a chemical reaction. With that knowledge, we may be able to develop cleaner and more efficient catalysts for propylene oxidation."

To study the role of two types of catalysts, platinum on alumina and zeolites, which are cage-like molecules, Fischer and his colleagues use a technique called near-edge x-ray absorption fine structure (NEXAFS).

In NEXAFS, very intense x-rays are projected toward a cell containing the reactants and the catalyst. By precisely tuning the wavelength or "color" of the x-rays, the scientists select x-rays with low-energy, soft x-rays, which probe every step of the chemical reaction.



At the National Synchrotron Light Source, National Institute of Standards & Technology's Dan Fischer (front) and former Dow Chemical chemist Simon Bare prepare equipment for a catalysis experiment.

The soft x-rays are scattered or re-emitted by the chemicals, then analyzed by a detector. The detector produces a spectrum of signals, each providing information on the chemical bonds between the atoms present in the reactants and the catalyst.

"The signals act like fingerprints," says Fischer. "You can watch bonds form and break as the signals wax and wane with time. It is like looking inside a black box, the eyes being the detector and the synchrotron radiation providing the illumination."

Getting clearly separated signals, however, was not easy. When the scientists first looked at their NEXAFS spectra six years ago, the signals of interest were camouflaged by many others.

So Fischer and NIST postdoc Sharadha Sambasivan developed and refined an instrument that would filter the x-rays coming off the reaction cell by reflecting the desirable x-rays and absorbing the others. The instrument, called a normal incidence focusing multi-layer mirror, is a state-of-the-art device that dramatically reduces the background.

"The originality of this work is in being able to understand how these reactants and catalysts work together under real conditions," Fischer says. "Now we have the tools to really find out what is going on at the atomic level in catalysts at work to catch a glimpse of their private lives!"

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - February 8, 2002.]

Curbing Nitric Oxide In Car Exhaust

Smog and acid rain could be reduced in large cities if nitric oxide could be removed from car exhaust.

Nitric oxide, which also causes skin irritation and blood damage, is usually destroyed inside a car's catalytic converter, but existing catalysts are unable to meet new standards for pollution control and fuel efficiency.

Now, funded by DOE's Division of Chemical Sciences, Geosciences & Biosciences in the Office of Basic Energy Sciences, Oak Ridge National Laboratory (ORNL) scientists working at the NSLS have unveiled some of the inner workings of a compound that promises to curb cars' nitric oxide emissions more efficiently.

For the last four years, led by ORNL chemist Dave Mullins, the scientists have been looking at how a catalyst - a material used to accelerate chemical reactions - made of cerium oxide and rhodium, could be improved to destroy nitric oxide in car catalytic converters.

"In a car's catalytic converter, nitric oxide is broken down into nitrogen gas and oxygen," Mullins says. "Until now, catalysts have been selected hit or miss, so very little is known about how they actually work."

The scientists use model catalysts either powders made of cerium oxide and very finely dispersed particles of rhodium or thin oxide films grown in vacuum and pass reactant gases over them. Then they direct x-rays produced by the NSLS toward the samples while adjusting the sample temperature or reactant gas composition. Acting as a camera, the x-rays produced "snapshots" of how the catalyst broke the nitric oxide gas apart.

Two processes, called photoemission and photoabsorption, allowed the scientists to get a glimpse at the nitric oxide breakup mechanism.

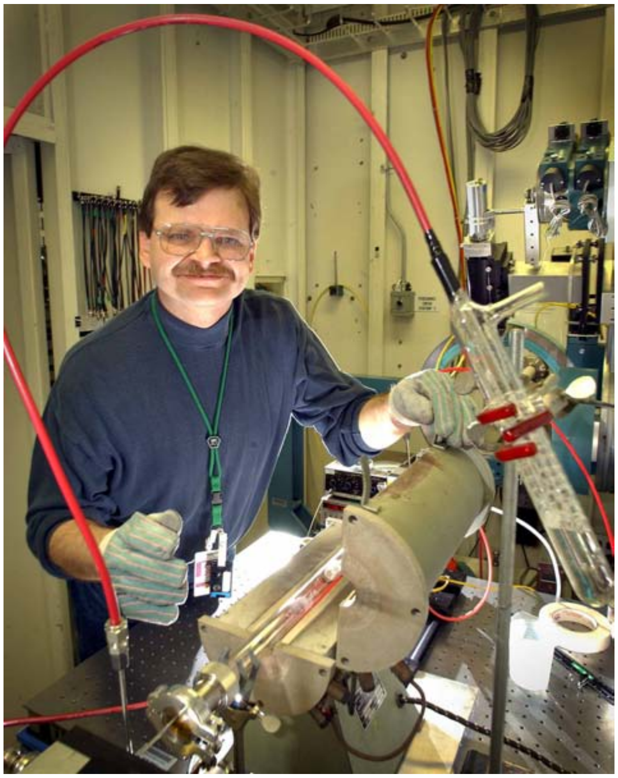
In the photoemission process, light absorbed by the compounds ejects tiny particles called electrons. By measuring the energies of these electrons, the scientists can determine the identities of gases adsorbed on the catalyst. In the photoabsorption process, light that is absorbed by the catalyst provides complementary information about the composition of the catalyst itself. These experiments confirm that the rhodium particles are the movers and shakers responsible for destroying nitric oxide.

"The rhodium particles first weaken the bond between nitrogen and oxygen," Mullins explains. "Then the nitrogen is released as a gas, and the oxygen goes into the cerium oxide or reacts with surrounding hydrogen and carbon, leading to water and carbon dioxide, respectively."

Much to their surprise, the scientists also found that the rhodium particles were being influenced by the cerium oxide.

"We thought that a rhodium particle and the cerium oxide were as independent as an object sitting on a table," Mullins says, "but you can get indirect interactions between the powder and the particles."

The interaction between the rhodium and the cerium oxide makes the rhodium more active,



Dave Mullins

increasing its ability to break the nitrogen-oxygen bond, Mullins adds.

Adding impurities in the cerium oxide can also make it work better, the scientists observed. After trying various types of impurities, they noticed that zirconium, a steel-gray metal used in alloys and ceramics, "dramatically improved the ability of the cerium oxide to exchange oxygen," Mullins says.

The scientists also looked for ways to reduce the temperature at which nitric oxide is broken down. Much of the pollution emitted by an automobile is produced just after a car is started, before the engine and catalyst reach their operating temperature. Reducing the temperature for nitric oxide decomposition will improve the catalysts performance during the warm-up period. As it turned out, the added zirconium lowered the temperature at which cerium oxide exchanges oxygen by about 100 degrees Celsius.

Mullins and his collaborators are now investigating the mechanisms by which zirconium improves the catalytic properties of cerium oxide. They are also studying the action of other impurities such as dysprosium.

"The research efforts needed to optimize the destruction of nitric oxide in car exhaust are extensive," admits Mullins, "but the knowledge that we have gained so far will lead to more new discoveries."

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - May 17, 2002.]

Discovery of a New Molecular Sieve: Zeolite SSZ-55

M. G. Wu[1], M. W. Deem[2], S.A. Elomari[3], R. C. Medrud[3], S. I. Zones[3],
T. Maesen[3], C. Kibby[3], C. Y. Chen[3], and I. Y. Chan[3]

[1]Inductive Devices, Pasadena, California; [2]Rice University, Houston, Texas; [3]ChevronTexaco Energy Research and Technology Company, Richmond, California

Scientists at the University of California in Los Angeles, and the ChevronTexaco Energy Research and Technology Company in Richmond, California, have discovered a new large-pore zeolite, which is a compound that can be used to separate molecules of various sizes and shapes, and can act as a sieve by constraining a chemical reaction medium. Due to their high thermal stability, zeolites are also used as catalysts, both in the petroleum and the chemical industries.

BEAMLINE
X7A

PUBLICATION
M.G. Wu, M.W. Deem, S.A. Elomari, R.C. Medrud, S. I. Zones, T. Maesen, C. Kibby, C. Y. Chen, I.Y. Chan, *J. Phys. Chem. B,* **106**, 264-270 (2002).

FUNDING
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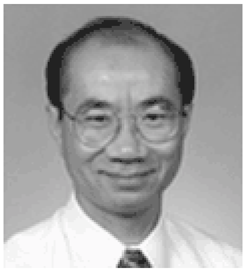

I. Y. Chan

C. Kibby

R. C. Medrud

S. I. Jones

Zeolites, which are primarily made of silicon and oxygen, have found widespread use as catalysts, molecular sieves, and ion exchangers. High-silica zeolites are of particular interest because of their importance in refining and petrochemical applications.

In virtually all instances involving high-silica zeolites, the crystallization involves complete filling of the void regions by an organo-cation guest molecule. Then, the guest molecule is thermally decomposed, leaving the ordered void regions of the zeolite, which can later be studied for catalysis or separation applications.

Our team has found a novel zeolite: $Si_{0.97}B_{0.03}O_2$, designated as SSZ-55, by using the organo-cation guest molecule shown in **Figure 1**. A key feature of this molecule is that it has both structural rigidity, conformational flexibility, and a positive charge for overall system neutrality.

It is usually difficult to grow zeolite crystals for single crystal diffraction experiments, although progress is being made at synchrotron sources to collect data from micrometer-sized single crystals. Instead, micrometer-sized crystals are easily grown and are ideal for powder diffraction. At NSLS beamline X7A, which is dedicated to powder diffraction, our team has collected high-resolution data on micrometer-sized zeolite crystals.

The unit cell was determined by indexing the experimental data with the commonly used Visser, Werner, and Louer programs. The structure was solved using ZEFSA-II, a Monte Carlo program designed specifically for solving tetrahedral framework structures. The final structure parameters were obtained by Rietveld refinement with a widely used program called General Structure Analysis System (GSAS).

SSZ-55 is tetrahedral (has four faces) with large pores surrounded by twelve silicon atoms, also called ATS. **Figure 2** shows projections of the zeolite molecule in each of the three crystallographic directions. Of particular interest is a one-dimensional channel composed of 12 tetrahedral (T) atoms. The oxygen atoms are omitted for clarity but are located halfway between each pair of silicon atoms. (Silicon-oxygen-silicon angles are usually in the range 140 – 160 degrees.)

Adsorption experiments with argon and selected hydrocarbons support the results of the structure determination. Argon adsorption shows that the pore opening is 7 ± 1 Å, which is in good agreement with the structural values of 6.5 Å x 7.2 Å. Hydrocarbon adsorption of molecules of various shapes and sizes reveal that the material has a pore opening of 12 to 14 rings, in agreement with our structural results.

Though ATS framework structure has been previously found in a variety of metal-substituted aluminophosphates (compounds containing aluminum and phosphate), SSZ-55 is the first discovered high-silica zeolite with an ATS structure. Also, the synthesis conditions are considerably different for the two classes of compounds. SSZ-55 is also interesting in that the structure does not contain any five-ring silica subunits, which tend to predominate in high-silica zeolites. Instead, the structure contains only four-ring and six-ring subunits.

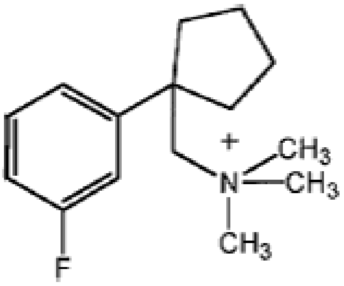

Figure 1. *Molecule used in the SSZ-55 synthesis: [(1-(-fluorophenyl) cyclopentyl)-methyl]trimethyl ammonium ion.*

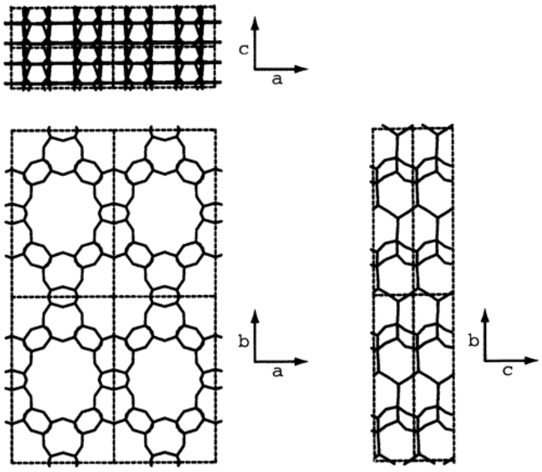

Figure 2. *Three axial projections of the SSZ-55 structure obtained from Rietveld refinement. Note that only the silicon atoms are shown.*

Role of Oxygen Vacancies in the Reduction of Nickel Oxide by Hydrogen

J.A. Rodriguez[1], J.C. Hanson[1], J.Y. Kim[1], and A.I. Frenkel[2]

[1]Brookhaven National Laboratory; [2]Yeshiva University, New York

Metal oxides are widely used as catalysts for the conversion of hydrocarbons in the chemical industry and the destruction of the sulfur dioxide and nitric oxide produced during the combustion of fuels in automobiles, factories and power plants. Though these catalysts have been designed empirically so far, scientists are now starting to understand how they operate. Using x-rays produced by the National Synchrotron Light Source, scientists from BNL and Yeshiva University in New York, have shown a direct oxidation of nickel oxide to nickel without accumulation of any intermediate phase, which may be explained by the presence of oxygen vacancies in the oxide. These results illustrate the complex role played by oxygen vacancies in the oxide reduction process.

BEAMLINE
X7B and X16C

PUBLICATION
J.A. Rodriguez, J.C. Hanson, A.I. Frenkel, J.Y. Kim, and M. Perez, *J. Am. Chem. Soc.*, **124**, 346 (2002).

FUNDING
U.S. Department of Energy,
Office of Basic Energy Sciences
Seitz Materials Research Laboratory
Yeshiva University Research Fund

FOR MORE INFORMATION
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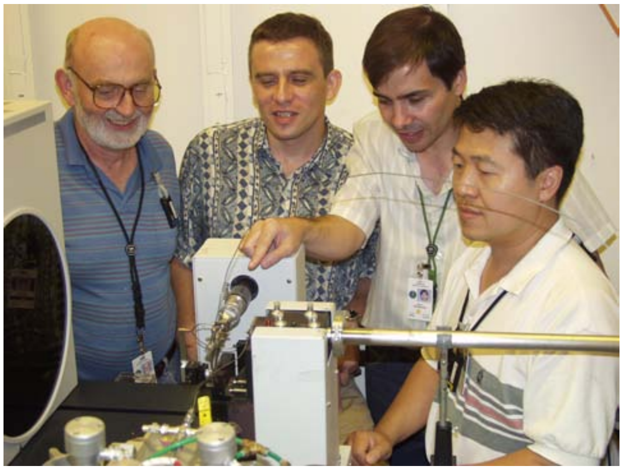
Metal oxides are used as catalysts in a large variety of commercial processes for the conversion of hydrocarbons. Also, due to their low cost, metal oxides are also used as supports of many other catalytic materials. But in most cases, oxides alone are not very good catalysts. So, one method frequently used to prepare active oxide catalysts is to partially reduce the oxide with hydrogen at elevated temperature.

Reduction of nickel oxide by hydrogen has been the object of numerous studies, because nickel oxide is a component of many industrial catalysts and electromagnetic devices. So, we decided to look at the atomic mechanisms responsible for the reduction of nickel oxide under temperature ranging from 25 to 400 degrees Celsius. Such investigations resulted from the successful combination of very intense synchrotron radiation generated by the NSLS and new parallel data-collection devices installed at NSLS beamline X7B.



Members of the team who conducted the study (from left to right): Jon Hanson, Anatoly Frenkel, Jose Rodriguez (lead author) and Jae Kim.

Experiments at beamline X7B have previously shown that *in situ* time-resolved x-ray diffraction (XRD) is a powerful technique to study oxide reduction. By using XRD and x-ray absorption techniques, we have shown that crystal and powder forms of nickel oxide are directly reduced to nickel at atmospheric pressures and elevated temperatures (250 to 350 degrees), but only after an induction – or delay – period.

During the induction period, oxygen vacancies are created within the crystal of nickel oxide. These vacancies then favor the adsorption of hydrogen to the oxide and substantially lower the energy barrier associated with the cleavage of hydrogen bonds. At the same time, adsorbed hydrogen can induce the migration of oxygen vacancies from inside the nickel oxide crystal to the surface. By inducing more oxygen vacancies on the surface, the hydrogen atoms thus "autocatalyze" the oxide reduction process, which becomes more and more efficient over time.

XRD data collected by studying the reaction of hydrogen with a nickel oxide powder at 280 degrees Celsius are shown in **Figure 1**. During the first 50 minutes, the intensity of the diffraction lines for nickel oxide remains constant (red). Then, these lines begin to disappear while lines for metallic nickel appear (blue), but no line associated with a well-ordered intermediate phase is visible.

By using near-edge and extended x-ray absorption fine structure (NEXAFS/EXAFS) techniques at beamline X16C, we also observed a similar induction time. One might argue that an amorphous phase (NiO_x, $0<x<1$), with special chemical properties, might form during the induction time. This possibility was ruled out by the NEXAFS data shown in **Figure 2**, which reveal only two compounds after oxide reduction: the produced nickel and the nickel oxide that was left from the reaction. (If an amorphous phase were created, the curve in **Figure 2** would have a different line shape, corresponding to three compounds: nickel, nickel oxide and the amorphous phase.)

Our studies indicate that oxygen vacancies play a key role in the reduction of nickel oxide, by generating the necessary sites for the dissociation of molecular hydrogen and the subsequent auto-activation of the oxide catalyst.

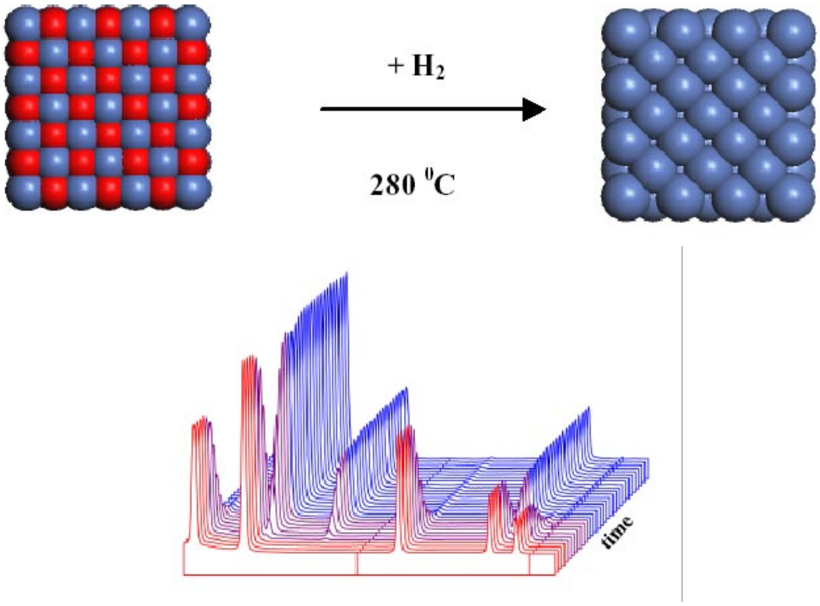


***Figure 1**. (Top) Crystal structure of nickel oxide and nickel (nickel: blue; oxygen: red). (Bottom) Time-resolved x-ray diffraction results for the reaction of a nickel oxide powder with hydrogen at 280 degrees Celsius (red lines: nickel oxide; blue lines: nickel).*

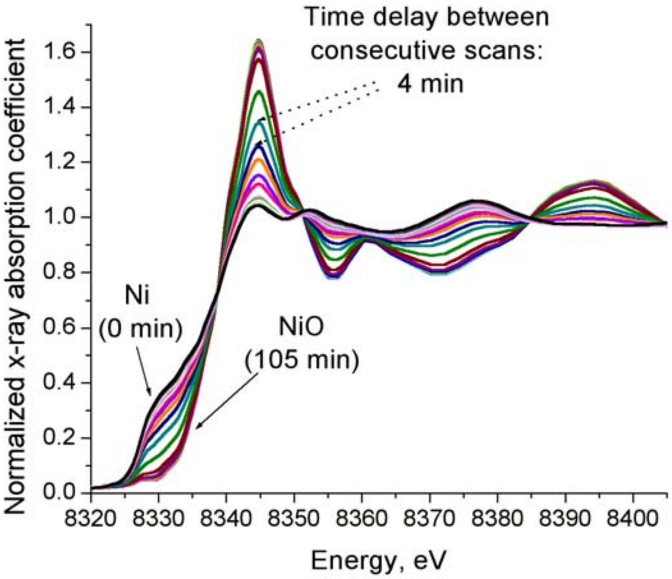


***Figure 2**. Time-resolved near-edge x-ray absorption fine structure (NEXAFS) data for the reduction of nickel oxide powder at 280 degrees Celsius under a flow of hydrogen.*

BEAMLINE
X9B

PUBLICATION
F. Al-Mjeni, T. Ju, T. C. Pochapsky, and M. J. Maroney, "XAS Investigation of the Structure and Function of Ni in Acireductone Dioxygenase," *Biochemistry*, **41**, 6761-6769 (2002).

FUNDING
University of Massachusetts, Amherst
National Institutes of Health; Division of Materials Sciences and Division of Chemical Sciences (U.S. DOE)

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Investigation of the Role of Nickel in the Regulation of the Cell Cycle

F. Al-Mjeni, T. Ju, T. C. Pochapsky, and M. J. Maroney

University of Massachusetts, Department of Chemistry

Acireductone dioxygenases (ARDs) are enzymes involved in cell cycle regulation. The ARDs produced by a bacterium called Klebsiella pneumoniae have been investigated in detail, but the precise function of one of them, a nickel-containing ARD (Ni-ARD), is unclear. Scientists from the University of Massachusetts in Amherst and Brandeis University in Waltham, Massachusetts, have examined the structure of the nickel-containing active site of Ni-ARD, in the presence and absence of substrate. The results of the study provide the first structural details of the nickel site in the only known nickel-containing dioxygenase, and provide insight into substrate binding and the role of nickel in catalyzing substrate oxidation.

Polyamines are molecules that are critical for proper regulation of the cell cycle. Addition of polyamines to cells accelerates their DNA replication and division, whereas inhibition of polyamine biosynthesis arrests DNA replication and prevents continuation of the cell cycle.

Methylthioadenosine (MTA) is a strong inhibitor of polyamine biosynthesis and transmethylation reactions, so the concentration of MTA in biological systems is tightly regulated. This control is achieved through a regulation mechanism called the methionine salvage pathway, where MTA is recycled through a series of reactions that convert it to methionine.

In *Klebsiella pneumoniae*, a bacterium causing pneumonia and urinary tract infection, the pathway has been investigated in detail, and all its intermediates have been identified. One of the intermediates of the pathway, called acireductone, is catalyzed by two enzymes called acireductone dioxygenases (ARDs). The two enzymes share a common protein and differ only in the metal ion present, which is either iron or nickel.

Reaction of acireductone with iron-ARD and oxygen produces a precursor of methionine, while reaction of acireductone with nickel-ARD does not recycle MTA to methionine. The precise function of the nickel-ARD reaction in *K. pneumonia* is unclear, and may be considered a shunt in the methionine salvage pathway, aiding in the regulation of methionine. So, we decided to investigate the structure of the catalytic nickel center in resting nickel-ARD enzyme and the enzyme-substrate complex, by using x-ray absorption spectroscopy at beamline X9B of the National Synchrotron Light Source at Brookhaven National Laboratory. We considered the following substrates: acireductone, carbon(14)-labeled substrates or substrate analogues, and oxygen(18).

X-ray absorption spectroscopy data obtained on the enzyme-substrate complex show that the substrate binds to the nickel binding site via two contact sites by displacing two ligands, at least one of which is a histidine ligand. These results show that the nickel-ARD binds to a substrate by activating its oxidation by oxygen.


Faizah Al-Mjeni (lead author)


Michael Maroney

Analyses of x-ray absorption near-edge spectroscopy (XANES) and extended x-ray absorption fine structure spectroscopy (EXAFS) data show that the structure of the nickel binding site in the resting enzyme consists of six nickel centers with six oxygen or nitrogen ligands in each center. A schematic representation of a nickel center is provided in **Figure 1.**

A comparison of the EXAFS spectra for resting nickel-ARD and the nickel-ARD enzyme-substrate complex (**Figure 2**, top-right and bottom-right spectra) reveal changes implying that the substrate binds to the nickel site. One of these changes is a dramatic decrease in the intensity of features at a distance of 3 to 4 angstroms, which we believe are due to the presence of histidine imidazole ligands, suggesting that one or more histidine ligands are displaced when a substrate binds to nickel-ARD.

Dioxygenases are known to operate by two distinct types of mechanism: One involves a redox active metal center to activate oxygen to oxidize the substrate. The second mechanism involves activation of the substrate via coordination to the metal center in order to activate it toward oxidation by dioxygen. This latter mechanism, where the metal center need not be redox active, is supported by the XAS results.

We are currently investigating the use of nuclear magnetic resonance methods to characterize in more detail metal-bound substrate species in iron- and nickel-ARDs, and to understand better the differences between the differing activities of these two enzymes.

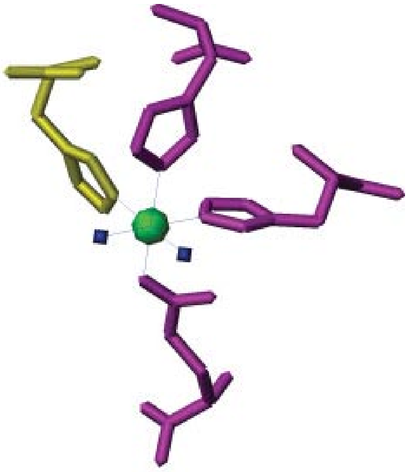

Figure 1. Nickel site structure in nickel-ARD, drawn from information obtained from x-ray absorption spectroscopy and nuclear magnetic resonance. The nickel ion (green) is surrounded by three histidine residues (purple), a carboxylate residue (yellow) and two non-protein ligands (blue squares), presumably water and/or hydroxide.

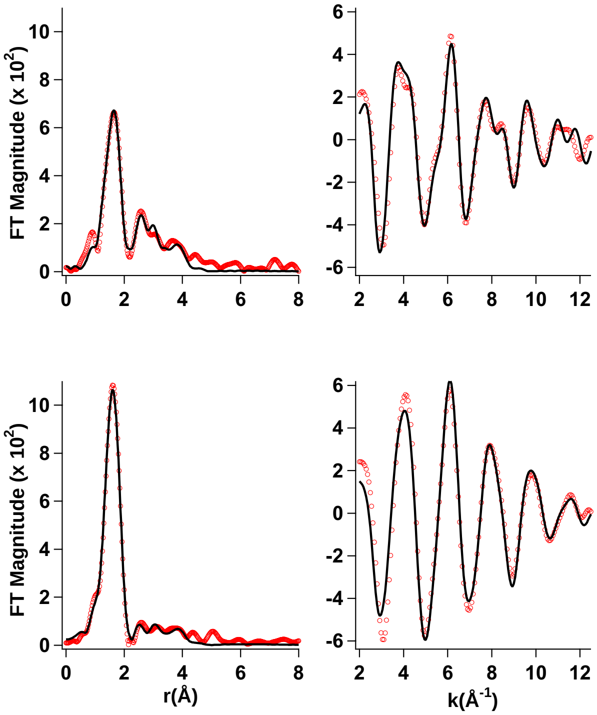

***Figure 2.** Nickel K-edge EXAFS spectra. Fourier-tranformed (left) and Fourier-filtered (right) spectra of the resting nickel-ARD (top) and the nickel ARD-substrate (bottom) of* Klebsiella pneumoniae. *Data points are represented by red open circles and the fit by a solid line.*

Synthesis and Characterization of Carboxylate-FeOOH Nanoparticles (Ferroxanes) and Ferroxane-Derived Ceramics

J. Rose[1], M. M. Cortalezzi[2], A. R. Barron[2], M. R. Wiesner[2], A. Masion[3], S. Moustier[3], J.-Y. Bottero[3] and P. Bertsch[3]

[1]Centre Européen de Recherche et d'Enseignement des Géosciences de l'Environnement (CEREGE), Aix en Provence, France, [2]Center for Biological and Environmental Nanotechnology (CBEN), Rice Univ.; [3]Savannah River Ecology Lab.

Scientists from the Univ. of Aix-Marseille in France, Rice Univ. in Houston, and the Savannah River Ecology Lab. in Aiken, S.C., have shown that the reaction between a natural iron mineral named lepidocrocite (γ-FeOOH) and acetic acid ($C_2H_4O_2$, denoted AA) in water results in the formation of carboxylate-FeOOH nanoparticles called ferroxane-AA. Upon thermolysis (cleavage of atomic bonds by exposure to high temperature), the nanoparticles evolve to homogeneous iron-metal oxides. A low firing temperature for conversion to ceramic as well as the use of environmentally benign reagents suggest that this process for creating ferroxane-derived ceramics should have minimal environmental impact.

BEAMLINE
X23A2

PUBLICATION
J. Rose, et al. "Synthesis and Characterization of Carboxylate-FeOOH Nanoparticles (Ferroxanes) and Ferroxane-Derived Ceramics," *Chem. Mater*, ***14***, 621-628 (2002).

FUNDING
Institut National des Sciences de l'Univers (INSU), France; U.S. DOE, Division of Materials Sciences and Division of Chemical Sciences; National Science Foundation; and Center for Biological and Environmental Nanotechnology (CBEN), Rice University

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Ceramic oxides are widely used in industry (for example as catalysts, paint pigments, medical supplies, chemical sorbents, and magnetic products), so new synthetic methods to produce iron oxide are constantly developed. Two common processes are currently used to produce oxide ceramics: powder processing and sol-gel. But powder processing – which is typically used to produce bulk quantities of the ceramic – involves potentially toxic agents, such as binders and solvents, and the sol-gel process may yield environmentally harmful products such as strong acids, binders, and solvents.

It is thus desirable to develop new synthetic methodologies to overcome the drawbacks of current processing techniques. We have developed a new approach for the synthesis of iron oxide ceramics based upon the reaction of large minerals with carboxylic acids. A new iron precursor for iron-ceramics, called carboxylate-ferroxane (carboxylate-FeOOH), was prepared by reacting lepidocrocite (γ-FeOOH), a layered mineral, with acetic acid (AA) in water.

The atomic environment of iron within the ferroxane was determined using iron K-edge x-ray absorption spectroscopy (XAS) of the dehydrated samples. The ferroxanes were also doped with zirconium, the structural site of

Members of the team at the University of Aix-Marseille in France (from left to right): Jean-Yves Bottero, Stéphane Moustier, Armand Masion, and Jérôme Rose (lead author).

Members of the Rice University team (from left to right): Mark Wiesner, Maria Cortalezzi, and Andrew Barron.

which was characterized at the zirconium K-edge. XAS spectra were recorded at NSLS beamline X23A2.

The structure and texture of the ferroxane were characterized by combining spectroscopic methods (Fourier-Transform Infrared (FTIR), XAS, x-ray diffraction (XRD), and light scattering), microscopic methods (atomic force microscopy (AFM) and scanning electron microscopy (SEM)) and gas adsorption data.

The current results (**Figure 1**) show that AA may cleave and break the lepidocrocite, yielding a new product: ferroxane. Ferroxanes are 300 nanometers (nm) in size and are composed of nanodomains of 20 nm in size with a γ-FeOOH structure. Each ferroxane particle is composed of a lepidocrocite core, on which AA is chemically adsorbed, as revealed by the spectroscopic techniques.

Thermolysis (cleavage of atomic bonds by exposure to high temperature) of the ferroxane-AA yields iron ceramic with the crystallographic structure of hematite. The specific surface area does not increase from the initial FeOOH mineral to the ferroxane-AA and the iron-ceramic, while the pore size distribution becomes monodisperse and the pore size diameter decreases from 55 to 12 and 13 nanometers after firing.

The ferroxane-AA was successfully doped with zirconium, which was incorporated within the structure of the ferroxane-AA. After firing zirconium-doped ferroxane-AA samples, a mixed iron-zirconium oxide was formed.

Key to this work was the development of nanoparticles using a "green" chemistry approach. Also, the ferroxanes are indefinitely stable under ambient conditions and are adaptable to a wide range of processing techniques.

We are currently testing the possibility of using the ferroxanes for different applications such as the synthesis of iron membranes and the development of new catalytic materials.

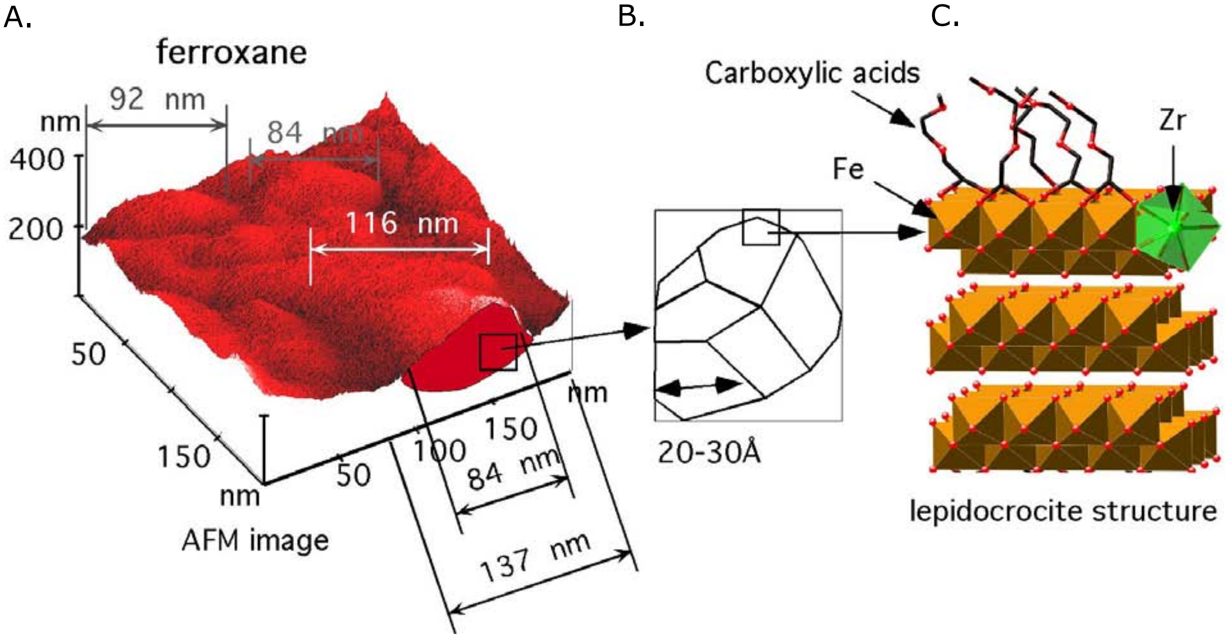

Figure 1. (A) *Atomic force microscopy (AFM) image of the texture and structure of ferroxane nanoparticles on a metallic surface. Apparent sizes of four ferroxanes are shown (116, 84, 92, and 84 nanometers).* ***(B)*** *Size of the nanodomains of each ferroxane.* ***(C)*** *Atomic structure of the nanodomains (brown: iron octahedra; red: oxygen atoms; green: zirconium site for doped sample).*

BEAMLINE
X11A

PUBLICATION
A.M. Argo, J.F. Odzak, F. S. Lai, and B.C. Gates, "Observation of Ligand Effects during Alkene Hydrogenation on Supported Metal Clusters," *Nature*, **415**, 623 (2002).

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National Science Foundation

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A Catalyst in Action: Structure and Bonding of Supported Iridium Nanoclusters During Propene Hydrogenation Catalysis Determined by EXAFS and Infrared Spectroscopies

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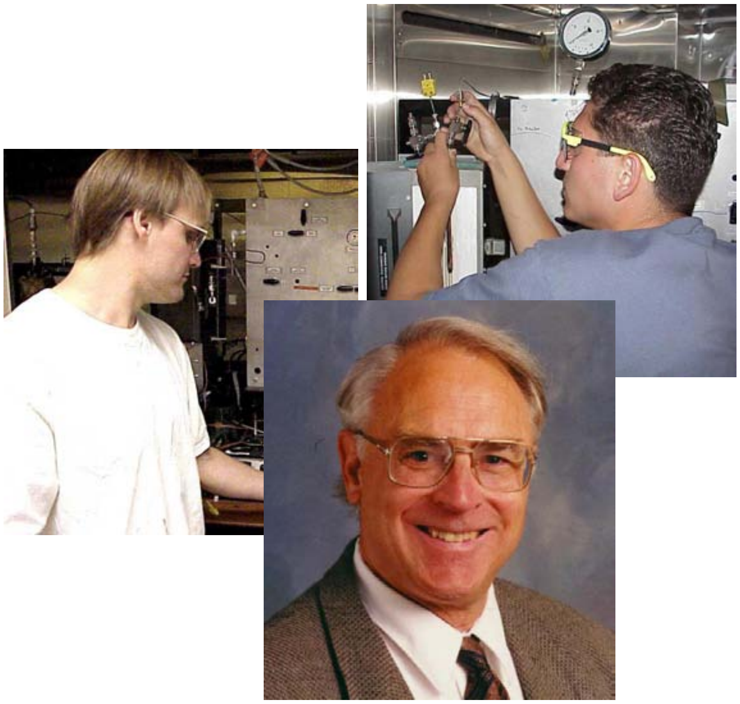
The structures of catalysts – ranging from molecular complexes and enzymes to clusters dispersed on supports – depend on the conditions under which they are used. So, the most incisive determinations of catalyst structures require techniques characterizing functioning catalysts. Scientists at the University of California, Davis have used X-ray absorption spectroscopy in combination with infrared spectroscopy to investigate supported metal cluster catalysts as they functioned, revealing the structure of the catalyst, consisting of four iridium atoms (Ir_4), and the roles of the ligands, including the support. This is the first characterization of a solid catalyst, elucidating the interplay of the support and reactant-derived ligands through their bonding with Ir_4.

Metals in catalysts used in petroleum refining or automobile exhaust conversion usually are in the form of clusters, each a few billionths of a meter in size, attached to a support. Because the clusters are so small, a large fraction of the atoms on the cluster surface is exposed, thus increasing the rates of chemical reactions. But such cluster-based catalysts are not well understood because they are made of particles of varying sizes. Scientists have developed structurally simple models, usually of single crystals, but such models do not provide information about the support's role in a chemical reaction.

We prepared nearly uniform metal nanoclusters on supports, identified the catalytic clusters and their interactions with the support by using extended x-ray absorption fine structure (EXAFS) spectroscopy, and identified the reactant-derived ligands by applying infrared spectroscopy. The EXAFS spectroscopy techniques were developed at NSLS beamline X11A.

We investigated the reaction of propene with molecular hydrogen (H_2) to form propane, catalyzed by clusters consisting of four iridium atoms (Ir_4) dispersed on porous γ-Al_2O_3 or MgO. The catalytic activity of Ir_4 depends on the support. Replacement of the MgO support with γ-Al_2O_3 boosts the catalytic activity tenfold.

When the catalyst attaches to propene or H_2 alone, we observed no significant changes in the iridium-iridium or iridium-oxygen distances. But during a catalytic reaction, the iridium-iridium and the longer, non-bonding iridium-oxygen distances exceeded the corresponding distances observed under non-catalytic conditions. We attribute the distance elongations to reaction intermediates bonded to Ir_4.



Authors (clockwise from left): Andrew Argo, Josip Osdak, and Bruce Gates.

The two supports have different characteristics that affect the electronic properties of the clusters, which in turn affect the interactions of the clusters with the ligands formed from the reactants. So, the support influences the reactivities of the reactant-derived ligands – determining which ones are observed during catalysis – and thus affects the rate of the catalytic reaction.

Our observations are consistent with the reaction mechanism shown schematically in **Figure 1**. In addition to electronic effects caused by the support, geometric properties of the clusters distinguish them from other metal catalysts. The smallness of the clusters limits the structures that can be bonded to them, thereby affecting the reactivity.

The electronic and geometric effects of the ligands are analogous to those known in molecular and enzymatic catalysis. Our results bolster theoretical cluster models that incorporate the support, and indicate new opportunities for tuning the catalytic properties of small-supported clusters by changing the nature of the support. None of the understanding emerging from this work would have been possible without the use X-ray absorption spectroscopy and its application to working catalysts.

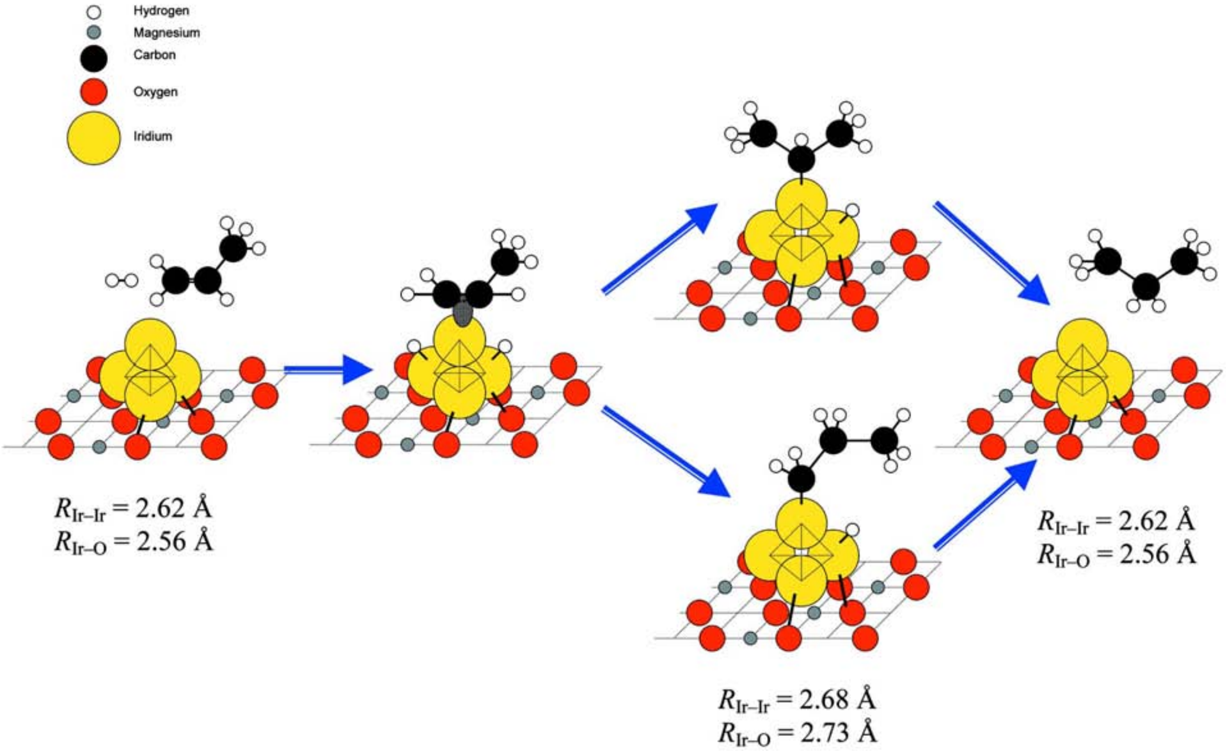
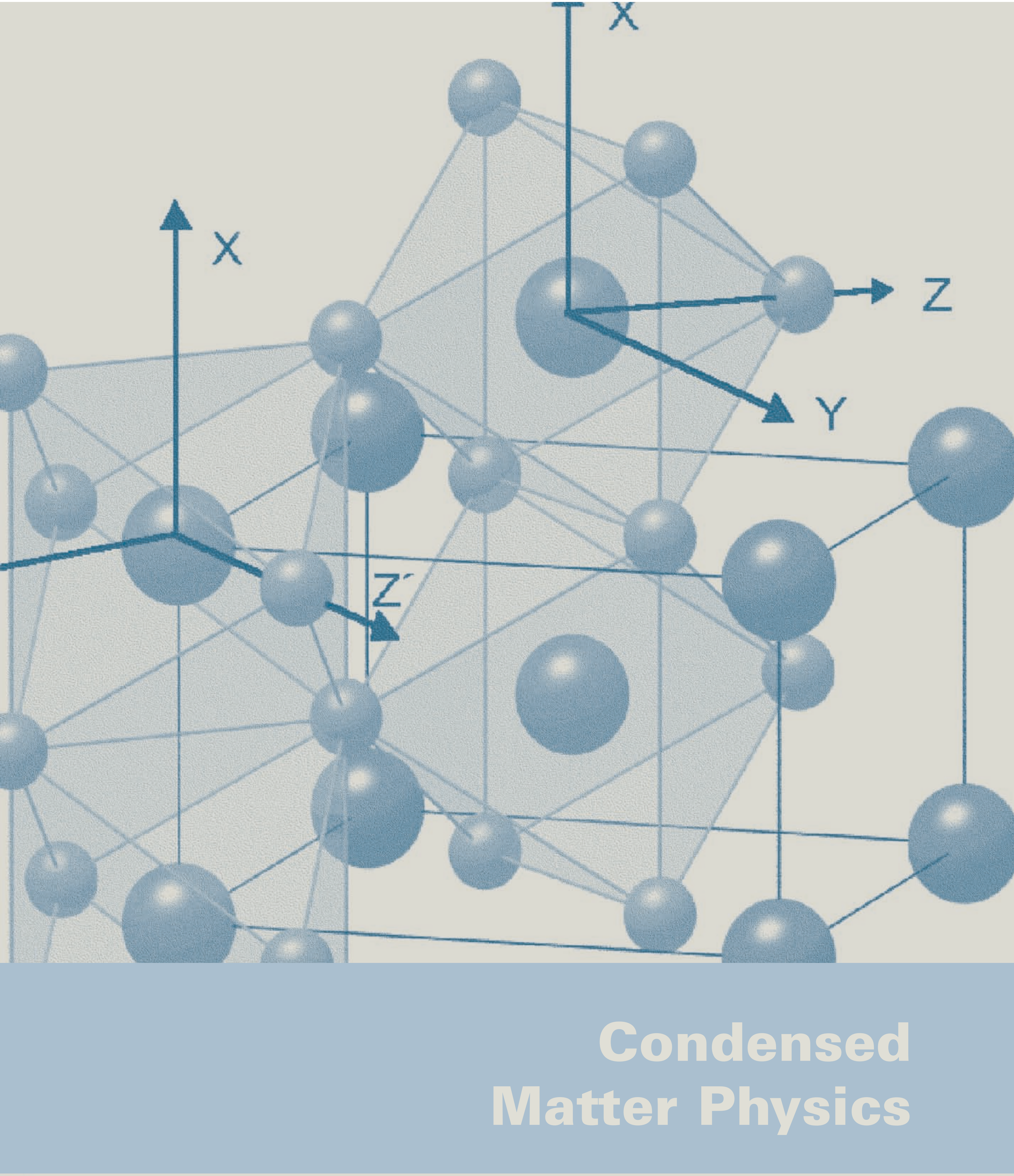


***Figure 1**. Schematic representation of the reaction of propene with hydrogen on MgO-supported Ir_4 catalyst. The structural changes shown were determined by extended x-ray absorption fine structure (EXAFS) spectroscopy of the working catalyst.*



Condensed Matter Physics

Origin of Superconductivity in Fullerenes Still a Mystery

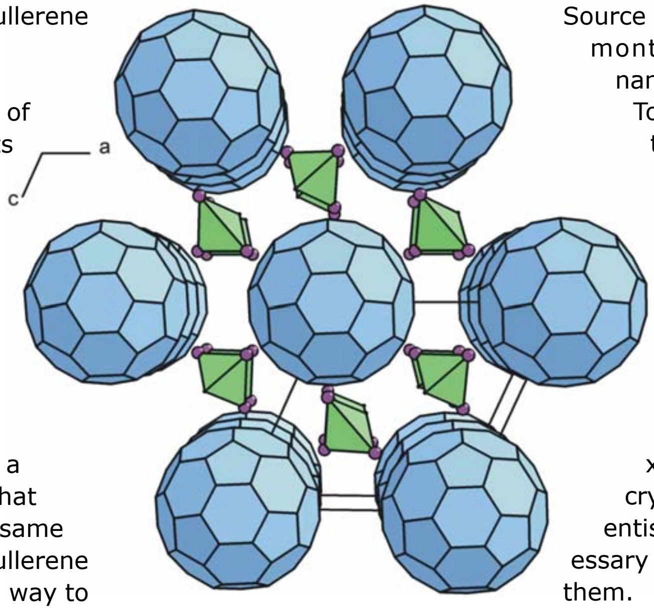
Soccer-ball-shaped molecules called fullerenes can carry electricity with no energy loss – a phenomenon called superconductivity – at temperatures below -387 degrees Fahrenheit. To increase this temperature, and thus make fullerenes' use more convenient for applications in electronics, drug delivery, and nanotechnology, scientists have long assumed that they simply needed to introduce larger and larger atoms inside fullerene compounds.

In the April 5, 2002 issue of *Science*, however, scientists working at the NSLS report an unexpected result: When they added molecules of bromoform and chloroform to a fullerene compound, the resulting compound changed its structure.

This result contradicts a widely held assumption that the structure remains the same in both pure and "mixed" fullerene compounds, and paves the way to a new understanding of how superconductivity works in these compounds.

"The theory of superconductivity in fullerenes is still too simple," says Robert Dinnebier, a physicist at the Max Planck Institute for the Physics of Solids in Stuttgart, Germany, one of the world leading labs in materials science. "Our finding means that the increase in temperature seems to be due to other causes than just an increase in spacing between the fullerene molecules."

Dinnebier and his colleagues came upon this new result while attempting to understand findings published a month earlier in *Science*. A team of physicists led by physicist Hendrik Schoen, of both Bell Laboratories in Murray Hill, New Jersey and the University of Konstanz in Germany, had, for the first time, reported that when chloroform and bromoform were added to fullerene compounds, their superconducting temperature increased dramatically.



Structure of a fullerene crystal (blue) with chloroform molecules (green/purple) inserted within the lattice. Although the structure was expected to be cubic, Dinnebier and his coworkers found that it was, in fact, hexagonal, as shown in the figure.

"When this work was published, we immediately thought it would be very interesting to look at the structure," Dinnebier says. Detailed structural studies of the spacing between fullerenes might explain the increase in temperature, the scientists assumed. "Within two weeks, we prepared the material and sent it to the NSLS," Dinnebier says.

At the NSLS, Dinnebier's collaborators Peter Stephens, a physicist at the State University of New York at Stony Brook, and his doctoral student, Ashfia Huq, set up an experiment to look at the structure of the chromoform- and bromoform-based fullerenes.

"When Robert [Dinnebier] told me that the material would be available, I was very excited," Stephens says. "We had to act quickly, because this was just before the Light Source would shut down for two months for routine maintenance."

To reveal the material's crystalline structure, the scientists used a technique called powder x-ray crystallography. They projected x-rays produced by the NSLS toward a powder of tiny crystals of the compound. Then, they determined its structure by looking at how the x-rays scattered off the crystals. In one week, the scientists had produced the necessary data and started analyzing them.

By measuring the distances between the fullerene molecules in three directions (up-down, left-right, and forward-backward), the researchers found that the expansion of the molecules due to the bromoform or chloroform mainly takes place in one direction, instead of three directions, as predicted. Surprised at first, the scientists later realized that the structure of the compounds was different from what was theoretically predicted.

In a fullerene compound, the molecules form a series of connected cubes in three directions. The molecules are on the corners of each cube, and at the center of the cube's faces.

Schoen's group expected that when bromoform or chloroform molecules are inserted inside a fullerene compound, they would sit in between the fullerene molecules. In this way, the fullerene molecules would be pushed apart similarly in the three directions, Dinnebier explains.

The new results show that the fullerene compounds with chloroform or bromoform were no longer cubic. Instead, the fullerene molecules form six-sided hexagonal shapes. The compound is made of parallel planes, each containing a series of connected hexagons. "The chloroform and bromoform molecules sit in between the planes, pushing the fullerene molecules apart only in the direction perpendicular to the planes," Dinnebier says.

While these results give a clearer picture of how a fullerene compound can be modified by adding chloroform or bromoform, they still do not provide answers to what causes the increase of the superconducting temperature. One possibility is that when chloroform or bromoform molecules are added to the fullerene compound, their electrons add to the ones already existing in the compound. The collective motion of all the electrons then creates heat that increases the temperature.

According to the theory, the number of electrons would have to increase by 25 to 35 percent to explain the measured superconducting temperature. However, theoretical physicist Olle Gunnarsson, a member of the Stuttgart team, showed that, in a hexagonal structure, the number of electrons increases by, at most, 10 percent. Another possibility, suggested by Dinnebier and his colleagues in their article, is that the chloroform and bromoform molecules may play a role through interactions between their electrons, but this hypothesis has not yet been tested. "Understanding the causes of superconductivity and the origin of the temperature below which it operates are still elusive," Stephens says. "But these results form the basis for uncovering how fullerene-based compounds generate superconductivity at the atomic level."

"This kind of research is a beautiful advertisement for the way the NSLS works," he adds. "At the NSLS, like many other synchrotron sources, it is curiosity-driven scientists, not committees, that make the decisions to schedule most of the experiments. We owe the success of this work to this well-established culture at the NSLS."

BEAMLINE
X3B1

PUBLICATION
Robert E. Dinnebier, et al., "Structure of Haloform Intercalated C_{60} and Its Influence on Superconductive Properties," *Science*, **296**, 109 (2002). -Patrice Pages

Conducting-Insulating Materials Reveal Their Secrets

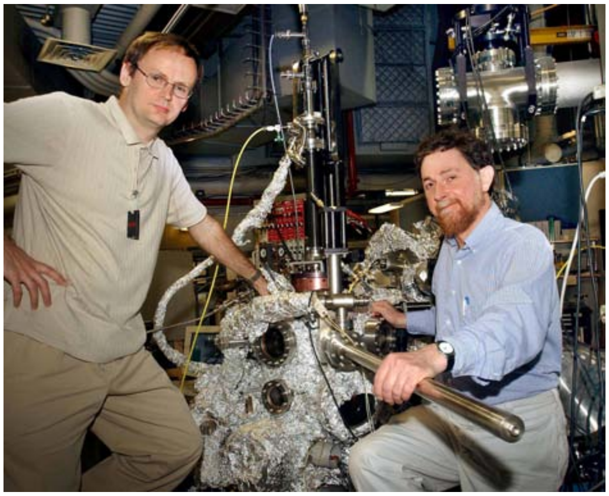
Research by physicists at the NSLS provides new insight into why some materials made of stacks of metallic planes are conductors in the direction of the planes and are insulators in the direction perpendicular to the planes. Such behavior is in marked contradiction with scientists' traditional understanding of metallic conductivity, where the electrical current is carried by electrons in every direction. Understanding how materials that are both conducting and insulating will help scientists gain new insight into superconductors – materials that conduct electricity with no energy loss.

Scientists have suspected that the dual conducting-insulating property is due to electrons interacting so strongly with each other that they do not move individually, but collectively, to carry the current within the planes. But there has been no evidence of such interactions – until now.

"A material that is both conducting and insulating is quite intriguing," says Brookhaven physicist Tonica Valla, the lead author of the study, which appears in the June 6, 2002 issue of *Nature*. "Such a dual behavior has puzzled physicists for several years. And though theoretical explanations have been suggested, we now show for the first time that the strength of the interactions between excited electrons influences their behavior."

Valla and his collaborators from Brookhaven, the University of Connecticut in Storrs, Princeton University, and Osaka University in Japan studied two different conducting-insulating materials, and showed that electrons that were confined in the planes at high temperatures are able to move between the planes at lower temperatures, allowing the material to behave more like a metal.

The "critical" temperature at which the change occurs ranges between -100 and -300 degrees Fahrenheit, depending on the material.



Physicists Tonica Valla (left) and Peter Johnson at the NSLS beamline U13, where they study the properties of conducting/insulating materials.

"These planes act like trains and electrons like passengers in the trains," says study coauthor Peter Johnson, a Brookhaven physicist. "At high temperatures, the electrons are bound together in the planes like passengers inside moving trains. Then, below the critical temperature, the electrons are not bound anymore and start moving around in the same way as passengers leave a stopped train."

To examine the interactions between the electrons, the scientists used extremely intense ultraviolet light generated by the NSLS. They looked at how the light excites the electrons in each of the three materials, and used a method called angle-resolved photoemission spectroscopy (ARPES) to accurately measure the intensity of the light emitted by the electrons as a function of their energy. The resulting ARPES spectrum was determined for various temperatures.

Below the critical temperature, a signal started to appear in the spectrum. Weak at first, the signal became more apparent as the temperature decreased. "This signal is the telltale evidence of individual electrons," Valla says. "Theorists had predicted the existence of such a signal, but nobody had observed it before."

The results of this study promise to provide further insight into how superconductors conduct electricity without heat dissipation when they are cooled below a certain temperature. In particular, the cause of high-temperature superconductors, with critical temperatures ranging from -396 to -216 degrees Fahrenheit, remains mysterious, but is assumed to be due to strong interactions between electrons.

The new study also gives insight into materials with new electrical and magnetic properties, expected to arise from strong interactions between electrons. "We expect to see dramatic new results and applications stemming from the study of materials with strongly-interacting electrons," Johnson says.

-Patrice Pages

BNL, NIST, Norway Gain New Insight into the Superconductivity of MgB_2

New insight into the superconductivity of magnesium diboride (MgB_2) has been recently revealed by a team of scientists from BNL, the National Institute of Standards & Technology (NIST), and the University of Oslo in Norway. The findings appear in the June 17, 2002 issue of *Physical Review Letters*.

The new information about MgB_2, an unusual superconductor discovered only last year, promises more understanding of superconductivity – the ability of some materials to conduct electricity without losing energy – which could lead to improved magnetic resonance imaging, more efficient electric power transmission, and smaller, more powerful electronic devices.

"Scientists usually assume that superconductivity arises from electrons coupling in pairs," said Yimei Zhu of BNL's Energy Sciences & Technology Department, lead author of the study. "Though this is the case for most superconductors, it has not been shown yet how electrons contribute to superconductivity in magnesium diboride." Therefore, the team decided to investigate this through research supported by DOE and DOC.

Since the discovery of superconductivity in MgB_2, BNL theoretical scientists, led by physicists James Davenport, Energy, Environment, & National Security Directorate, and Guenter Schneider, Physics Department, made extensive calculations involving interactions between electrons or between electron "holes," which are empty locations that could be filled by electrons.

"Superconductivity in MgB_2 is expected to arise from interactions between holes," says BNL physicist Arnold Moodenbaugh, a member of the team. "Because MgB_2 is made of alternating planes of boron and magnesium atoms aligned parallel to one another, these holes are expected to interact more easily within the planes than between adjacent planes."

"Compared to other superconductors, MgB_2 has a relatively simple structure," says Johan Tafto, a physicist at the University of Oslo and another team member. "We hoped to get more insight into superconductivity by focusing on a simple compound rather than on more complex ones."

To test the theoretical predictions about MgB_2, the researchers used two complementary techniques: x-ray absorption spectroscopy and electron-energy-loss spectroscopy. In the first technique, very intense x-rays enter the sample and are absorbed by the electrons inside it. In this experiment, the electrons, which are ejected out of their original positions, are tracked in a unique x-ray detector, designed and built by NIST physicist Daniel Fischer and his team.

"When the ejected electrons fall into the holes, they reveal the number and density of the holes in the sample," said Fischer, who has worked with the x-ray absorption technique for the last 18 years at the NSLS.

The second technique uses state-of-the-art transmission electron microscopes (TEMs) at BNL. Unlike optical microscopes, which use visible light, electron microscopes project electrons toward the sample. These electrons transfer some of their energy to electrons in the sample, which bump around the sample atoms and reveal the

Near the NIST/Dow beamline U7A at the NSLS, where part of the MgB_2 study was conducted, are team members Daniel Fischer, Yimei Zhu, Genda Gu, Arnold Moodenbaugh (back), Johan Tafto, Tom Vogt and James Davenport. Absent from the photo are team members Guenter Schneider and Qiang Li.

positions of electronic holes in the sample.

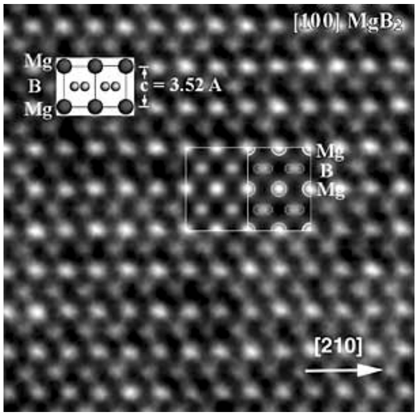
"We needed to use both techniques because they complement each other so well," Zhu said. "They lead to a very accurate determination of the distribution and number of electron holes in MgB_2." The leader of BNL's TEM group at the Advanced Electron Microscopy Facility, Zhu has been investigating the electronic structure of materials at the nanoscale (one billionth of a meter) for the last 20 years.

By showing that interactions between holes in the boron planes do occur in MgB_2, and that superconductivity stems from such interactions, the scientists showed that results from both techniques agree with the theoretical predictions.

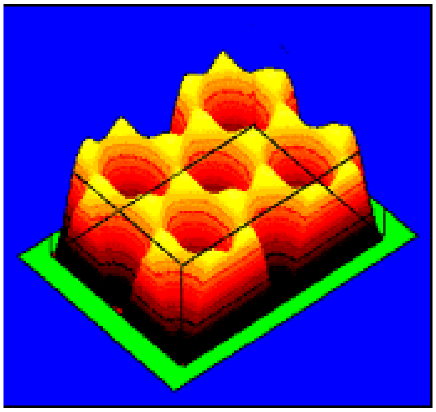
Said Tafto, "As we gain more understanding of the properties of MgB_2 at the atomic level, I am confident that, in the near future, we will be able to relate them to macroscopic properties such as superconductivity - and maybe explain the origin of superconductivity in general."

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - June 21, 2002.]



A plane of magnesium atoms (white spheres) above a plane of boron atoms (grey spheres) in MgB_2, as seen by the transmission electron microscope used in the study. The embedded image (top left) is a model of the structure, showing the expected positions of the atoms and the distance between two magnesium planes.



An illustration of the distribution of electrons between boron and magnesium atoms. Electrons cluster around the boron atoms (yellow peaks) and move away from the magnesium atoms (orange valleys).

Scientists Create ‘Light Tweezers’ to Study the Electronic Properties of Solids

Many techniques are currently available to investigate electronic properties – such as conductivity and magnetism – of solids, but for materials made up of different chemical elements, the contributions of the electronic properties of the atoms of each element are difficult to distinguish. Now, a team of scientists has perfected a new technique that does just that.

The scientists have been working on the technique, called site-specific x-ray photoelectron spectroscopy, for the past two years, and have recently used it to investigate the electronic properties of oxygen and titanium atoms in rutile, a mineral made of titanium dioxide usually with a little iron, and used to accelerate chemical reactions. The results of the study, reported in the August 12, 2002 issue of *Physical Review Letters*, represent the first experimental application of the technique to a metal oxide, thus paving the way to understanding the properties of more complex metal oxides used in the chemical and electronics industry.

“Our technique can unambiguously determine the individual contributions of either the titanium or the oxygen atoms to the electronic structure of rutile,” says physicist Joseph Woicik, of the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, and the lead author of the study. “In contrast, other techniques describe the electronic structure of rutile as a whole, which does not say much about how titanium and oxygen atoms interact with each other.”

Picking Different Atoms With Optical “Tweezers”

The new technique, which combines two widely used techniques, called photoelectron spectroscopy and x-ray diffraction, works as follows: Very intense x-rays are first projected towards the material and then deflected by the atomic planes of the material, creating “diffracted” x-rays. When the incident and diffracted x-ray waves combine, they form a standing wave, which is used to excite electrons inside the material. The period of the standing wave, which is the distance between two crests, can be made equal to the distance between two atoms of a given element. As a result, the crests of the wave selectively excite the electrons of the atoms of a given element, but not the electrons of the atoms of other elements. The crests of the standing wave can then be moved in a controlled fashion, thereby allowing them to stimulate electrons of the other elements’ atoms at will.

To study the electronic properties of titanium and oxygen atoms in rutile, Woicik and his colleagues used x-rays generated by the NSLS.

“Because the waves have the same period as the inter-atomic distance, we can selectively excite either the titanium atoms or the oxygen atoms,” Woicik says, “and when one of these atoms is excited, all the other atoms of the same element are excited at once.

“The crests of the standing wave act like tweezers that reach in and pick out electrons,” Woicik says. “The advantage is that you know that the electrons you are studying are coming from either the titanium or the oxygen, but not both.”

The scientists analyzed the energy of the ejected electrons to gain information about the electrons’ positions and energies in the crystal before the electrons were excited.

At beamline X24A (from left to right): Physicist Joseph Woicik (lead author), beamline technical support Barry Karlin and physicist Lonny Berman (co-author). (courtesy of Daniel Fischer, NIST).

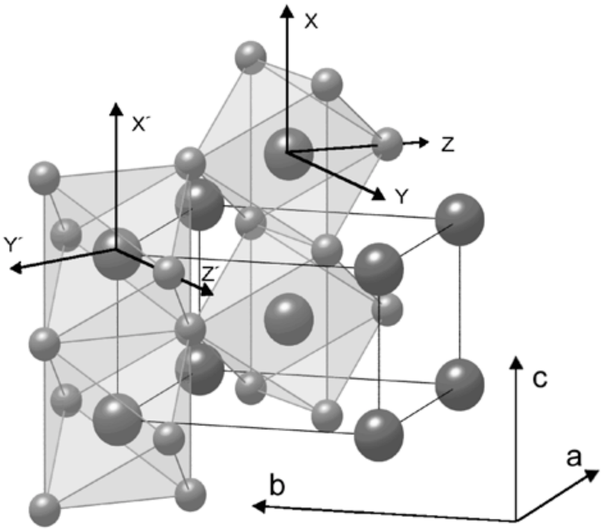


Figure 1: *Crystallographic structure of rutile. Titanium atoms are represented by large spheres and oxygen atoms by small spheres.*

The analysis was performed with a state-of-the-art device called an electron analyzer, and ultra-high vacuum tools specially designed and built by scientists from the University of Rhode Island in Kingston, the NSLS, and NIST, working at NSLS beamline X24A.

Covalent Bonds and Electric Insulation

By closely looking at the positions of the electrons, Woicik and his colleagues found that the binding of the titanium and oxygen atoms does not follow the more conventional ionic picture, in which the electrons responsible for the binding hover around oxygen atoms but not titanium atoms. The new study shows that, instead, titanium and oxygen atoms share their electrons, making “covalent” bonds.

“We were surprised to notice such a substantial electron density coming from the titanium atoms,” Woicik says. “This means that electrons are present around the titanium atoms, which shows that the bonding is covalent.”

The researchers also examined the positions of electrons responsible for the conducting or insulating properties of a solid. In the case of rutile, they found that the electrons with the highest energies are not jumping between the atoms, as would be expected in a conductor, but are localized around the oxygen atoms, justifying that rutile is an insulator.

Unexpected Experimental Results

The new experimental results nicely agree with theoretical expectations. But this agreement came as a surprise. Last year, Woicik and his colleagues were testing their technique on copper when they discovered that “valence” electrons, which are peripheral to the nuclei, displayed emission patterns similar to “core” electrons, which are localized close to the nuclei.

“We could not believe what we were seeing,” Woicik says. “Because valence electrons are free to roam in between atoms, there was no reason that their emission patterns would be the same as those of core electrons.”

Half convinced that their presumptions might be incomplete, the scientists considered more carefully the nature of the electron waves within the crystal. “Our presumptions were wrong,” Woicik says. “Only when the valence electrons are near the cores can they be excited by the x-rays.”

These observations demonstrated that the scientists need to consider only the contributions from the individual atoms. “The technique truly works like tweezers that only reach in and grab the electrons in the immediate vicinity of each atom,” Woicik says. “This was the

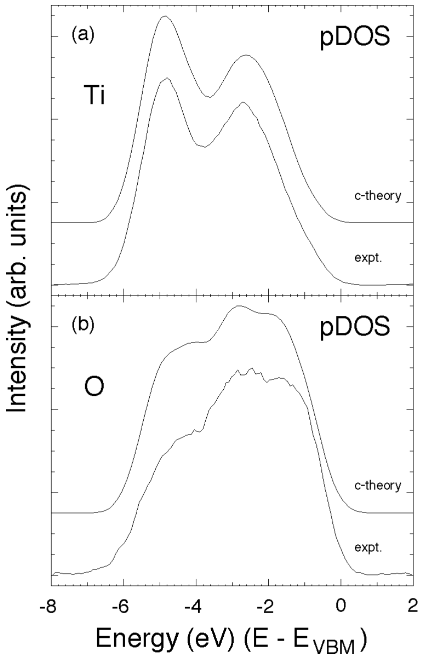


Figure 2: *Comparison of theoretical and experimental densities of states of electrons surrounding the titanium (top) and oxygen (down) atoms. The experimental curves, which were obtained from the site-specific x-ray photoelectron spectroscopy technique, agree nicely with the theoretical curves, attesting for the ability of the technique to act as “light tweezers,” picking out either the titanium or oxygen atoms.*

most startling observation that I have ever made in 13 years working in this field."

More Results to Follow

The researchers are now interested in applying their technique to more complex metal oxides than rutile, including materials in which electrons interact collectively, a phenomenon responsible for superconductivity – electrical conductivity without energy loss below a certain temperature. The technique could also be used to shed light on the magnetic properties of materials by looking at the magnetic properties of individual nuclei and electrons.

"The beauty of our technique is that it can unravel the electronic structure of simple metal oxides in a way similar to the way you peel an onion," Woicik says. "By looking at how electrons interact with each other, you can then make more sense of the electronic properties of more complex metal oxides and possibly address intriguing phenomena such as high-temperature superconductivity and metal-insulator transitions."

BEAMLINE
X24A

PUBLICATION
J.C. Woicik, E.J. Nelson, L. Kronik, M. Jain, J.R. Chelikowsky, D. Heskett, L.E. Berman, and G.S. Herman, "Hybridization and Bond-Orbital Components in Site-Specific X-Ray Photoelectron Spectra of Rutile TiO_2", *Phys. Rev. Lett.* **89**, 077401 (2002).

-Patrice Pages

BEAMLINE
U13UB

PUBLICATION
Z. Yusof et al., "Quasiparticle Liquid in the Highly Overdoped $Bi_2Sr_2CaCuO_{8+d}$", *Physical Review Letters* **88**, 167006 (2002).

FUNDING
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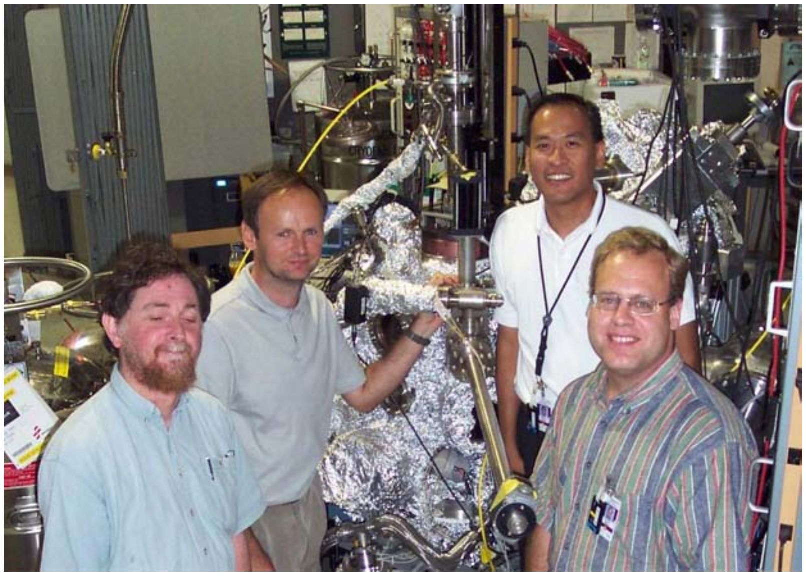
Quasiparticles Refuse to Go Away in High-Temperature Superconductors

Z. Yusof[1], B.O. Wells[1], T. Valla[2], A.V. Fedorov[2], P.D. Johnson[2], Q. Li[2], C. Kendziora[3], Sha Jian[4], and D.G. Hinks[4]

[1]University of Connecticut, Storrs; [2]Brookhaven National Laboratory; [3]Naval Research Laboratory, Washington, D.C.; [4]Argonne National Laboratory, Illinois

Scientists working at NSLS beamline U13UB have shown that provided new insight into the properties of overdoped cuprates (bismuth-strontium-copper-oxides). The researchers found that while underdoped cuprates have exotic properties, overdoped cuprates behave more like regular metals.



(From left) Peter Johnson, Tonica Valla, Zikri Yusof, and Barry Wells

Fifteen years after the discovery of cuprate (bismuth-strontium-copper-oxide) superconductors (1986), the origin of superconductivity in these materials is still unknown, and scientists are still investigating the nature and behavior of the charge carriers in these materials. One of the most studied of these compounds is $Bi_2Sr_2CaCu_2O_{8+\delta}$, also called Bi2212, which has provided considerable insight - as well as new puzzles - on the origin of high-temperature superconductivity.

Cuprates share a set of generic features. These compounds become superconductors only when they are doped with electrons or holes. Undoped, they are antiferromagnetic insulators. As they are doped either with holes or electrons, they evolve first into a strange metal and eventually become superconducting with increasing critical temperature T_c until this temperature reaches a maximum value at what is known as optimal doping. Increasing the doping level beyond this point causes Tc to drop again (**Figure 1**). A doping level below the optimum value is known as underdoping, while that above the optimum value is known as overdoping.

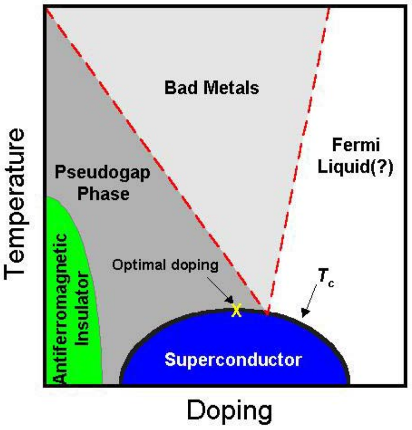


Figure 1. *Generic phase diagram of high-T_c superconductors.*

The underdoped and optimally-doped superconductors have a number of unusual properties, especially in the normal state (above T_c). One puzzling property is the apparent absence of "quasiparticles", entities that are defined in Landau's Fermi Liquid theory. This theory has successfully described the properties of ordinary metals, semiconductors and conventional superconductors (discovered before 1986), but cuprates, especially in their normal state, appear to defy explanation with this theory.

Samples of overdoped cuprates have been studied using angle-resolved photoemission spectroscopy

(ARPES), which is a more sophisticated version of the familiar photoelectric effect. In ARPES, the outgoing photoelectrons are collected by an electron analyzer that allows us to obtain the energy and momentum distributions of these electrons. The presence of a quasiparticle gives a sharp peak in the ARPES spectrum.

We found that the superconducting state has quasiparticles at all doping levels but they persist to the normal state only for the overdoped compound (**Figure 2**). We also established that while underdoped cuprates have exotic charge transport, the overdoped cuprates have more familiar quasiparticle physics - closer to a regular metal like copper. The next step is to understand how the material evolves from one form to another, either through a gradual crossover or a sharp phase transition.

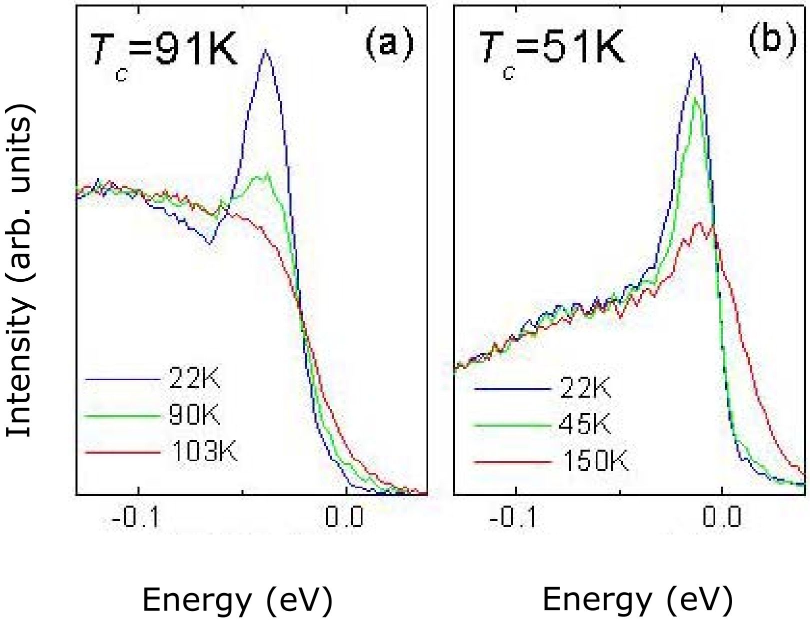


***Figure 2**. Energy distribution of photoelectrons. (a) Optimally doped superconductor with T_c=91K. The sharp peak in the spectra, representing well-defined quasiparticles, is not detected above T_c [Fedorov et al. PRL 82, 2179 (1999)]. (b) Overdoped superconductor with T_c=51K. Sharp peak (quasiparticles) exists well above T_c.*

BEAMLINE
X21

PUBLICATION
Q. Qian, T.A. Tyson, C.-C. Kao, M. Croft, and A.Yu. Ignatov, "Local Magnetic Ordering in $La_{1-x}Ca_xMnO_3$ Determined by Spin-Polarized X-ray Absorption Spectroscopy," *App. Phys. Lett.* **80**, 17, 3141 (2002).

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Probing Local Magnetic Order in Transition Metal Oxides

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In the transition metal oxide $La_{1-x}Ca_xMnO_3$ (with $0<x<1$), electrical conductivity is strongly influenced by external magnetic fields, making this material an exceptionally good candidate for a new generation of magnetic sensors that may significantly improve magnetic data storage. To probe the local spin arrangement in transition metal oxides, we have developed a method to directly assess the local magnetic ordering about specific metal atoms. This method promises to lead to a better understanding of magnetism in magnetic oxides and to assist in improving theoretical models of correlated electron systems.

Intriguing phenomena such as high-temperature superconductivity (electrical conductivity without resistance) and colossal magnetoresistance (change in electrical resistance due to the presence of a magnetic field) have stimulated renewed interest in the physics of transition metal oxides based on perovskite ($CaTiO_3$), by using techniques such as x-ray absorption spectroscopy (XAS) and x-ray emission spectroscopy (XES).

High resolution XAS work has provided evidence of intriguing temperature dependencies of the manganese K-XAS pre-edges of $La_{1-x}Ca_xMnO_3$ materials which accompany changes upon crossing phase boundaries involving combinations of insulating, paramagnetic, antiferromagnetic, ferromagnetic, and charge-orbital ordered phases. But these interphase changes are not well understood. Consequently, we used a powerful combination of XAS and XES, called spin-polarized x-ray absorption near edge spectroscopy (SPXANES), to extract spin-polarized x-ray absorption spectra and integrate the results into a model which is sensitive to local magnetic order.

SPXANES is based on energy resolving the 3p to 1s transition (K_β emission) and measuring the emission from the main or satellite lines of the spectra as a function of the incident x-ray energy. Previous studies focused on the nature of the splitting between spin up and spin down channels in the main line, but no emphasis was made on the pre-edge region and on the temperature-dependent changes in SPXANES.

Manganese SPXANES measurements were performed at beamline X21 of the National Synchrotron Light Source. SPXANES spectra were collected by monitoring the K_β fluorescence yield at two energies, specific to spin up and down final states, while the incident energy across the near-edge region was scanned. Measurements were performed for x = 0, 0.3, 0.5, and 1 (corresponding to $LaMnO_3$, $La_{0.7}Ca_{0.3}MnO_3$, $La_{0.5}Ca_{0.5}MnO_3$ and $CaMnO_3$, respectively) at two temperatures: 15 and 300 Kelvin (K).

All the K-edge SPXANES spectra of $La_{1-x}Ca_xMnO_3$ are shown in **Figure 1**. They reveal a marked splitting between the spin up and spin down channels of both the pre-edges (energy between 6.535 and 6.55 kiloelectronvolts (keV)) and the main edges (energy between 6.55 and 6.58 keV).

In **Figure 2**, we expand

Members of the team of scientists who performed the study (from left to right): Trevor Tyson (lead author), Qing Qian, and Chi-Chang Kao.

the pre-edge region of the SPXANES spectra. Three peaks (labeled a1, a2, and a3) in $La_{1-x}Ca_xMnO_3$ (x = 0, 0.3, 0.5, and 1) are observed. By examining the changes in the pre-edge spectra for temperatures above and below magnetic ordering temperature, we have developed an electron excitation model to understand the origin of the features a1, a2 and a3.

In **Figure 3**, we display our transition model, which shows that the changes in the pre-edge region with temperature can be directly linked to changes in magnetic ordering of the manganese ions around absorbing manganese sites. The model also shows a transition from a random local magnetic arrangement to an ordered arrangement.

Our model could be used to investigate a great range of perovskite transition metal materials, and, more generally, octahedrally-coordinated transition metal materials. Our model and SPXANES measurements could also be used to predict magnetic ordering changes in materials such as thin films, single crystal or powders.

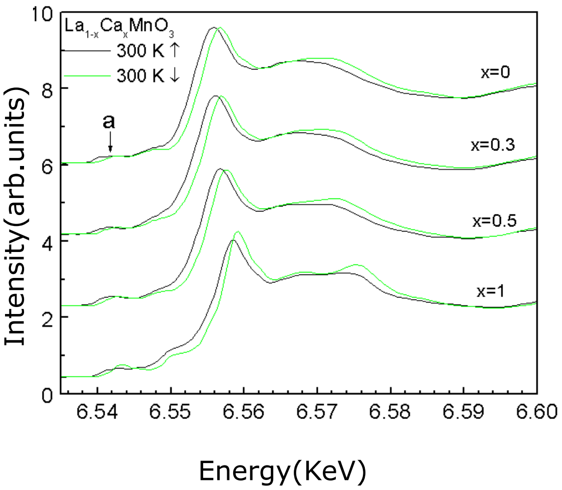

Figure 1. *SPXANES spectra of $La_{1-x}Ca_xMnO_3$ for x = 0, 0.3, 0.5, and 1. The solid and green lines correspond to the spin up and down channel, respectively, measured at 300 Kelvin.*

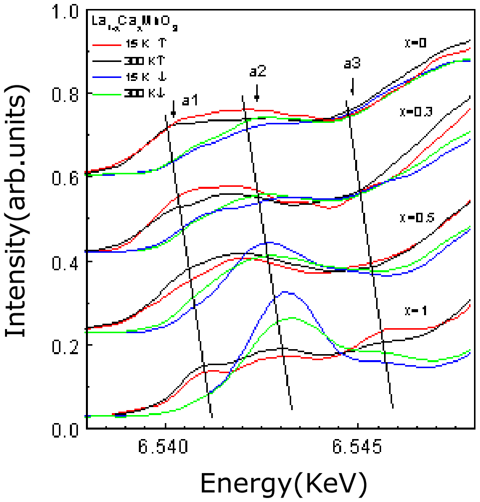

Figure 2. *Temperature-dependent pre-edge SPXANES spectra of the region near feature* a *in Figure 1. The three straight lines indicate the a1, a2, and a3 features.*

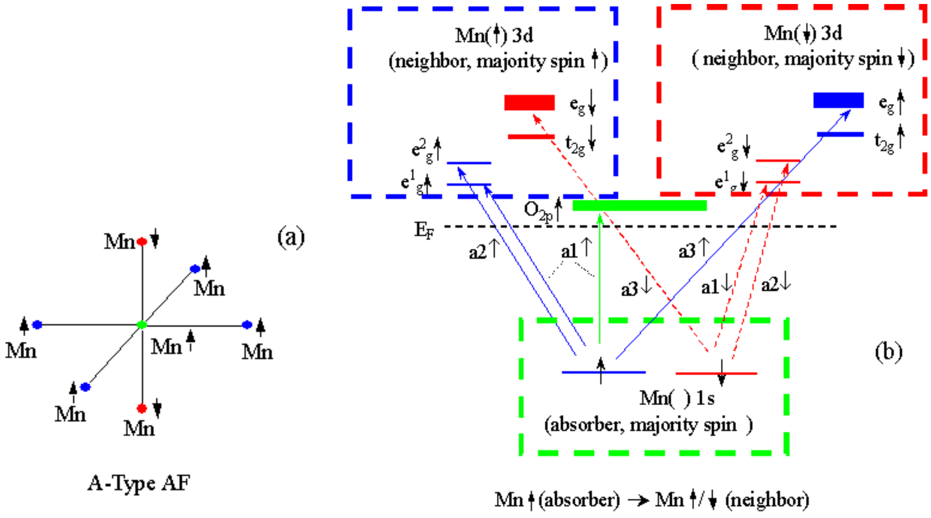

Figure 3. *Schematic diagram showing the transitions in the pre-edge for spin-polarized absorption of $LaMnO_3$ in the low-temperature A-type antiferromagnetically ordered state. (a) Local magnetic ordering of magnetic ions. (The oxygen atoms are not shown for clarity.) (b) Allowed transitions for excitation of manganese, in which the d final state of a neighbor is of the same spin polarization as the absorber (left panel) or the spin polarization is reversed (right panel).*

BEAMLINE
X15B

PUBLICATION
P. M. Voyles, D. A. Muller, J. L. Grazul, P. H. Citrin, and H.-J. L. Gossmann, "Atomic-scale Imaging of Individual Dopant Atoms and Clusters in Highly *n*-type Si", *Nature* **416**, 826 (2002).

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Imaging Single Dopant Atoms and Clusters in Bulk Silicon

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As silicon-based transistors in integrated circuits grow smaller, the concentration of charge carriers generated by impurity dopant atoms must steadily increase to maintain the transistors' performance. But current technology is rapidly approaching the limit at which introducing additional dopant atoms ceases to generate additional charge carriers, because the dopants form electrically inactive clusters. Scientists from Bell Laboratories have used x-ray absorption measurements at beam line X15B of the National Synchrotron Light Source, and scanning transmission electron microscopy (STEM) to understand the nature of these dopant clusters in silicon. The STEM data represent the first observation of single atoms and clusters the size of a few billionths of a meter inside a bulk solid, and they provide insight into how the clusters are formed.

The electrical properties of a semiconductor – a material having electrical conductivity greater than insulators but less than good conductors, and used especially as a base material for computer chips and other electronic devices – can be controlled by adding small amounts of foreign atoms, called dopants. When dopants such as antimony are added to a semiconductor such as silicon, the dopants occupy lattice sites (the locations occupied by atoms in the crystal) in place of silicon atoms, and donate electrons, thus changing the electrical properties of the silicon.

The shrinking size of electronic devices requires that the concentration of electrons – and therefore the concentration of dopants needed to generate them – must increase to maintain device performance. However, at the high dopant concentrations needed for future silicon device technology, not all the dopants remain electrically active. Instead, they form electrically inactive clusters. It is therefore important to understand how the dopant atoms become electrically inactive, leading to the observed saturation of free-electron densities.

We have shown, by performing extended x-ray absorption fine structure (EXAFS) measurements at beamline X15B that, in highly antimony-doped silicon samples, the most commonly accepted models for the dopant clusters, in which three or four antimony atoms surround a silicon vacancy, were not correct. We used first-principles calculations, which suggest that the dominant clusters in these samples contain only two dopant atoms with no silicon vacancies.

These findings have now been tested using scanning transmission electron microscopy (STEM) on the same antimony-doped samples after mechanically thinning them to a thickness of less than five nanometers. (One nanometer is one billionth of a meter.) The thinning procedure was particularly demanding, because surface roughness and oxidation had to be kept to a minimum to ensure maximum imaging contrast between the crystalline silicon and antimony atoms. The small sample thickness was important for optimizing the likelihood that only a single antimony atom would occupy a given column of silicon atoms when viewed in projection.


Authors of the study (from left to right): David Muller, Paul Citrin (seated), Hans Gossmann, John Grazul, and Paul Voyles (lead author).

Figure 1 shows a STEM image of a sample. The atomic columns of silicon containing at least one antimony atom are seen as the brightest dots on the left, whereas no such dots are seen in the undoped region on the right. Applying filtering and particle-counting algorithms to such images from samples only 2.3 and 1.5 nanometer-thick (corresponding to 11 and 9 silicon atoms per column) revealed that the distribution of single dopant atoms was purely random. This suggests that overcoming free-electron density saturation at very high dopant concentrations will be a major challenge.

Figure 2 compares calculated structures for clusters containing two and three antimony atoms with corresponding simulated and experimental images. Careful analysis shows that the two-atom clusters outnumber those with three or four atoms by 50:1.

The observed random distribution of dopants provides insight into the formation mechanism of the clusters. We have recently performed additional experiments and calculations to characterize the two-atom structures more completely, and the results are being prepared for publication.

Our study provides the first, unambiguous observation of single dopant atoms within a bulk crystal. This observation paves the way for further analysis of single atoms and clusters of two to four atoms, and for the study of the structure of impurities and alloy constituents in crystalline solids.

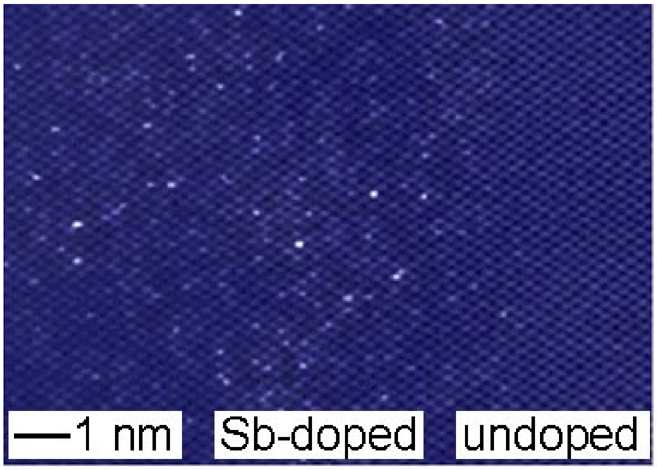


__Figure 1__. Scanning transmission electron microscopy image of a cross-section of highly antimony-doped silicon. The brightest dots (left) are atomic columns of one or more antimony atoms, which are absent in the undoped sample (right).

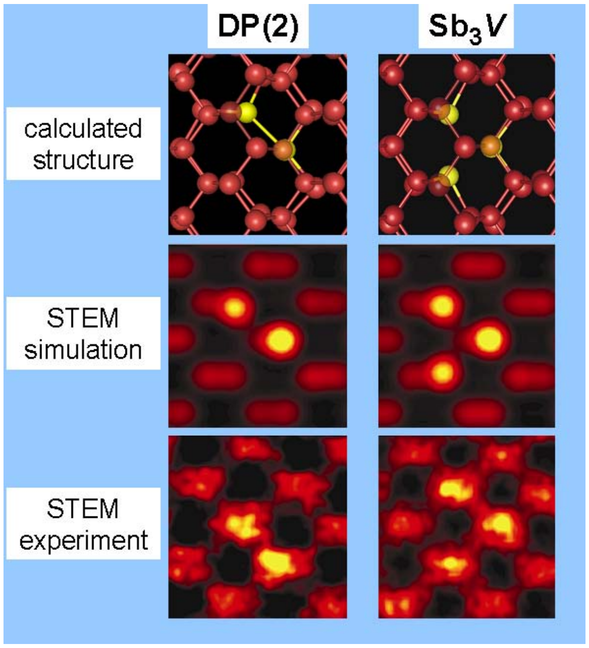
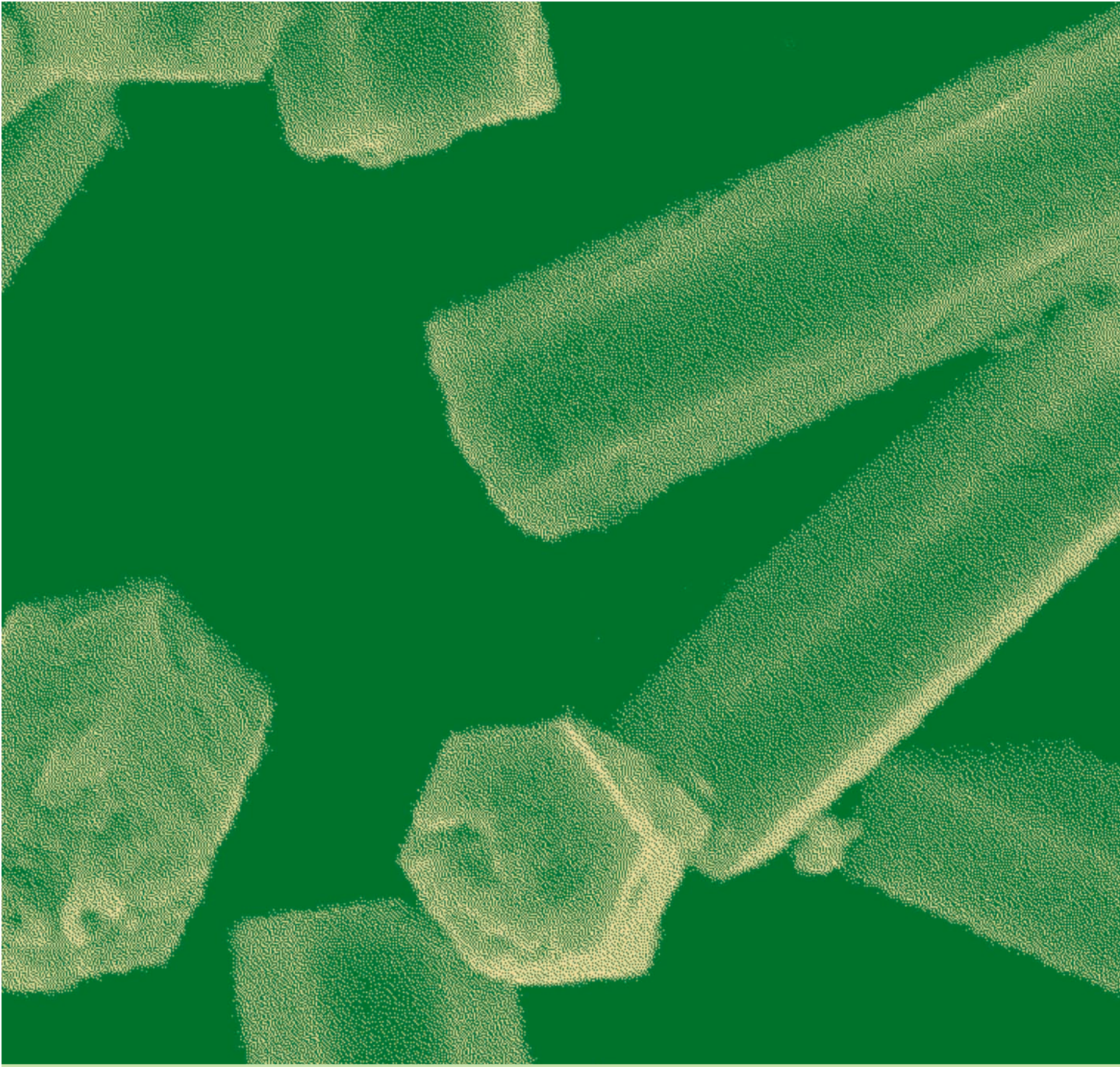


__Figure 2__. Calculated structures compared with simulated and experimental scanning transmission electron microscopy images of a donor-pair defect cluster with 2 antimony dopant atoms and no silicon-atom vacancy (left) and of a of 3-antimony-atom cluster surrounding a vacancy (right). Antimony and silicon atoms are in yellow and red, respectively. The silicon atoms appear orange when in front of antimony atoms.



Geology and Environmental Sciences

Scientists Provide Evidence that Life on Earth May Have Come from Comets

Scientists have provided new evidence for the existence of iron sulfide – iron combined with sulfur – in comets, which are large chunks of ice and rock that have remained nearly unchanged since the beginning of the solar system. The catalytic behavior of iron sulfides is involved in the formation of hydrocarbons – key building blocks of life – so this discovery reinforces the idea that seeds of life may have come to Earth from comets. The results of the study are described in the May 9, 2002 issue of *Nature.*

"Sulfur is one of the more abundant elements in space," says Lindsay Keller, a scientist at NASA's Johnson Space Center (JSC) and lead author of the study, "yet astronomers have never been able to account for it around young stars – until now."

Two groups of scientists participated in the study. The first looked at dust particles coming from comets and falling in Earth's atmosphere. The second made astronomical observations of the disks of dust and gas surrounding young stars, and determined the chemical composition of these disks.

"When the solar system formed, the dust that was not swept up in the solar system ended up in comets," Keller says. "This dust has been sitting inside the icy comets for 4.5 billion years. So comets represent a sort of a freezer of presolar materials. By looking at the particles given off by comets, we can track down the particles that were present in the beginning of the solar system.

"Another way to understand how our solar system started to form is to look at disks of dust and gas around young stars," he adds. "What happens there is very similar to what is thought to have occurred in our solar system."

Cosmic vs. Earth Dust

Although the origin of comets is still uncertain, scientists believe that most reside outside the orbits of the planets in the Kuiper belt and Oort cloud. As a comet makes its way towards the sun, it releases dust and gases as the surface is warmed. A fraction of the dust that is released is swept up by the Earth.

"Because these particles are so small, many of them slow down in the upper atmosphere (at ~100 km altitude) without getting strongly heated – so they are not strongly altered – and they slowly settle over weeks to months on to the ground," Keller says, adding that the Earth accretes roughly 40,000 tons of interplanetary dust each year.

NASA has, for the past two decades, sent aircraft with special sticky collectors to altitudes of 12 to 14 miles to capture the cosmic dust as it falls through the stratosphere before it becomes mixed with Earth dust.

The collected dust particles, which are one-tenth the diameter of a human hair, are examined in the ultra-clean Cosmic Dust Laboratory at JSC.

"The dust particles are so tiny that they are invisible to the naked eye," Keller says, "so you need a microscope to see them and a very clean environment not to contaminate them with dust coming from the air."

Scientists wearing clean-room overalls – often called "bunny suits" – use high-resolution, light optical microscopes to examine the morphological characteristics and chemical composition of the par-

The inner solar system is suffused with a vast cloud of interplanetary dust. This dust cloud is invisible to the naked eye as the zodiacal light - a triangular glow rising above the horizon shortly after sunset or before sunrise. The picture to the right shows the zodiacal light together with comet Hale-Bopp. (Courtesy of Richard Wainscoat).

ticles carefully. The particles are then stored in dust-free, nitrogen-filled cabinets, which are available to other scientists for more detailed investigations.

Ultra-thin samples of dust

But the dust particles are so tiny that scientists cannot handle them easily. To investigate the chemical composition of some of the dust particles, Keller and his colleagues John Bradley, George Flynn and Don Brownlee surround the particles by beads of epoxy resin 10 to 20 times larger than the grains, thus making the beads easier to handle than the grains. "Having a dust grain inside an epoxy bead is like having a bug inside of an ice cube," Keller says.

Once the epoxy hardens, the scientists mount the bead on a sample-holding device called a microtome, which is a very finely-machined instrument that cuts the bead into extremely thin slices, the thickness of each slice being three thousandth of the bead's diameter. "Iron sulfide is a very dense and strongly absorbing mineral," Keller says, "so when the samples are that thin, you can finally get light through the sample."

The scientists determined the composition of the dust-containing slice by using two complementary techniques. The sample was first analyzed at JSC with a transmission electron microscope, in which electrons are sent through the sample to reveal its atomic structure and chemical composition. The slice of dust was also studied by using very intense light from beamlines U10B and X1A at the NSLS. By looking at how infrared light is absorbed in the samples, the scientists can determine what kinds of minerals and compounds are present in the particles.

Keller and his colleagues discovered that several of the dust particles were mainly made of iron sulfides. "When we looked at the infrared spectra, lo and behold, we had a very nice signal that corresponded exactly to iron sulfide," Keller says. Using very intense light was key to the detection of iron sulfide, he adds, because the samples are "so small and so thin" that the intensity generated by the sample is too weak to be detected by off-the-shelf lab instruments. "The infrared intensity in NSLS's VUV ring is about 500 – 1000 times brighter than conventional instruments, which translates into a much improved signal," he says.

Cosmic Dust Laboratory (CDL) at NASA JSC.

But more exciting results awaited Keller and his colleagues when they compared their results with those of another group of scientists looking at young stars trillions of miles away from Earth.

An unknown signal from the cosmos

What inspired Keller and his collaborators to analyze their particles were the results from astronomers studying the chemical composition of disks of dust and gas around two stars recently formed. The two balls of fire, called the Herbig stars, are surrounded by disks of dust and gas, which have not given birth to planets yet – if planets were to arise from the disks.

The scientists studied the two stars with the Infrared Space Observatory Observatory (ISO), an astronomical satellite that investigated the infrared light emitted by the dust and gas surrounding fledgling stars from 1995 to 1998.

The scientists, working at the Astronomical Institute in Amsterdam in The Netherlands, the European Space Agency in Noordwijk, The Netherlands, and the Astronomical Institute and University Observatory in Jena, Germany, identified many of the chemical elements present in the two stars. But, when they decided to remove from the stars' infrared spectra the contributions from all known elements, a signal of unknown origin appeared.

"When the astronomers subtracted everything from their spectra that they knew was there – the silicates, the glasses, the carbon, and ices – there was always a big residual feature," Keller says.

Iron sulfide revealed again

"The ISO scientists interpreted the signal incorrectly as being possibly due to iron oxide," Keller says. "In fact, our signal occurred at the same position on the spectra as their signal. It was a very nice match in terms of peak position, shape, and width."

After studying carefully their spectra, the two groups agreed that the "unknown" signal was indeed produced by iron sulfide. They also considered spectra of terrestrial samples known to contain iron sulfide and found that they also matched very well with their respective experimental spectra.

The researchers also found that their results agreed with predictions on the abundance of solid sulfur around young stars. "Astronomers have predicted that sulfur was all present as dust grains, not in the form of gas," Keller says. "Our results could account for the abundance of sulfur in the iron sulfide grains, as predicted."

Seeds of life?

By showing for the first time that iron sulfides are both present in comets and around young stars, Keller and his collaborators provide further evidence that "seeds of life," in the form of dust grains or hydrocarbon molecules, may have led to the development of life on Earth.

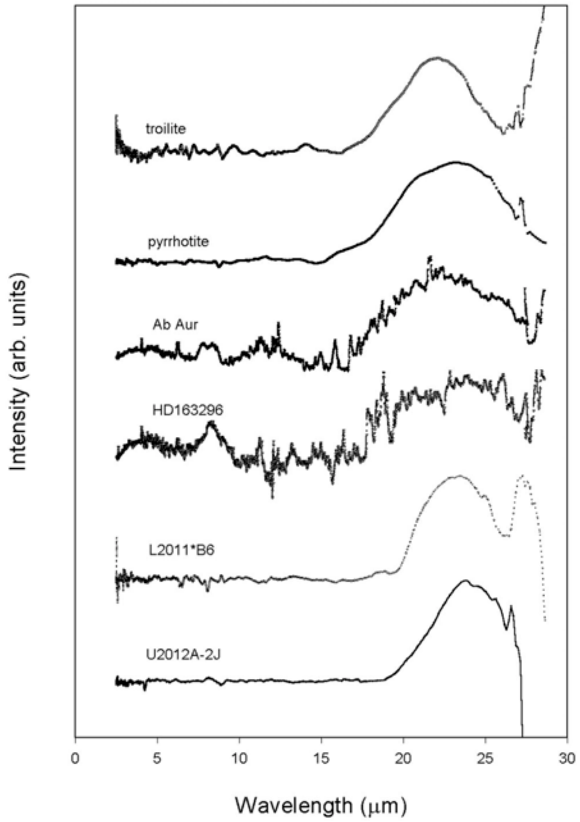
"Many of the current models for how Earth acquired early organic molecules are from particles that came out of space," Keller says. "So comets may have brought to Earth the chemicals that are necessary to life, such as organic molecules and water."

It is interesting to consider that "seeds of light" may have been pouring on Earth from the sky since Earth's creation. "I am fascinated to be working on grains of materials that are older than the solar system, "Keller says. "To realize that the accumulation of vast amounts of star dust that ended up forming our sun and the planets around it is just as fascinating."

BEAMLINE
U10B and X1A

PUBLICATION
L.P. Keller, et al., "Identification of Iron Sulphide Grains in Protoplanetary Disks," *Nature*, **417**, 148 (2002).

-Patrice Pages



The first two spectra are from terrestrial standards, which are samples known to contain iron sulfide. The two spectra in the middle are from observations of young stars called the Herbig stars. In these spectra, the scientists have subtracted all the known components such as silicates, ices, and carbon from the original spectra. The bottom two spectra are from dust particles, found to be very rich in iron sulfide. All three types of spectra show a peak that is infrared active at the same wavelength. Keller and his collaborators show that the peak is due to iron sulfide, a chemical that was probably present during the early times of the solar system formation and may have sowed the seeds of life on Earth.

SBU Researchers at the NSLS Discover Properties of Perovskite, Explain Why No Earthquakes Start in Earth's Lower Mantle

Recent research at the NSLS, reported in the October 24, 2002 issue of *Nature* by Stony Brook University (SBU) scientists, illustrates for the first time why earthquakes do not start in the Earth's lower mantle, the lower part of the roughly 1,400-mile-thick portion surrounding Earth's core.

Using bright, hard x-rays from the superconductor wiggler beamline X17, Jiuhua Chen of SBU's Mineral Physics Institute led research in measuring the strength of $Mg_{0.9}Fe_{0.1}SiO_3$ perovskite, a dominant mineral of the lower mantle, at high pressure and temperature. Collaborators in this research, which is funded by the National Science Foundation, are Donald Weidner and Michael Vaughan, both of SBU.

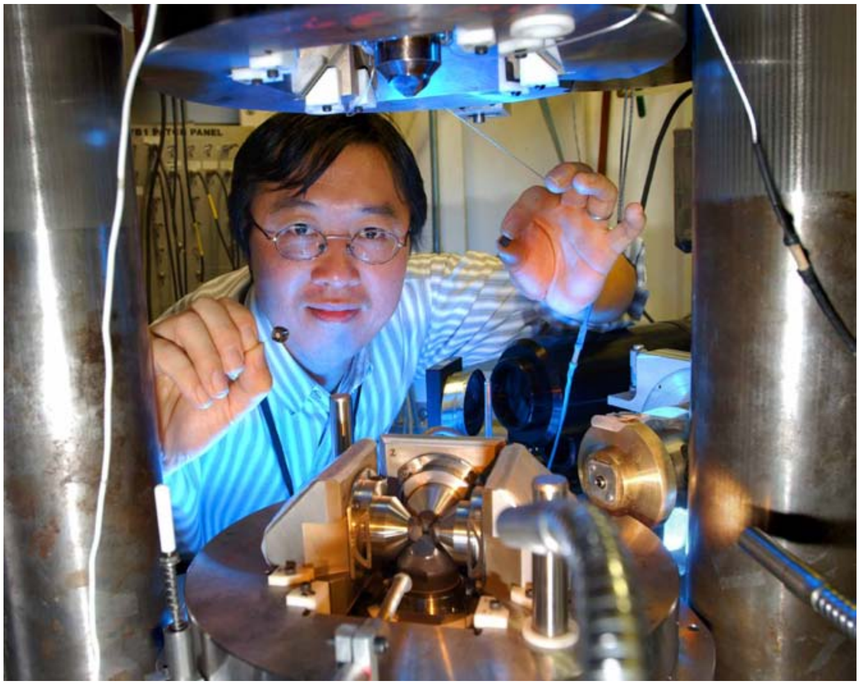
"We found that the perovskite not only is stronger than other minerals at high pressure and temperature, but also has a temperature-insensitive plastic-flow character," said Chen. "The result revises the existing prediction of the rheological property — which is the flow of a material under stress — of Earth's lower mantle and thus supplies the first experimental evidence for understanding why earthquakes do not start in the lower mantle.

"This result also sheds light on many other of Earth's dynamics," added Chen.

He explained that, from surface to center, Earth's structure is made up of the crust; the upper mantle; the transition zone; the lower mantle; the outer core, which is liquid; and the inner core. Earth's crust is divided into areas called tectonic plates which move separately and rub against each other. Some earthquakes occur when adjacent plates "catch" and then suddenly release the pressure that has built up over time. These are called near-surface or shallow earthquakes.

When two plates collide, the thinner, denser plate is forced under the thicker, lighter plate and sinks deep into the mantle. This process is called subduction.

Earthquakes can also start along the subducting slab. These earthquakes are called deep earthquakes.



Jiuhua Chen is at beamline X17B at the NSLS, where a Stony Brook University team has developed equipment that can simulate temperature and pressure conditions needed for studying Earth's structure and dynamics.

"Our result helps in understanding more about the deflection of the subducting slab at the top of the lower mantle, because the strong perovskite may create a mechanical barrier to the subduction," said Chen. "We can now also predict a viscosity jump at the boundary between the transition zone — the portion of Earth that is between 250 and 410 miles deep — and the lower mantle, which is very important information for modeling convection processes in Earth."

Chen explained that the mechanism of deep earthquakes has been a long-standing question. Geophysicists know that seismic observations show no earthquakes starting in the lower mantle, but until the SBU study of perovskite, the reason was unclear.

"We have studied the rheological properties of many mantle minerals," Chen said. "Our results indicate that, among many possible mechanisms, plastic instability may be responsible for the earthquakes."

Investigating the properties of perovskite is difficult because this mineral, unlike others, is unstable under ambient conditions.

"However, with the experimental instrumentation that we developed at the NSLS, we could finally challenge the problem," said Chen. "Our goal is to gain a full understanding of Earth's structure and dynamics. With the NSLS right on our doorstep, we are approaching this goal."

-Liz Seubert

[Editor's note: Reprinted with permission from the BNL Bulletin - November 1, 2002.]

BEAMLINE
X17C

PUBLICATION
H. Cynn, J. E. Klepeis, C.-S. Yoo, and D.A. Young, "Osmium has the Lowest Experimentally Determined Compressibility," *Physical Review Letters* **88**, 135701 (2002).

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Osmium Is Stiffer Than Diamond

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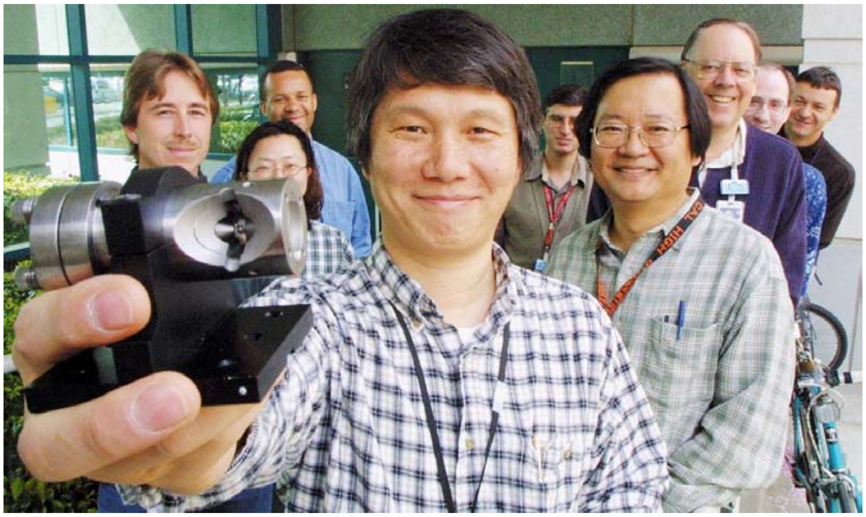
Scientists from the U.S. Department of Energy's Lawrence Livermore National Laboratory (LLNL) working at the NSLS and LLNL's Stanford Synchrotron Radiation Laboratory have reported the surprising discovery that metallic osmium is stiffer than covalently bonded diamond. The researchers also found that iridium and ruthenium are as incompressible as rhenium. In addition, they performed first-principles calculations that independently confirm the observed trend in the transition metal bulk moduli.



Members of the LLNL high-pressure physics experimental group and collaborating theoretical physicists. Physicist Hyunchae Cynn (front), who is the lead author of the study, is holding a diamond-anvil cell of the type used in the osmium experiments.

Diamond is the hardest known material. However, in searching for other hard materials it is generally simpler to study the compressibility, which is often correlated with the hardness. The compressibility is a measure of how much the volume changes for a given change in pressure and is equal to the reciprocal of the bulk modulus. A material that has a low compressibility (incompressible, high bulk modulus) is stiff or difficult to compress.

In addition to being the hardest known material, diamond also held the record for the highest bulk modulus at 443 gigapascals (4.43 million times atmospheric pressure). However, our newly measured value of 462 gigapascals for osmium exceeds that of diamond. We also found that iridium and ruthenium are as incompressible as rhenium, which has previously been reported to be a strong metal.

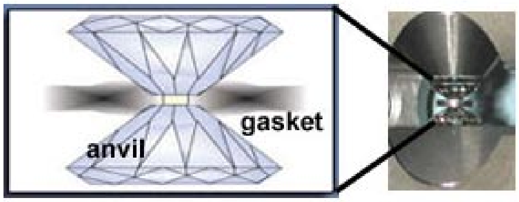


***Figure 1**. Two opposing diamond anvils supported by tungsten carbide mounts provide a sample chamber by placing a metal gasket in between the flat tips (approximately 300 micrometers in diameter) of the two diamond anvils. The gasket has a small hole (less than 100 micrometers) drilled at the center.*

It has long been known on the basis of the simple Friedel model of chemical bonding in transition metals that the largest bulk moduli should occur in the middle of each transition metal row in the periodic table. The position of osmium in the middle of the 5d row is thus consistent with the high value of the bulk modulus we have obtained. As far as we know, no compressibility data for osmium at high pressures have been reported previously.

We performed compression studies with a diamond-anvil cell (**Figure 1**) up to 65 gigapascals for osmium, ruthenium, and iridium. Condensed argon was used as a pressure medium, and ruby grains smaller than three micrometers in diameter were used to determine the pressure. Powder samples with grain sizes less than five micrometers were contained inside a small hole (80 micrometers in diameter) drilled into a rhenium gasket.

The measured diffraction patterns were dispersed either in energy using unfocused white x-rays from the superconducting wiggler beamline X17C at NSLS, or in angle using focused monochromatic x-rays from the wiggler beamline 10-2 at SSRL. **Figure 2** shows a typical energy-dispersive x-ray diffraction pattern for osmium at 65 gigapascals, consisting of the diffraction lines from hexagonal close packed osmium and face-centered cubic argon, and the x-ray emission lines from osmium.

Measured compression data for osmium, iridium, and ruthenium were fit using the third-order Birch-Murnaghan equation of state (EOS). The osmium data and fit are both shown in **Figure 3**. We have also carried out first-principles calculations of the EOS for these three metals. The calculations yield the same trend in the bulk moduli, thus providing an independent confirmation of our experimental results.

In summary, we found that osmium has the largest bulk modulus yet measured. This result provides impetus for a continued search for superhard materials, including transition metal carbides, nitrides, borides, and oxides.

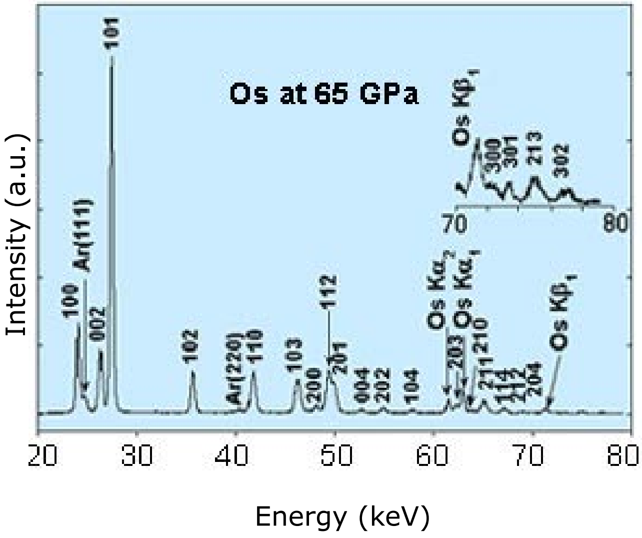


Figure 2. *Energy-dispersive x-ray diffraction pattern (EDX) of osmium at 65 gigapascals. Inset shows expanded view from 70 to 80 keV. The EDX were measured at the X17C beamline of NSLS, using unfocused white x-rays from the superconducting wiggler beam.*

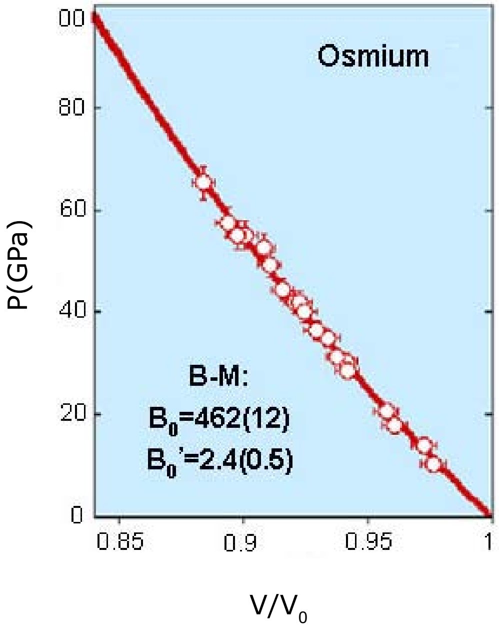


Figure 3. *Volume compression data for osmium, fit with the Birch-Murnaghan equation of state, yielding the bulk modulus B_0 and its first pressure derivative of B_0'.*

BEAMLINE
X19A

PUBLICATION
S. Beauchemin, D. Hesterberg, and M. Beauchemin, "Principal Component Analysis Approach for Modeling Sulfur K-XANES Spectra of Humic Acids," *Soil Sci. Soc. Am. J.*, **66**, 83-91 (2002).

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Modeling Sulfur K-XANES Spectra of Humic Acids With the Principal Component Analysis Approach

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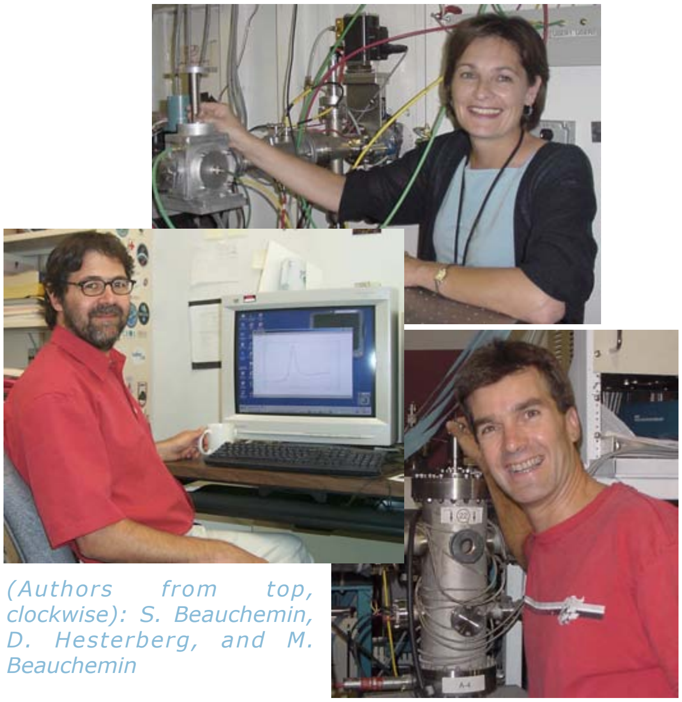
Characterization of the chemical species of trace elements in soils, sediments, and other geochemical systems is essential for predicting their solubility and potential mobility in the environment. To determine metal speciation, scientists usually model x-ray absorption near edge structure (XANES) spectra by using standards of well-defined chemical species. But when scientists deal with complex matrices such as soil– in which many different chemical forms of the element may co-exist – other modeling techniques called principal component analysis (PCA) and target analysis, might be more convenient to use. The PCA was successfully applied to sulfur K-XANES spectra of six humic acid samples.

X-ray absorption near edge structure (XANES) spectra from unknown soil samples are usually modeled using techniques called spectral deconvolution and/or least-squares linear combination fitting (LCF). Both approaches rely on *a priori* information. Spectral deconvolution is based on an assumption about the shape of the band, while least-squares LCF involves fitting pure standard species to resolve an unknown mixture.

We tested a different modeling technique, called principal component analysis (PCA), to analyze soil mixtures. This technique considers the statistical variance within an experimental data set composed of a group of unknown samples. The data set is first redefined in terms of a reduced number of independent sources of variability, called components. These components have no chemical or physical meaning.

A subsequent analysis, the target transformation, is used to test if suspected known species are part of the structural variation of the sample set. Based on the results from target analysis, abstract matrices obtained in the first step are transformed into chemically meaningful matrices.

A major advantage of the PCA approach is that no *a priori* assumption is needed regarding the shape of the band. Also, suspected species can be evaluated individually without *a priori* knowledge of other species present in the sample.



(Authors from top, clockwise): S. Beauchemin, D. Hesterberg, and M. Beauchemin

PCA coupled with target transformation was used to model sulfur K-XANES spectra of six soil humic acid samples (**Figure 1**) using the spectra of seven chemical compounds as sulfur standards: sodium sulfate, chitin sulfate, cysteic acid, benzyl sulfoxide, benzyl disulfide, elemental sulfur, and methionine. The objective was to compare the results from PCA approach to those obtained from least-squares LCF.

PCA identified three main components. As illustrated in **Figure 2**, target analysis showed how closely the predicted targets fitted the suspected known species. Chitin sulfate was a better sulfate species than sodium sulfate to explain our experimental data. Sodium sulfate, cysteic acid and benzyl disulfide were marginal candidates, which means that the nature of the real species in the spectra of humic acids may be slightly different. Other standards were rejected. Therefore, we retained chitin sulfate, cysteic acid, and benzyl disulfide to derive the relative proportion of each selected standard species in each sample.

The PCA approach provides a statistical basis for (i) gaining insight into the identification of chemical species in a multi-component sample, and (ii) defining the number of individual species to include in the spectral fitting. Therefore, it is a valuable complementary tool for band resolution or LCF as it helps to avoid an excessive number of bands or standards in the fit.

Figure 1. *Soil humic acids were extracted from a coastal salt marsh in eastern North Carolina. Soils in salt marshes such as this one near Beaufort, NC, contain reduced organic sulfide that has a high binding affinity for mercury and perhaps other trace elements. (Photo courtesy of Stephen W. Broome, NC State.)*

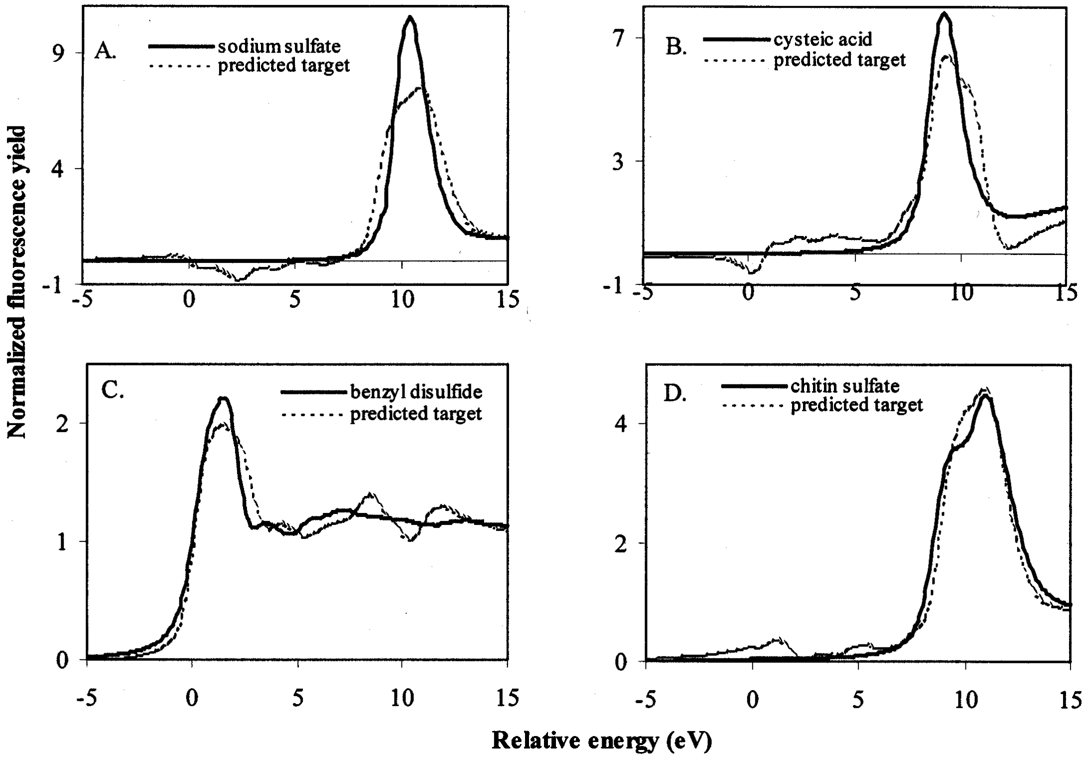


Figure 2. *Predicted targets obtained through target transformation of the sulfur K-XANES spectra for (A) sodium sulfate, (B) cysteic acid, (C) benzyl disulfide, and (D) chitin sulfate compared with the normalized K-XANES spectra for these species. The energy scale is relative to S(0) K-edge at 2472 eV.*

BEAMLINE
X11A

PUBLICATION
K.G. Scheckel and J.A. Ryan, "Effects of Aging and pH on Dissolution Kinetics and Stability of Chloropyromorphite," *Environ. Sci. Technol.* **36**, 2198 –2204 (2002).

FOR MORE INFORMATION
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Influence of Aging and pH on Dissolution Kinetics and Stability of Pyromorphite

K.G. Scheckel and J.A. Ryan

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Immobilization of metal contaminants in soil shows great potential as a cost-effective and environmentally friendly remediation technique. Lead-contaminated soils are typically removed from site and replaced with clean soil at great costs. Innovative technologies for sequestering lead in situ have been proposed and studied. One of these methods, called immobilization-remediation, seeks to both sequester most of the contaminant and retain the metal for the long-term. Scientists at the Environmental Protection Agency's Office of Research and Development in Cincinnati, Ohio, have examined how the most stable lead mineral, called chloropyromorphite, forms and dissolves over time, thus providing a better understanding of lead sequestration in soil.

Previous research has shown that adding phosphate materials to lead-contaminated soils leads to an environmentally stable and biologically inert lead mineral called chloropyromorphite ($Pb_5(PO_4)_3Cl$). Because chloropyromorphite is the most stable lead mineral known, other solid-phase lead species should be transformed to chloropyromorphite by a dissolution-precipitation mechanism to immobilize soluble soil lead *in situ* by simply adding phosphate to the soil. But the long-term fate of immobilized lead as precipitated chloropyromorphite is not fully understood.

We investigated the kinetics of dissolution and crystallization dynamics of synthetic chloropyromorphites with increasing aging times ranging from one hour to one year, to understand better what may be happening in the field setting. The chloropyromorphites were dissolved in a solution containing not only phosphoric acid, but also nitric acid, thus simulating environmental conditions one may find in areas ranging from acid mine drainage to agricultural soil settings.

The objectives of this research were to understand the effect of aging time on the stability of chloropyromorphite dissolved in phosphoric and nitric acid, to examine physical and chemical alterations of the chloropyromorphite samples, and to model the kinetic data collected from the dissolution experiments. Aging of the samples prior to dissolution was investigated by using x-ray absorption fine structure (XAFS) and x-ray diffraction (XRD) spectroscopies, and the stability of the chloropyromorphite material with temperature was studied with high-resolution thermogravimetric analysis (HRGTA).


Kirk Scheckel

Figure 1 shows data for XAFS, XRD, and HRTGA analyses of aged chloropyromorphite prior to dissolution. The XAFS data (**Figure 1a**, solid line), collected at beamline X11A of the National Synchrotron Light Source (NSLS), show relatively little difference between the one-hour and one-year samples.

These same chloropyromorphite samples were also examined by XRD (**Figure 1b**) and, again, do not suggest any alteration in the chemistry or crystallinity of the chloropyromorphite phases as aging time increases. But HRTGA showed that aging of the chloropyromorphite crystals did change with increasing residence time (**Figure 1c**), suggesting, perhaps, an increase in the crystals' Ostwald ripening, in which small crystals, more soluble than large ones, dissolve and re-precipitate onto larger particles.

The dissolution experiments conducted for this research were designed to observe and compare data as influenced by aging time, pH (potential Hydrogen) of the dissolution agent (the pH describes the hydrogen-ion activity of the solution, and determines if the solution is acid, basic or neutral), and dissolution method (stirred flow or mixing in batches, instead of continuously, also called batch method).

Regardless of pH or dissolution method, it appeared that the one-hour-aged chloropyromorphite was the most soluble. Aging of chloropyromorphite from one day to one year did not lead to a significant increase in stability, suggesting that crystal aging was accomplished within 24 hours.

We also observed that, as the pH of the nitric acid decreased, the amount of released lead increased. By removing the dissolution reaction products in the stirred-flow experiments, more chloropyromorphite was dissolved in the stirred-flow than in the batch studies, the difference in released lead between these two methods becoming more pronounced as pH increased. So, once chloropyromorphite forms in contaminated soils and sediments, it should remain immobilized and stable in these environments under near-neutral pH.

The potential impact of stabilization remediation methods to safely sequester metal contaminants in the natural environment holds great promise but must be managed carefully and intelligently. The results of this work and of others demonstrate that chloropyromorphite formation can be easily accomplished by the reaction of available lead and phosphorus. Our study suggests that chloropyromorphite's persistence would endure most environmental conditions, thus making lead-immobilization via phosphorus an ideal remediation mechanism.

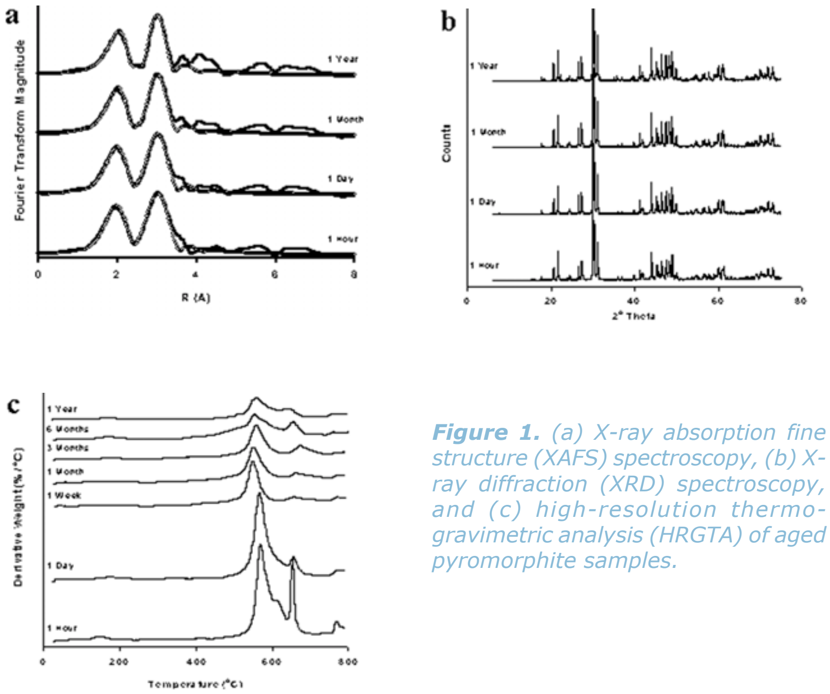

Figure 1. *(a) X-ray absorption fine structure (XAFS) spectroscopy, (b) X-ray diffraction (XRD) spectroscopy, and (c) high-resolution thermogravimetric analysis (HRGTA) of aged pyromorphite samples.*

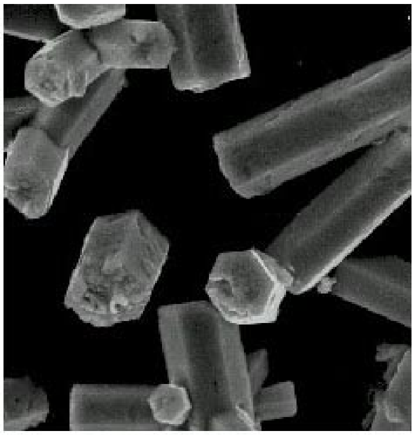

Figure 2. *Crystals of chloropyromorphite.*

BEAMLINE
X11A

PUBLICATION
A. Voegelin et al., "Slow Formation and Dissolution of Zn Precipitates in soil: A Combined Column-transport and XAFS Study", *Environ. Sci. Technol.* **36**, 3749 (2002).

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Slow Precipitation and Dissolution of Solids Affects the Mobility of Heavy Metals in Soils

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What happens if soils are contaminated with heavy metals by agriculture, mining, or industry? Will the metals slowly leach into groundwater and pollute our drinking water resources? Will they be taken up by plants and finally end up in our diet? Environmental soil chemists are trying to answer these questions by determining the chemical forms, or species, of heavy metals in soils. Scientists at the Swiss Federal Institute of Technology in Zurich, Switzerland, have combined x-ray absorption fine-structure (XAFS) spectroscopy with a chromatographic soil column technique to determine the species and mobility of heavy metals in a typical, slightly acidic soil. Their results suggest that some metals (such as zinc) were relatively tightly bound by forming mineral precipitates, while others (such as cadmium) remain highly mobile.

Soil, or "dirt," as some people call it, is made up of an extremely complex mixture of minerals and natural organic substances. So, trying to understand how contaminants behave in such a complex environment might seem daunting, but soil chemists are currently making rapid progress by combining field observations and controlled laboratory experiments with modern spectroscopic techniques.

Our research team has combined two techniques, a chromatographic soil column technique and X-ray absorption fine structure spectroscopy (XAFS) to investigate the chemical behavior of zinc and other transition metals in a slightly acidic loamy soil, at realistically low metal concentrations.

Previous experiments have suggested that certain transition metals including cobalt, nickel and zinc can form structures called layered double hydroxide (LDH) when these metals chemically react with clay minerals in neutral soils (neither acidic, nor basic) and relatively high metal concentrations. But these experiments have not shown clearly whether such structures can form in soils under more acidic conditions and at realistically low concentrations of metals from an infiltrating solution.

The objectives of our study were: (1) to investigate the rapid and slow absorption of cobalt, nickel, zinc and cadmium in a slightly acidic loamy soil; (2) to examine the subsequent release of metals due to cation exchange and soil acidification; and (3) to assess the predominant absorption mechanisms of zinc in the soil.

One of the key techniques that we used is a chromatographic soil column technique. The basic idea is to pack a column with soil, and then leach it over a long time period with a solution containing small concentrations of cobalt, nickel, zinc and cadmium. When solid precipitates form slowly, the products accumulate over time in the soil column. At the end of this loading period, we can identify the precipitates and study how they redissolve by following changes in solution chemistry.

Solid precipitates of zinc were identified by using a technique called x-ray absorption fine-structure (XAFS) spectroscopy at beamline X11A of the National Synchrotron Light Source. In this technique, x-rays generated at the NSLS

Some of the Authors of the study (from left to right): Andreas C. Scheinost, Andreas Voegelin (lead author), Kurt Barmettler, and Ruben Kretzschmar.

are projected toward the precipitate, in which some of the photons of light are absorbed. By measuring the ratio of absorbed photons versus the total incident photons as a function of the energy of the incident photons, we can determine the nature of the chemical elements present in the precipitate.

Figure 1 shows three spectra, called radial structure functions, obtained by applying the XAFS technique to zinc-aluminum-LDH synthesized in the laboratory, soil from the column experiment after 41 days of loading, and the same soil with zinc adsorbed in a geometry called inner-sphere surface complex (**Figure 1**, bottom right).

By analyzing the spectra, we suggest that a solid phase similar to zinc-aluminum-LDH has formed in the soil column. While zinc-aluminum-LDH and zinc-phyllosilicate (a phyllosilicate is a type of silicate mineral) cannot be distinguished easily based on the local structure, our leaching experiments demonstrate that the precipitates formed in the soil are readily re-dissolved upon acidification. Because zinc-phyllosilicates are expected to be much less soluble, formation of zinc-aluminum-LDH seems most likely.

When different transition metals were compared, we found that the importance of precipitation of solid phases in the slightly acidic soil decreased in the order zinc-nickel-cobalt-cadmium, where zinc is the most tightly bound and cadmium is the most mobile. Our study has demonstrated that combining well-controlled laboratory studies with XAFS spectroscopy is a promising approach to investigate the behavior of metal contaminants in soils.

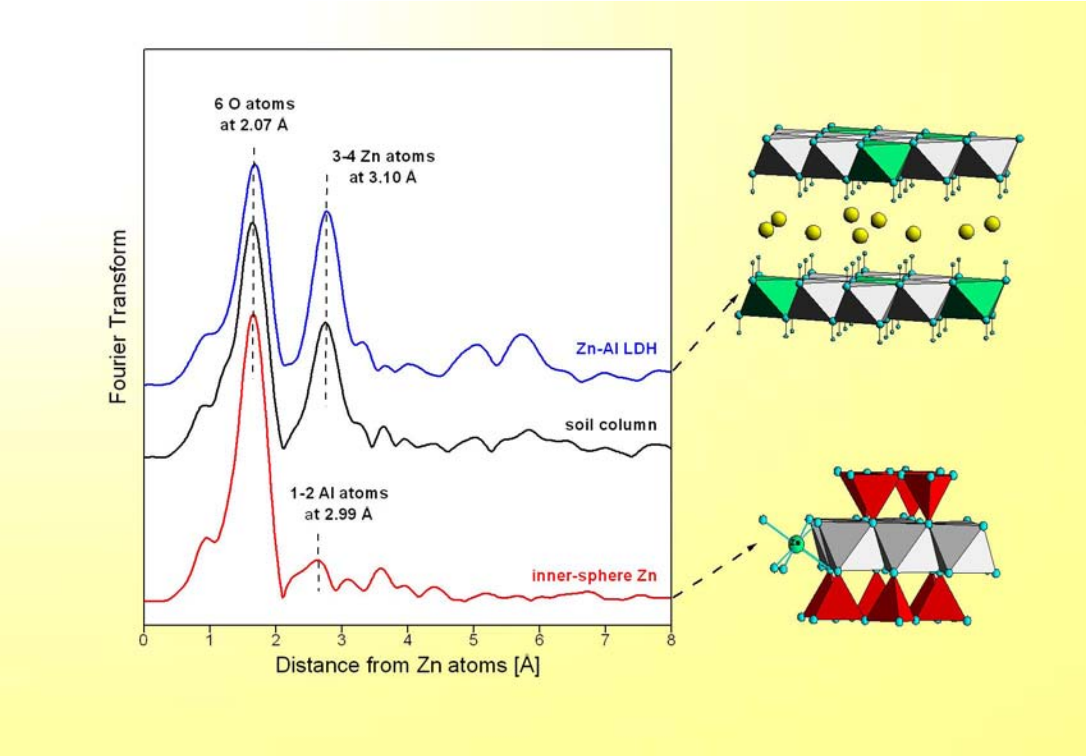
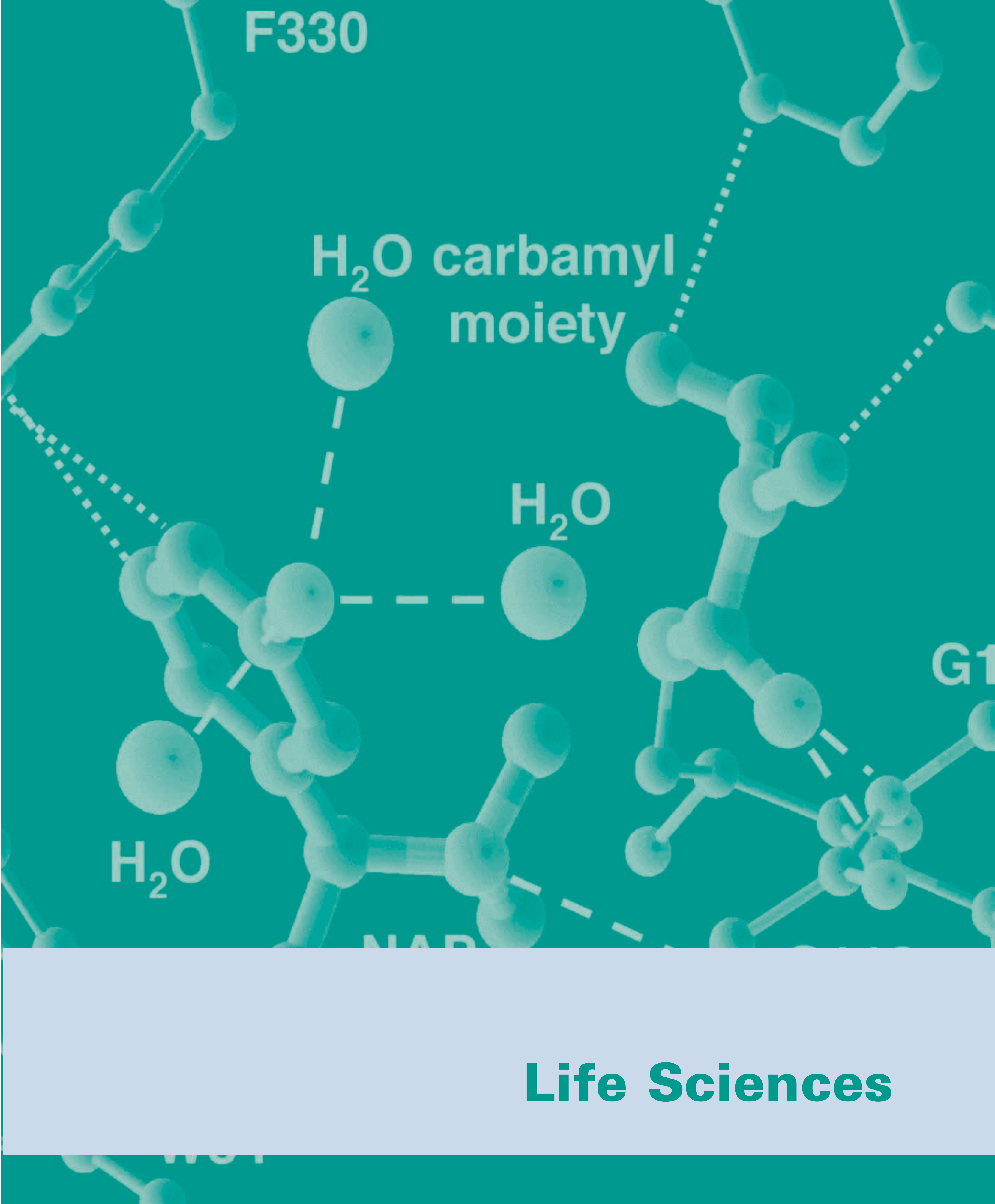


Figure 1. Identification of zinc species with x-ray absorption fine structure (XAFS) spectroscopy. After a slightly acidic soil column has reacted with a dilute zinc solution, zinc atoms are coordinated to six oxygen atoms at a distance of 2.07 angstroms (one angstrom is one tenth of a billionth of a meter), and to three or four zinc atoms at 3.10 angstroms. This local structure (middle curve) is similar to the solid phase zinc-aluminum-layered double hydroxide (Zn-Al-LDH), but is unlike the zinc inner-sphere surface complex. The structural drawings on the right side depict the two binding mechanisms (green: zinc octahedral; gray: aluminum octahedral; red: silicon tetrahedral; yellow: aqueous cations).



Life Sciences

Enzyme Studies May Lead to New Antiviral Agents

Funded by DOE and the National Institutes of Health, three new enzyme studies at BNL have yielded a new strategy for blocking infection by human adenovirus. The findings, reported in the October, November, and December, 2001 issues of the journal, *Biochemistry*, have already been used to design novel antiviral drugs.

Adenoviruses cause a number of acute infections, including respiratory and gastrointestinal infections, and conjunctivitis. In patients with compromised immune systems, such as those infected with human immunodeficiency virus (HIV), an opportunistic adenovirus infection, is frequently deadly.

“Our new antiviral drugs are expected not only to inhibit adenovirus, but also, possibly, to be effective against other organisms that use the same enzyme - including *Chlamydia*, one of the most prevalent sexually transmitted bacteria, and *Yersinia pestis*, the organism that causes the black plague,” said Walter Mangel, Biology Department, the lead scientist on the studies.

Infection Process

During infection, these viruses make an enzyme called a protease, which cleaves or degrades other proteins. The protease is used by the virus to complete the maturation of newly synthesized virus particles.

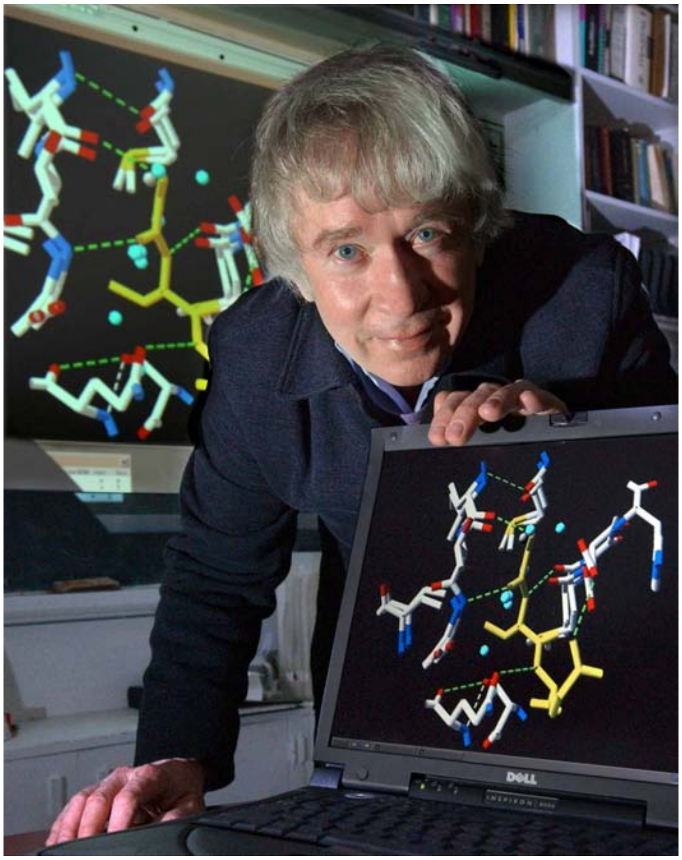
To explain this process, Mangel uses the example of building a cathedral around internal scaffolding. Once the cathedral is in place, the last step is to remove the scaffolding. “Similarly,” says Mangel, “adenovirus particles are built with scaffolding proteins inside. Once the virus particle is formed, the protease becomes activated and cleaves the scaffolding to render the virus particle infectious.”

The three recent studies at BNL reveal that the protease is initially synthesized in an inactive form. The inactive enzyme binds to the viral DNA to become partially activated.

The partially activated enzyme then cleaves out a cofactor, which is a protein fragment that binds to the protease to activate it further. The fully active complex of enzyme and cofactor then moves along the viral DNA, cleaving the scaffolding proteins.

“Such activation of a protease by DNA has never been seen before,” Mangel said. And this presented a problem.

“Two other laboratories repeatedly claimed in the literature that they saw no stimulation of the enzyme by DNA,” Mangel continued. “So, in one of the three papers, we not only presented definitive evidence that the enzyme interacted with DNA, but also showed why the two other groups had not seen stimulation of the enzyme by DNA. Essentially, they did not use the correct conditions. We hope that, with the publication of these papers, this controversy is now resolved.”



Walter Mangel

Next Step: To Design Drugs

"These studies suggest that drugs that bind to the active site of the enzyme, which is the part involved in cleaving proteins, the cofactor binding site, or the DNA binding site should block the enzyme's action and serve as effective antiviral agents," Mangel said.

To design drugs able to bind to and block these sites, the scientists first had to characterize the molecular structures. The active site of the enzyme had been previously characterized by William McGrath, a postdoc in Mangel's team, using an intense beam of x-rays available at the NSLS. The pattern of x-rays bouncing off the atoms reveals the three-dimensional molecular structure.

In the current studies, Stony Brook University graduate student Mary Lynn Baniecki characterized the binding of the cofactor to the protease, identifying which parts bind to the enzyme and which parts stimulate the enzyme's activity. McGrath and Baniecki then deciphered how the protease binds to the DNA.

Among the other coauthors of the study, Mangel said, are three undergraduate students, David Green, Caroline Li, and Sarah McWhirter, who were participants in the DOE Energy Research Undergraduate Laboratory Fellowships program managed at BNL by the Office of Educational Programs.

New, Three-Pronged Therapy

Based on the findings, Mangel has proposed a new form of antiviral therapy using three different drugs against these three target sites - the active site, the cofactor binding site, and the DNA binding site - on the same virus-coded protein. This three-pronged approach may overcome one of the biggest challenges in antiviral therapy - the spontaneous evolution of drug-resistant strains.

As Mangel explains the idea, a mutation conferring drug resistance at one site may alter the physiological functions at the other two sites because the three sites are interdependent, thereby making drug resistance much less likely to arise.

"The adenovirus protease may be a good model system within which to test the efficacy of this form of combination therapy," Mangel said. Already, his team has developed two new drugs, one that binds reversibly and another irreversibly to the active site of the protease. These drugs will soon be tested as antiviral agents by the National Institutes of Health.

- Karen McNulty Walsh

[Editor's note: Reprinted with permission from the BNL Bulletin - January 18, 2002.]

A graphic representation of the structure of the adenovirus protease. The active site is in the groove at the top where the "ball-and-stick" figures are located. The cofactor is the strand in the center at the bottom. The DNA binding sites are identified with "plus (+)" signs.

Anti-Alzheimer's Drug Mechanism Revealed With X-Rays

Using x-rays produced by the NSLS, a team of scientists has gained new insight into the effects of a newly approved drug, called rivastigmine, in the treatment of Alzheimer's disease – a debilitating brain disease causing memory loss and other cognitive deficits in about 10 percent of the elderly. The new results, which may provide important information for generating improved drugs for this as-yet incurable neurodegenerative disease, were published in the March 19, 2002 issue of the American Chemical Society journal *Biochemistry.*

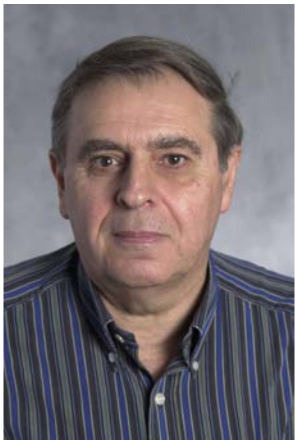
"We were very surprised by our results," says Joel Sussman, a structural biologist at the Weizmann Institute of Science in Rehovot, Israel, and the lead author of the study. "They show that we can safely treat Alzheimer's disease with much lower quantities of rivastigmine, thus minimizing unwanted adverse effects."

Though the drug is currently available under the trade name Exelon, its mechanism of action at the atomic level had not been studied until now. So, the team of scientists, composed of Sussman's team and scientists from Novartis, a pharmaceutical company based in Basel, Switzerland, decided to take a close look at how the drug helps to slow the memory loss of Alzheimer's patients.

One of the main pathological phenomena in Alzheimer's disease is the deterioration of nerve cells releasing acetylcholine, a chemical that helps to carry messages among brain cells. The inadequate supply of acetylcholine in Alzheimer's patients is compounded by the action of an enzyme called acetylcholinesterase (AChE), which breaks down acetylcholine in the body at the rapid rate of 20,000 molecules per second.

The desired effect of potential Alzheimer's treatments, such as rivastigmine, is to inhibit AChE long enough to offset the absence of acetylcholine. But rivastigmine and other anti-Alzheimer's drugs have side effects and may merely slow deterioration rather than halt it.
To look at the action of the drug over time, Israel Silman, a neurochemist at the Weizmann Institute of Science and a co-investigator on the study, together with Pazit Bar-On, a joint graduate student with Sussman and Silman, tested the drug on various types of AChE, extracted from an electric ray, the fruit fly, and human beings. "We wanted to see how long it takes AChEs to go back to normal, or become 'reactivated,' after being inhibited by the drug," Silman says.


Joel Sussman


Israel Silman

The scientists were very surprised to notice an "extremely low reactivation" of the AChEs from all three organisms. "Inhibition of AChE by rivastigmine appears to be almost irreversible, with little reactivation over a period of days," Silman says.

To explain what happens at the molecular level, the scientists took "snapshots" of rivastigmine while it was binding to AChE, using a method called x-ray crystallography. They projected x-rays produced at the NSLS on crystals of rivastigmine combined with AChE. They then determined the structure of the complex rivastigmine-AChE by looking at how the x-rays scattered off the crystal. By reconstructing the positions of these scattered x-rays, the scientists established a molecular map that revealed the locations of all the atoms of AChE and rivastigmine in three dimensions.

"When we looked at this map, things became clearer," Sussman says. "We had suspected that rivastigmine was binding very tightly to AChE, preventing surrounding fluid – mainly water – from breaking this bond quickly, as it usually does."

By looking closely at the AChE "active site" – the part to which rivastigmine binds to AChE – Sussman and his colleagues noticed that rivastigmine was broken in two, each part being ensconced comfortably in the active site (**Figure 1**). The scientists also precisely determined how each part was bound to the surrounding AChE atoms and moved other AChE atoms, which slowed down reactivation of AChE (**Figure 2**).

"The x-ray molecular maps allow us to see how every atom of rivastigmine interacts with the atoms of AChE's active site," Silman says. "This information will be important in designing new chemicals that will target specific atomic sites in AChE, possibly leading to better drugs that last longer and have less undesirable effects on Alzheimer's patients."

"I am very excited by the perspectives offered by these results," Sussman says. "By fine-tuning the properties of anti-Alzheimer's drugs or their targets at the molecular level, we can truly hope to find a cure for Alzheimer's disease in the future."

BEAMLINE
X12C

PUBLICATION
P. Bar-On, et al., "Kinetic and Structural Studies on the Interaction of Cholinesterases with the Anti-Alzheimer Drug Rivastigmine," *Biochemistry*, **41**, 3555-3564 (2002).

-Patrice Pages

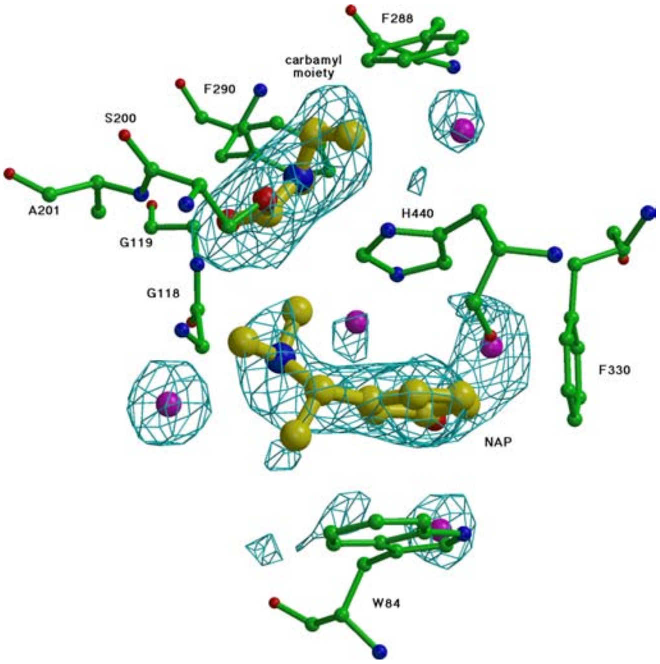


***Figure 1**. Close-up view of how the drug rivastigmine binds to the active site of the acetylcholinesterase (AChE) from the electric ray* Torpedo californica*. After binding to the active site of AChE, the drug is broken into two parts, called carbamyl moiety and NAP. Rivastigmine is rendered as a ball-and-stick model, with carbon atoms colored yellow, oxygen atoms colored red, and nitrogen atoms colored blue. Selected key molecules in the vicinity of rivastigmine are also rendered in ball-and-stick format, with carbon atoms colored green.*

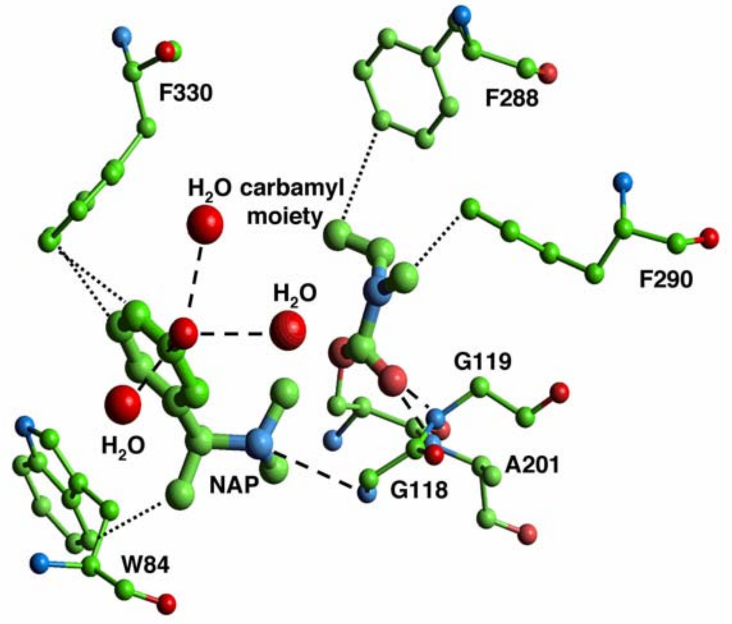


***Figure 2**. Active site of acetylcholinesterase (AChE) from the electric ray* Torpedo californica *after AChE is inhibited by the drug rivastigmine. Both parts of rivastigmine, the carbamyl moiety and NAP, are depicted with larger spheres and thicker lines for emphasis. Amino acids within the active site of AChE that may interact with the drug are shown. The carbamyl portion of rivastigmine is positioned to make two H-bonds (dashed lines) with the amide nitrogens of A201 and G119, as well as non-bonded contacts (dotted lines) with F288 and F290. NAP, the second part of rivastigmine, makes H-bonds with three water molecules (large red spheres) and the amide nitrogen of G118, as well as non-bonding contacts (dotted lines) with W84 and F330. (A, F and G represent the amino acids alanine, phenylalanine and glycine, respectively).*

Scientists at NSLS Discover how Papillomavirus ‘unzips’ DNA

Finding may lead to drugs to prevent sexually transmitted disease and cervical cancer

Infection with the human papillomavirus (HPV) is the most common sexually transmitted disease in the United States According to the Centers for Disease Control and Prevention in Atlanta, Georgia, an estimated 20 million Americans are currently infected – but the vast majority does not know it.

Though HPV sometimes causes genital warts, in most cases, it infects people without causing visible symptoms. Women with persistent infections from certain types of HPV are at risk for cervical cancer, as 99 percent of cervical cancers around the world are associated with HPV infection.

Preventing papillomavirus from multiplying is one way of stopping the infection. Toward that goal, a team of scientists from Cold Spring Harbor Laboratory (CSHL) in New York, working at the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory, have gained new insight into how papillomavirus – in this case, cow, or bovine papillomavirus, commonly used as a model system – starts to multiply, causing infection. This new understanding could be used to design drugs to stop HPV infection, which is of particular significance since no cure or vaccine are currently available, although vaccine development is underway.

“We know very little about how papillomavirus replicates,” says biologist Leemor Joshua-Tor, the Cold Spring Harbor team leader. “So we decided to look at the molecular details of how the replication mechanism is initiated, with the aim of helping to design drugs that act like monkey wrenches in the replication process.”

Authors of the study (from left to right): Leemor Joshua-Tor, Arne Stenlund and Eric Enemark.

Like all viruses, a papillomavirus is an infectious agent that uses the cells it infects to reproduce itself. Replication of HPV does not kill the host cells, but can make them cancerous.

The infection process starts as follows: The papillomavirus first inserts its DNA – a double-stranded helix containing the virus’s genetic information – into the host cell. The virus hijacks the protein production machinery of the host cell to produce a viral protein called E1. By attaching to the viral DNA, E1 proteins can initiate the DNA replication process, so that more viruses can be formed, and later multiply further.

“The DNA double helix can be replicated only if it is ‘unzipped,’ which allows proteins called DNA polymerases to make copies of each strand,” Joshua-Tor explains. “The E1 protein is known to initiate the ‘unzipping’ process, but how it does it is not very well understood.”

To look carefully at how E1 proteins attach to viral DNA, Joshua-Tor and her postdoctoral associate, Eric Enemark, in collaboration with Arne Stenlund, a renowned papillomavirus expert at CSHL, grew crystals of E1 and papillomavirus DNA at two different stages of the attachment process, in which either two or four E1 proteins bind to DNA.

The researchers then used a technique called x-ray crystallography to determine the positions of the atoms making up the E1 proteins and DNA. X-rays produced by the NSLS were projected toward the crystals, and the positions of the atoms were determined by looking at how the x-rays scattered off the crystal.

To their surprise, Joshua-Tor and her colleagues observed that E1 uses two separate modules, one shaped like a loop and the other

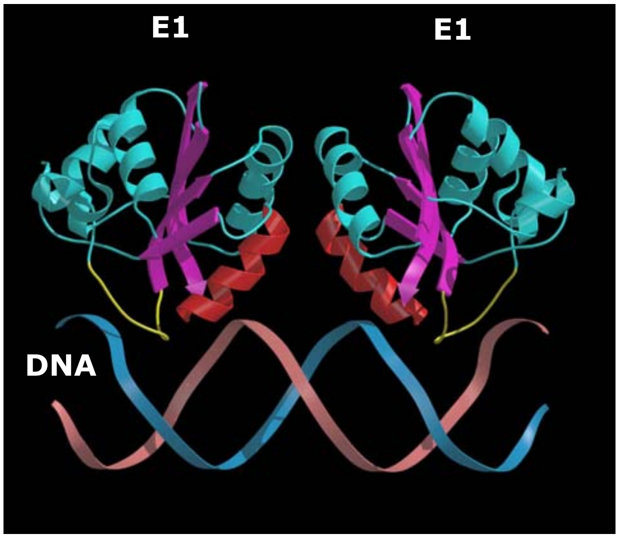


__Figure 1__. Crystal structure of two E1 proteins binding to papillomavirus DNA. Each E1 protein exhibits two modules: a loop (yellow) and a helix (red), each binding to a separate DNA strand, the loop binding more tightly than the helix to the DNA strands.

__Figure 2__. Crystal structure of four E1 proteins binding to papillomavirus DNA. The loops (yellow) of the two proteins on the right (purple and green) bind one DNA strand (pink), while the loops of the two proteins on the left (red and blue) bind the other DNA strand (blue).

like a helix, to bind DNA, each one binding to a different DNA strand (**Figure 1**). "This is very unusual," Joshua-Tor says. "We expected that both modules would bind the two strands simultaneously." The scientists also noticed that the loop bind more tightly than the helix, giving loops a larger role in E1-DNA binding than helices.

When two E1 proteins attach the DNA, Joshua-Tor and her collaborators observed that the loops bind different strands (**Figure 1**). When four proteins bind to DNA, they form two pairs facing each other, with proteins in each pair binding the same DNA strand as the ones on the opposite side (**Figure 2**).

These results suggested a mechanism by which the double-stranded DNA might 'unzip' (**Figure 3**). "We already know that, ultimately, the unzipping process involves two bundles of six E1 proteins each, called hexamers, each 'unzipping' the DNA in opposite directions," Joshua-Tor says. "So, we think that the initial assembly of the two hexamers from the four proteins shown in our structure is what causes the strands to separate by forming around the single strands."

Joshua-Tor and her colleagues suggest that the four proteins separate into two pairs, each recruiting four additional E1 proteins, thus creating two hexamers that would move in opposite directions (**Figure 3**). Each hexamer would encircle either strand, and act like a little propeller that rotates around the strand, thus 'unzipping' it from its partner DNA strand along the way.

"If this is the way these proteins operate, it is pretty clever," Joshua-

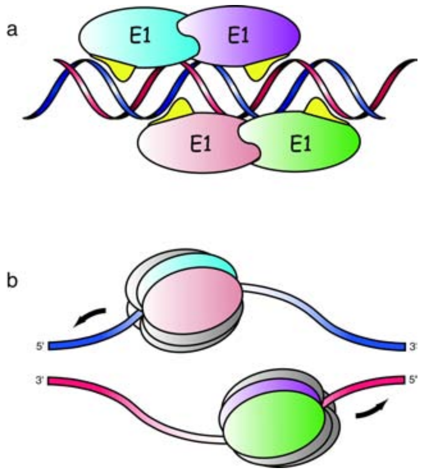


__Figure 3__. Schematic representation of how hexamers are formed and DNA 'unzipping' process is initiated. (a) When four E1 proteins bind to the double stranded DNA (red and blue), they link DNA strands with a separate loop (yellow) that acts like a clip and allows E1 to hold tight onto the strand. Note that two proteins facing each other hold onto the same strands: The blue and red E1 proteins bind to the blue DNA strand, while the purple and green E1 proteins bind to the red DNA strand. (b) The E1 proteins that face each other (blue and red on one side, purple and green on the other side) attach to each other, and recruit four more E1 proteins to form a hexamer on each side of the DNA. The hexamers act like little propellers moving in opposite directions and 'unzipping' each DNA strand from its partner.

Tor says. "This is the first time that it has been found that the two individual DNA strands bind to two separate protein modules prior to the DNA 'unzipping' process."

While Joshua-Tor and her colleagues are still investigating the papillomavirus-induced DNA replication, they are also starting to test compounds that interfere with papillomavirus DNA replication. For example, Anitra Auster, a graduate student, is developing compounds that could interfere with the DNA replication induced by a high-risk type of human papillomavirus that can lead to cervical cancer.

"Understanding these binding mechanisms could significantly improve the treatment of this sexually transmitted disease," Joshua-Tor says. "We can now design and test drugs aiming to prevent E1 proteins from attaching to the viral DNA, which is one of the first steps to making much-needed antivirals against HPV infections and HPV-induced cervical cancer."

BEAMLINE
X26C

PUBLICATION
E. Enemark, A. Stenlund, and L. Joshua-Tor, "Crystal Structures of Two Intermediates in the Assembly of the Papillomavirus Replication Initiation Complex," *The EMBO Journal*, **21**, 6, 1487-1496 (2002).

-Patrice Pages

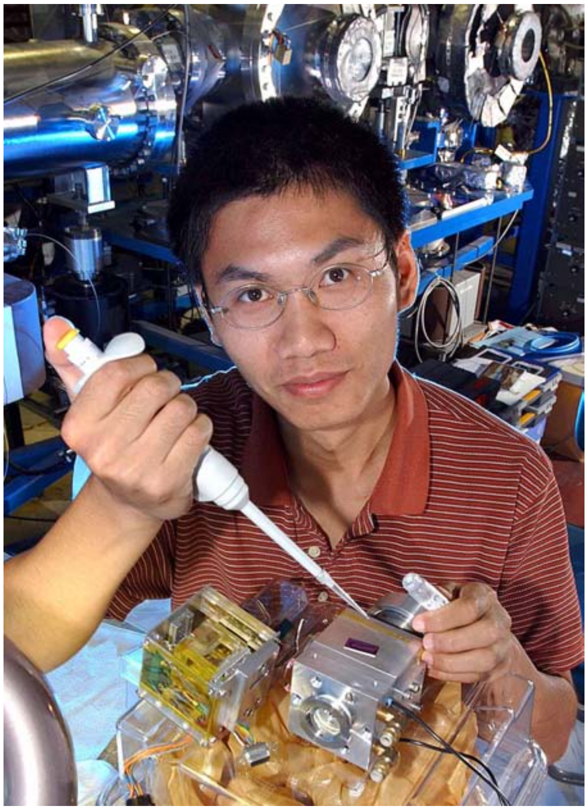
[Editor's note: Reprinted with permission from the BNL Bulletin - November 22, 2002.]

Biological Serendipity: Molecular Details of Cell Membrane Fusion Revealed

For the first time, scientists have observed the molecular details of biological cells fusing together, a fleeting event never before observed at this scale. This research, which could lead to more efficient drug delivery processes and gene therapy techniques, was performed on NSLS beamline X21 and Rice University. The results appear in the September 13, 2002 issue of *Science*.

Cellular membrane fusion is well known to scientists and is one of the most common ways for molecules to enter or exit cells, in processes such as fertilization and viral infection. When two cells fuse together, their membranes come together at one location and create a connection between the cells that allows the exchange of material between them. Eventually, the two membranes form one single, continuous membrane surrounding the contents of both cells.

"We have now confirmed the existence of a temporary structure that occurs during membrane fusion and that has been postulated by scientists for a long time," says Lin Yang, a postdoctoral physicist at the NSLS and the lead author of the study. Yang and coauthor Huey Huang, professor of physics and astronomy at Rice University in Houston, with whom Yang did his doctoral studies, made their observation by serendipity while studying how certain small proteins kill bacteria by digging holes into bacterial membranes.


Lin Yang

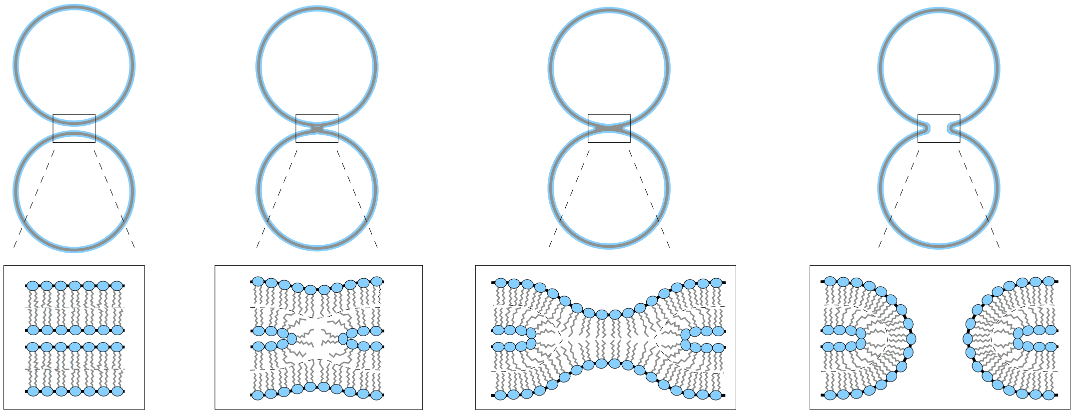
"We were trying to understand how changes in humidity and temperature affect the properties of a certain type of cell membrane," Yang says, "when, amid our results, we observed this structure that nobody had ever seen. That was pretty exciting."

To reveal the structure of the fused cell membranes, the scientists used a method called x-ray diffraction. They first produced small crystals composed of stacks of membranes made of phospholipids. Then, they projected x-rays produced by the NSLS toward the crystals. By looking at how the x-rays scattered off the crystals, Yang and Huang measured a pattern of points with varying intensities, called a diffraction pattern, which represents a map of the atomic structure of the phospholipid layers in the membranes.

"We noticed that by changing the humidity, we could significantly alter the structure of the membrane," Yang says. For certain humidity values, the scientists noticed that, instead of displaying a single line of regularly spaced points, the diffraction pattern revealed many more points at other positions. By studying these diffraction patterns more closely, the scientists realized that these patterns were those of two membranes caught in the act of fusing.

The diffraction pattern showed that, when the two membranes fuse, they form an hourglass-shaped structure called a stalk, confirming theoretical predictions. When the stalk stretches further, it creates a connecting bridge between the membranes. This connection then enlarges, and the two membranes ultimately become one single membrane.

Huang comments that the membrane considered in this study is relatively simple and does not reflect the complexity of more common natural membranes, which are made of phospholipids and proteins. "What is truly exciting is that we now have, for the first time, a model system that can provide clues about how more complicated membranes work," he says.



A simple representation of the fusion of two cells, with close-up views of the changes that the cellular membranes undergo. Each membrane is made of a double layer of phospholipids which intermingle to create a passage between the cells. (Each phospholipid is represented with a head and two tails.)

Understanding cell fusion may be key to preventing viral infection or designing new drug delivery methods, the scientists say. "Understanding the details of membrane fusion may help scientists find the appropriate conditions for preventing viruses such as HIV from fusing to and thereby infecting human cells," Yang says. "This knowledge could also lead to the design of systems in which a drug or a piece of DNA is enclosed in a membrane known to fuse with specific cells in our body, thus facilitating drug delivery or improving gene therapy."

— Patrice Pages

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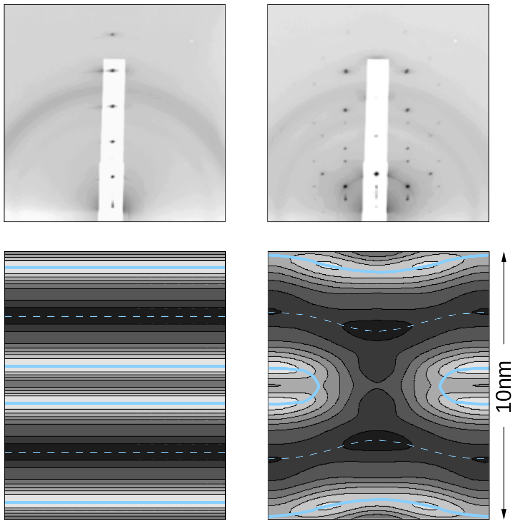


(Top) Diffraction patterns of two cell membranes before (left) and during (right) fusion. (Bottom) Three-dimensional representation of the membranes before (left) and during (right) fusion.

Role of Cancer Protein Identified by Princeton, LBNL Teams at NSLS

Scientists working at the NSLS beamline X25 have unveiled the details of an important cancer protein. Though the protein, called Ski - for Sloan Kettering Institute, where it was identified in the early 1980s - is known to trigger tumor growth, how it does this is still not well understood. The new results, which are reported in the November 1, 2002 issue of *Cell*, shed light on this process and may provide ways to design new anticancer drugs.

"We now have a very important clue as to how Ski interferes with key proteins that prevent cells from becoming cancerous," says Yigong Shi, a molecular biologist at Princeton University. Shi leads one of the two teams, one from Princeton and one from Lawrence Berkeley National Laboratory (LBNL), that conducted the study, which is supported by the National Institutes of Health and the Searle Scholar and the Rita Allen Foundations.

Yigong Shi

"Understanding how to stop Ski from disrupting the normal function of cells will probably be key to developing new anticancer drugs," continued Shi.

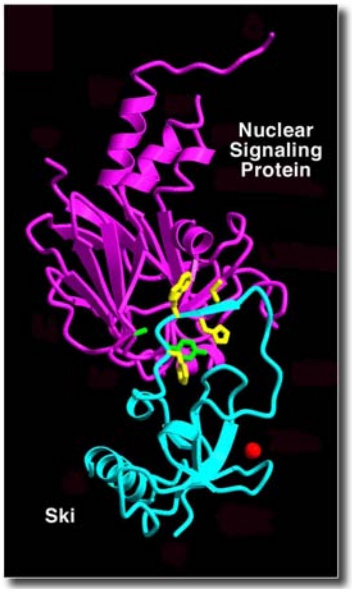
Ski prevents a protein called transforming growth factor-beta (TGF-β) from safeguarding cells against excessive growth.

"TGF-β acts like a molecular traffic light, ordering certain cells to slow down and stop dividing," Shi says. "When TGF-β is blocked, for example by Ski, cells manage to speed through this checkpoint, triggering runaway cellular growth that eventually results in cancerous tumors."

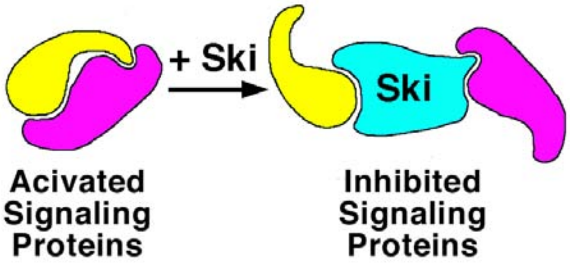
TGF-β cannot enter cells, so it transmits its signal inside the cell by attaching to receptor proteins on the cell's outer surface. The signal generated by this interaction is carried across the cell membrane to proteins inside the cell.

Some of these signaling proteins are triggered inside the cell cytoplasm and later bind to other proteins inside the nucleus. The combination of both types of signaling proteins activates genes necessary for the normal functioning of the cell.

Ski, which is already present in the human body, disrupts the signaling proteins when it is either overexpressed or introduced by a virus inside the body. The new



Overall structure of the complex of Ski and a nuclear signaling protein.



Schematic diagram of a proposed mechanism for the Ski-mediated repression of TGF-ß signaling. By simultaneously binding to the cytoplasmic (yellow) and nuclear (purple) signaling proteins, Ski prevents the two signaling proteins from binding to each other, thus suppressing the action of TGF-ß.

study focused on the first of these two possible processes.

"Scientists have previously shown that Ski disrupts normal cell functioning by directly disrupting the expression of genes inside the cell's nucleus," Shi says. "But nobody has ever investigated whether Ski could disrupt the signaling proteins that activate the genes."

The Princeton team looked at the molecular details of a complex made of Ski and nuclear signaling proteins by using x-rays generated at the NSLS.

The researchers saw that, as they had suspected, Ski disrupts the cytoplasmic signaling proteins, so that when Ski binds to the nuclear signaling proteins, the cytoplasmic signaling proteins cannot attach to their nuclear counterparts. "This binding process is probably one of the major ways in which Ski disrupts the signaling proteins and, thus, suppress the action of TGF-β," Shi says.

The LBNL team performed various biochemical tests that confirmed these results by also showing that Ski binds to nuclear signaling proteins.

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - November 1, 2002.]

Scientists Uncover Molecular Clues About Origin of Cancer

Scientists at Johns Hopkins University in Baltimore, Maryland, have provided new insight into the inner workings of a member of a family of proteins, called human epidermal growth factor receptors (EGFRs), that are involved in abnormal tissue growth and cancer. This research, which was performed at the NSLS could lead to more efficient drugs aimed at preventing or treating some forms of cancer. The results were reported in the August 23, 2002 issue of *Science.*

Dan Leahy

"Little is known about the structures of the EGFR family members," says Dan Leahy, a biophysicist at the Howard Hughes Medical Institute (HHMI) and the lead author of the study, "yet they are involved in many human cancers, including breast, gastric, colon, and prostate cancers. Our study is a starting point to see how a possible drug might help prevent or cure these cancers."

Leahy and Hyun-Soo Cho, an HHMI postdoctoral student, studied a protein called human epidermal growth factor receptor 3 (HER3), which, like all the other EGFR proteins, is located on the surface of human cells. When HER3 binds to specific types of proteins, called ligands, its shape changes, and HER3 tells the cell to divide and multiply.

Though HER3 is not known to be involved in cancer, the abnormal production of its close relative, called HER2, is associated with particularly aggressive forms of breast cancer. But attempts to study the structure of HER2 have proven unsuccessful so far. Instead, by providing details of the molecular mechanisms of HER3, Leahy and Cho can now unveil some insight into the inner workings of HER2.

"It is usually very difficult to determine the structure of EGFRs," Leahy says. "It took us more than two years to get HER3's structure, and, now that we have it, it might be easier to understand how its relatives work."

HER3 consists of three parts, which are located outside the cell, in the cell membrane, and inside the cell. Leahy and Cho studied the outside part of HER3, called sHER3, which binds to specific ligands and changes its shape during the binding process.

To reveal sHER3's molecular details, the scientists used a method called x-ray diffraction. They first crystallized sHER3, and then bombarded the resulting crystal with x-rays produced by the NSLS. By looking at how the x-rays scattered off the crystal, Leahy and Cho determined the positions of the atoms inside sHER3, thus establishing its three-dimensional structure.

The structure showed that sHER3 is made of four regions, and suggested how ligands attach to sHER3 and induce shape changes. One unexpected aspect of the structure is what the scientists call the "snap" region – two finger-like loops that reach out toward one another and interact (**Figure 1**). Leahy and Cho also noticed that the size of the sHER3 binding site, which is comprised between regions I and III (**Figure 1**), is twice as big as the ligand size when the two finger-like loops interact.

"The 'snap' region seems to prevent a ligand from binding," Leahy says. "So, the two finger-like loops would have to detach from each other to allow a ligand to bind to sHER3, which informs the cell that it can grow and divide.

"If future studies confirm that this is how a ligand binds to sHER3, then it is pretty exciting," he adds. "This means that we can now think of new therapeutic approaches to modulating – preventing or stimulating –ligand-binding to HER3 and other members of the EGFR family."

Leahy and Cho suggest two approaches to developing such modulation. In the first approach, intended to prevent ligand-binding, the scientists propose to design mutant forms of ligands that would only bind to either region I or III, but prevent normal ligands to bind to HER3. As the finger-like loops remain attached to each other, the cell does not receive signals prompting it to grow and divide.

In the second approach, Leahy and Cho suggest to use molecules that would affect the binding between the finger-like loops, by either breaking or reinforcing this binding, and thus favoring or inhibiting ligand-binding to HER3.

"I am very excited by the therapeutic opportunities that can be derived from the knowledge of the three-dimensional structure of this receptor," Leahy says. "This is just the beginning, but I am confident that similar studies will open more possibilities of tailoring appropriate drugs for the treatment of cancer."

BEAMLINE:
X4A and X25

PUBLICATION:
Hyun-Soo Cho and Daniel J. Leahy, "Structure of the Extracellular Region of HER3 Reveals an Interdomain Tether," *Science* **297**, 1330 (2002).

-Patrice Pages

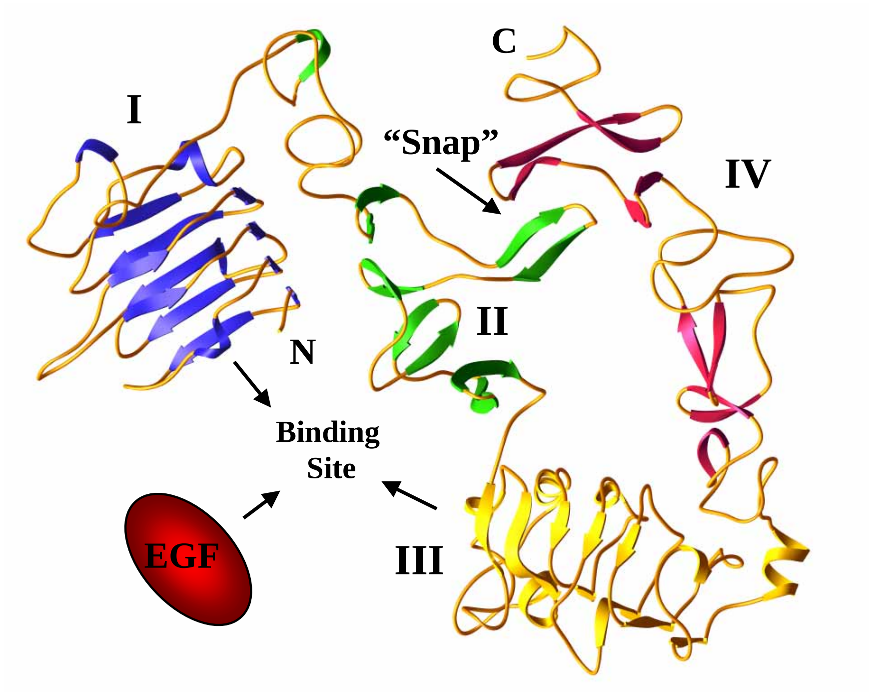


Figure 1. *Ribbon diagram of the human epidermal growth factor 3 (HER3), showing domains I (blue), II (green), III (yellow), and IV (red). Disulfide bonds are shown in purple and gold. The results of Leahy and Cho's study indicate that a ligand (left) may bind to HER3 when the "snap" region opens, which bends domain II to the left and brings domains I and III close enough to "trap" the ligand between them.*

BEAMLINE
X28C

PUBLICATION
G. M. Dhavan, D. M. Crothers, M. R. Chance, M. Brenowitz, "Concerted Binding and Bending of DNA by Escherichia coli Integration Host Factor," *Journal of Molecular Biology*, **315**, 1027-1037 (2002).

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Kinetics of Association Between the Integration Host Factor and DNA in *Escherichia coli*

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Scientists at the Albert Einstein College of Medicine in New York and Yale University in New Haven, Connecticut, have conducted the first analysis by synchrotron footprinting of the fast kinetics of a protein-DNA interaction. The work was conducted at NSLS beamline X28C, and provided a detailed, single base-pair resolution of the exact points of contact of a protein called Integration Host Factor on the DNA surface. The scientists have shown that when protein and DNA samples are mixed together, proteins capable of binding to DNA in a sequence-specific manner yield protections against hydroxyl radical attack along the nucleic acid surface. Two distinct phases are also observed in the association process: an initial "burst" phase, followed by a slower one.

Integration Host Factor (IHF) is a sequence-specific DNA binding protein that bends DNA and recognizes several sites in the Escherichia coli and lambda phage genomes. IHF's ability to bend lambda phage DNA makes it an important architectural protein in the formation of a nucleoprotein complex that precedes the integration of the phage into the bacterial genome, as well as in several other biological processes.

The kinetics of IHF-DNA complex formation affects the overall rate of IHF-dependent biological process. In our work we have determined the site-specific association kinetics of IHF binding to one of its sites from the lambda phage genome, the H site. A key discovery from our kinetic experiments is that IHF binding and bending of DNA happens in a concerted manner.

The IHF-DNA crystal structure (**Figure 1**) was solved in 1996 by University of Chicago's biochemist Phoebe A. Rice and her collaborators. We conducted our time-resolved x-ray footprinting experiments on this complex. Phosphorus(32)-radiolabeled DNA was mixed with IHF, the solution was incubated, and later exposed to NSLS-generated x-ray beams.

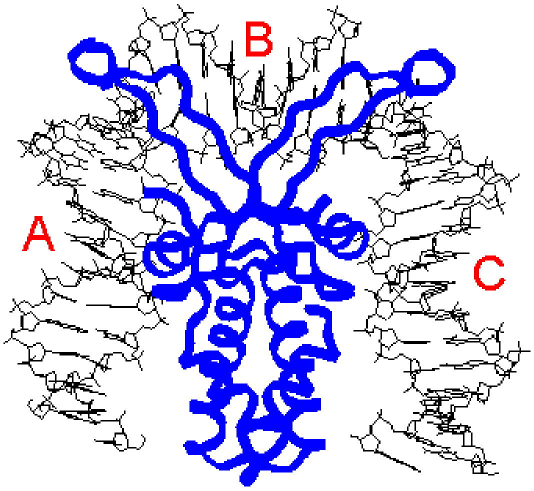

Figure 1. The IHF-DNA crystal structure is shown with the three sequence protein-DNA contact surfaces observed in our kinetic study labeled A, B and C.

Mark Chance

The cleaved fragments of DNA were run on a denaturing gel, as shown in **Figure 2**. The three protections along the DNA correspond to the three contacts made by a single molecule of IHF labeled A, B and C respectively. A kinetic fit to the protections calculated for each site relative to a control (unbound) DNA sequence shows that the three 'footprints' upon the DNA occur with similar kinetics (**Figure 3**).

All three DNA binding sites are bound by IHF with comparable kinetic profiles, indicating that they are contacted concertedly in the last step of IHF-DNA complex formation.

The best fit of the data to a bi-exponential equation is characterized by an initial burst phase in binding that displays rates faster than the speed of conventional diffusion (around 10^9 M^{-1} sec^{-1}) followed by a slower phase in binding corresponding to a bimolecular association rate around 10^8 M^{-1} sec^{-1}.

The time-resolved x-ray footprinting assay has also proved extremely useful in determining site-specific rates of RNA folding in another nucleic acids kinetic study. We envision that the set-up at our committed beamline at the NSLS will be instrumental in determining specific kinetics for individual molecules in multi-protein-nucleic acid complexes.

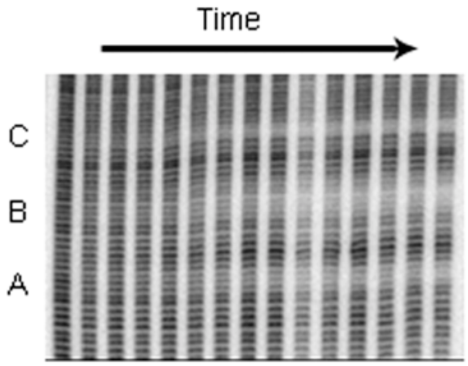

***Figure 2**. The image of the gel shown contained separated DNA fragments during complex formation for each time point (each column). The developing protection patterns as a function of time are labeled A, B and C and correspond to the appropriate contacts identified in the crystal structure.*

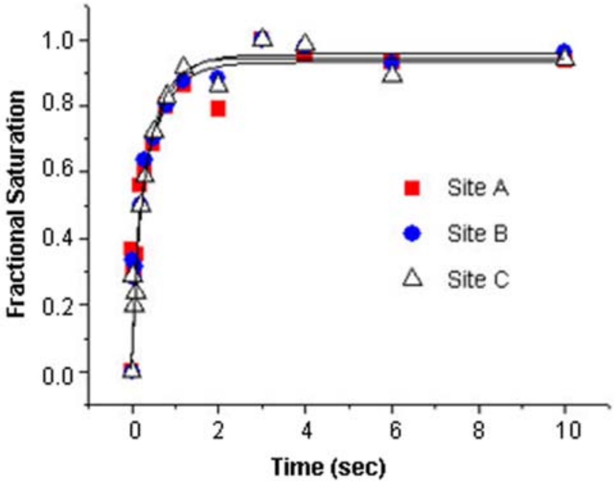

***Figure 3**. The kinetic curves obtained for each protection pattern are shown as a least squares fit to a bi-exponential equation.*

BEAMLINE
X12C

PUBLICATION
A.D. Ferguson et al., "Structural Basis of Gating by the Outer Membrane Transporter FecA," *Science*, **295**, 1715 (2002).

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Structural Basis of Gating by the Outer Membrane Transporter FecA

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Scientists working at the NSLS and Argonne National Laboratory's Advanced Photon Source (APS) have determined the crystallographic structure of the outer membrane receptor FecA from Escherichia coli with and without ferric citrate – an essential nutrient of bacteria – at 2.5 and 2.0 angstrom resolution. This study establishes the structural basis of gating for receptors dependent on the cytoplasmic membrane protein TonB.

Despite its relative abundance in Earth's crust, iron is biologically unavailable in the atmosphere. So, in response to iron deficiency, most microbes secrete organic chelators – compounds having a central metallic ion attached to two or more nonmetallic atoms – called siderophores, which are designed to sequester ferric iron.

Bacteria survive by using highly selective mechanisms designed to actively pump iron across the cell envelope. These mechanisms, responsive to both the internal and external iron concentration, control the transcription of genes involved in iron uptake. The ferric citrate uptake (*fec*) genes are responsible for the transport of ferric citrate from the external medium into the cell. Embedded within the outer membrane is FecA, a receptor that binds and transports ferric citrate, and is required to initiate transcription of the *fec* uptake genes.

We have determined the architecture of FecA from *Escherichia coli* and its gating mechanism by x-ray crystallography. Using mixed detergent-protein micelles, FecA crystals with and without ferric citrate were grown. Data was collected to 2.0 Å from unliganded FecA at beamline X12C of the National Synchrotron Light Source, and to 2.5 Å from liganded FecA at the Argonne National Laboratory's Advanced Photon Source.

Johann Deisenhofer, professor of biochemistry and investigator in the Howard Hughes Medical Institute at the University of Texas Southwestern Medical Center in Dallas, conducted the study on the outer membrane transporter FecA with lead author and postdoctoral researcher Andrew Ferguson. Deisenhofer received the 1988 Nobel Prize in Chemistry.

FecA is composed of three domains (**Figure 1**): (i) a 22-stranded antiparallel β-barrel embedded within the outer membrane, with long extracellular loops and short periplasmic turns; (ii) a "plug," consisting of a mixed four-stranded β-sheet with short interspersed helices, and extracellular and periplasmic pockets, located above and below the plug; (iii) a disordered third domain, the NH_2-terminal extension, which resides entirely within the periplasm and is required for the initiation of transcription.

Ferric citrate binding affects the conformation of the barrel and the plug domain of FecA by causing a dramatic change in the spatial arrangement and conformations of primarily the seventh and eighth extracellular loops, as shown in **Figure 2**.

From our structural observations, we propose the following transport mechanism.

Stage 1: Ferric citrate is adsorbed from the medium primarily by aromatic residues found within the upper portion of the external pocket of FecA.

Stage 2: Ferric citrate is transferred to its high-affinity binding site, causing an allosteric transition within the plug that signals the occupancy of FecA in the periplasm.

Stage 3: Multiple extracellular loops of the barrel change their relative conformation and position, thereby closing the external pocket of the barrel.

Stage 4: Transitions that modify the conformation of the plug domain and/or barrel are needed for transport to occur, and are mediated by physical interactions between FecA and the cytoplasmic membrane protein TonB.

Our findings clarify the current understanding of energy-dependent siderophore uptake across the bacterial outer membrane, and establish the structural basis of gating for TonB-dependent receptors. Further genetic and biophysical studies are needed to establish the molecular basis of energy-dependent transport.

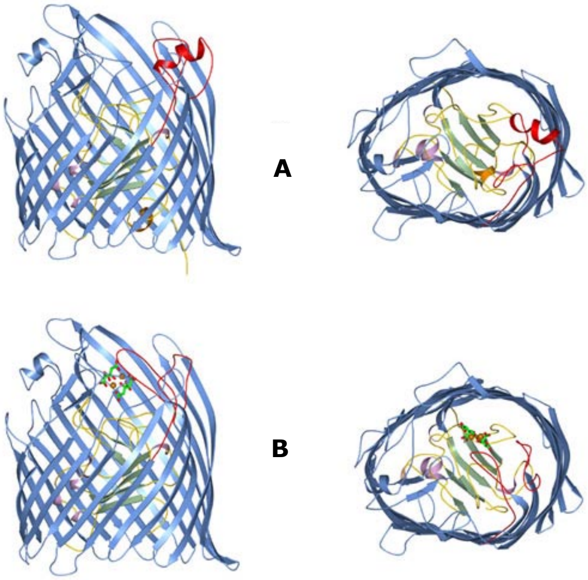

Figure 1: *Crystallographic structure of FecA, (A) unliganded, and (B) liganded. The 22-stranded β barrel is shown in blue. The mixed four-stranded β sheet of the plug domain is shown in green. The switch helix, located in the periplasmic pocket of FecA, is colored orange and is only observed in the unliganded conformation.*

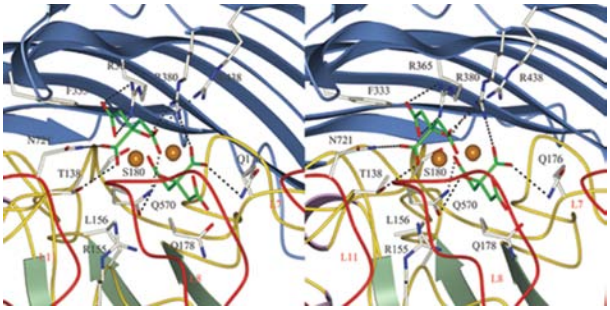

Figure 2: *Stereoview of the ferric citrate-binding site. All side chains within 3.5 angstrom of dinuclear ferric dicitrate are shown with carbon atoms in white, nitrogen atoms in blue, and oxygen atoms in red. The strands and extracellular loops of the barrel are shown in blue; the strands forming the plug domain are in green, and loops are in yellow. The dinuclear ferric citrate molecule is represented with oxygen atoms in red, carbon atoms in green, and ferric ions in orange.*

BEAMLINE
X12C

PUBLICATION
X. Chen et al., "Structural Identification of a Bacterial Quorum-sensing Signal Containing Boron," *Nature* , **415**, 545 (2002).

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Structural Identification of a Bacterial Quorum Sensing Signal Containing Boron

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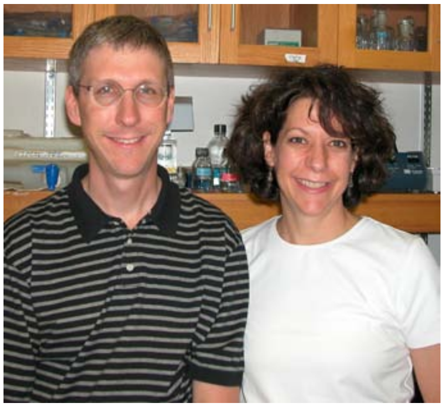
In a process known as quorum sensing, bacteria communicate with one another using chemical signalling molecules called autoinducers. This cell-cell communication allows a population of bacteria to coordinate the gene expression, and therefore the behavior, of the group. Recent work by researchers from Princeton University and the School of Chemistry, Polymers and Materials in Strasbourg, France, has led to the identification of a novel autoinducer, AI-2, that may serve as a 'universal' signal for communication between different bacterial species. AI-2 unexpectedly contains boron, an element that, while ubiquitous in the biosphere, has seldom been observed to play a role in biological processes.

Quorum sensing allows a population of bacteria to coordinate their gene expression, and therefore their collective behavior. Usually, quorum sensing controls processes that are effective when a large number of bacteria act together. Some bacteria, for example, produce visible light in response to autoinducers that accumulate in dense cultures. Pathogenic bacteria can use quorum sensing to evade the immune system, express toxic virulence factors, or coordinate the formation of antibiotic-resistant biofilms.

Recently, we identified a novel autoinducer, AI-2, that has the potential to mediate communication among different bacterial species. The presence of AI-2 can be detected by adding it to a specially engineered strain of the bioluminescent marine bacterium *V. harveyi,* which emits light in the presence of AI-2. Using this assay, it was possible to show that a large number of bacterial species produce AI-2. These and other findings suggest that communication via AI-2 could be a common mechanism that bacteria employ for inter-species interaction in natural environments.

The chemical identity of AI-2 was ascertained in a somewhat unusual way using x-ray crystallography. AI-2 was crystallized in a complex with its *V. harveyi* receptor protein, LuxP. Crystals formed by the LuxP-AI-2 complex diffracted beyond 1.5 Å resolution at NSLS Beamline X-12C. Because of the high quality of the resulting electron density maps, it was straightforward to construct an atomic model for AI-2 (**Figure 1**).

Earlier work had suggested that AI-2 is derived from the precursor 4,5-dihydroxy-2,3-pentanedione. The cyclic form of this precursor is a plausible substrate for the addition of borate. We were nonetheless surprised to find that the AI-2 electron density appears to match this borate addition product. Confirmatory evidence, including ^{11}B-NMR spectra and the ability of boric acid to stimulate bioluminescence in *V. harveyi* supported the conclusion that AI-2 is a novel furanosyl borate diester. Thus, interspecies quorum sensing represents one of the first biochemically defined roles for boron in biology.



Members of the Princeton University team: Team leader Fred Hughson (left) and collaborator Bonnie Bassler.

Many questions remain. For example, it is not yet clear how, or whether, the use of boron enhances signal transmission or signal specificity. Also, we do not yet know whether the use of boron in cell-cell signaling is confined to bacteria that live in marine environments or is more widespread. Nonetheless, biotechnological research is focused on the development of molecules that are structurally related to AI-2. Such molecules may be useful as anti-microbial drugs aimed at bacteria that control virulence through AI-2 quorum sensing. Since many bacteria produce AI-2, drugs that target AI-2 quorum sensing could be broadly used in the future.

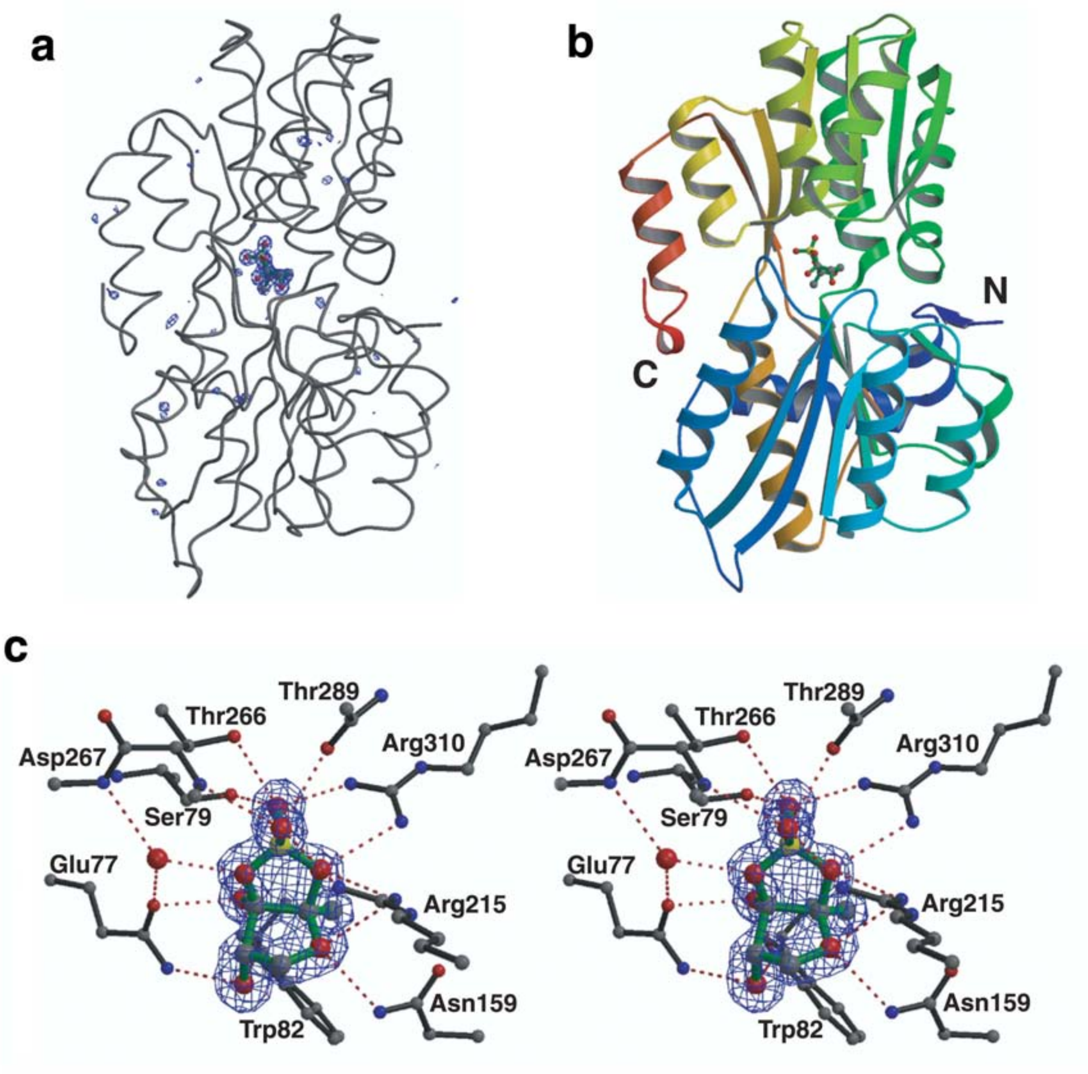


Figure 1. *Structure of the LuxP-AI-2 complex.* ***a.*** *Electron density from which the LuxP contribution has been subtracted to highlight the electron density for AI-2 in the center.* ***b.*** *Overview showing AI-2 (stick figure) bound to LuxP (ribbon). The Lux P protein backbone is shown in rainbow colors from its N- to its C-terminus.* ***c.*** *Stereoview close-up of AI-2 bound to LuxP, in the form of an electron density map (prior to AI-2 addition to the model). Boron, oxygen, nitrogen, and carbon are colored yellow, red, blue, and gray, respectively.*

BEAMLINE
X8C

PUBLICATION
D.H. Fong and A.M. Berghuis, "Substrate Promiscuity of an Aminoglycoside Antibiotic Resistance Enzyme via Target Mimicry," *EMBO Journal,* **21**, 2323 (2002).

FUNDING
Canadian Institutes of Health Research
Natural Sciences and Engineering Research Council of Canada
Rx&D Health Research Foundation

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Scientists Show that Resistance to Aminoglycoside Antibiotics is Due in Part to Target Mimicry

D.H. Fong and A.M. Berghuis

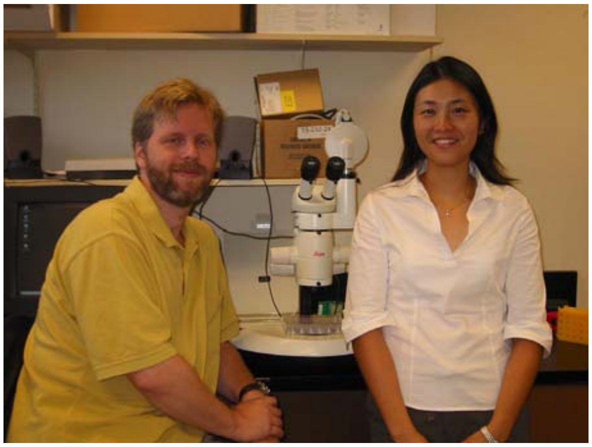
McGill University, Monteal, Canada

Bacterial resistance to antibiotics is a serious public health problem. The emergence of "superbugs" — pathogenic bacteria that can survive the effects of most commonly used antibiotics – significantly compromises existing treatments against infectious diseases. The predominant mechanism of resistance to aminoglycosides, a class of bactericidal antibiotics that are widely used in hospitals, is enzyme-catalyzed chemical modification of the drug. One of the enzymes that catalyze antibiotic detoxification is aminoglycoside kinase (3') type IIIa [APH(3')-IIIa]. Researchers from McGill University have obtained three-dimensional structures of this enzyme with two structurally diverse aminoglycoside antibiotics bound in the active site. These structures provide insight into how the enzyme is able to detoxify many different aminoglycosides, and suggest novel treatments.

Due to the widespread use and misuse of antibiotics, bacteria have developed mechanisms for evading the effects of these drugs. Enzymatic modification of the antibiotic is by far the major antibiotic resistance mechanism to aminoglycoside antibiotics. Ordinarily, an aminoglycoside exerts its bactericidal effect by binding to the A-site of the 16S ribosomal RNA, resulting in errors in translation, and ultimately bacterial cell death. But an aminoglycoside altered through enzymatic modification has dramatically reduced affinity for the ribosomal RNA, and is therefore no longer harmful to the pathogenic bacteria.

We studied aminoglycoside kinase (3′) type IIIa [APH(3′)-IIIa], an enzyme that can be found in several pathogenic bacteria, including *enterococci* and *staphylococci*. This enzyme can inactivate at least ten structurally diverse aminoglycosides by transferring one or two phosphate groups from adenosine triphosphate (ATP) to these drugs. We have studied the crystal structure of APH(3′)-IIIa in complex with adenosine diphosphate (ADP) and either kanamycin or neomycin. These two crystal structures represent the first aminoglycoside kinase structures with bound antibiotic substrates.

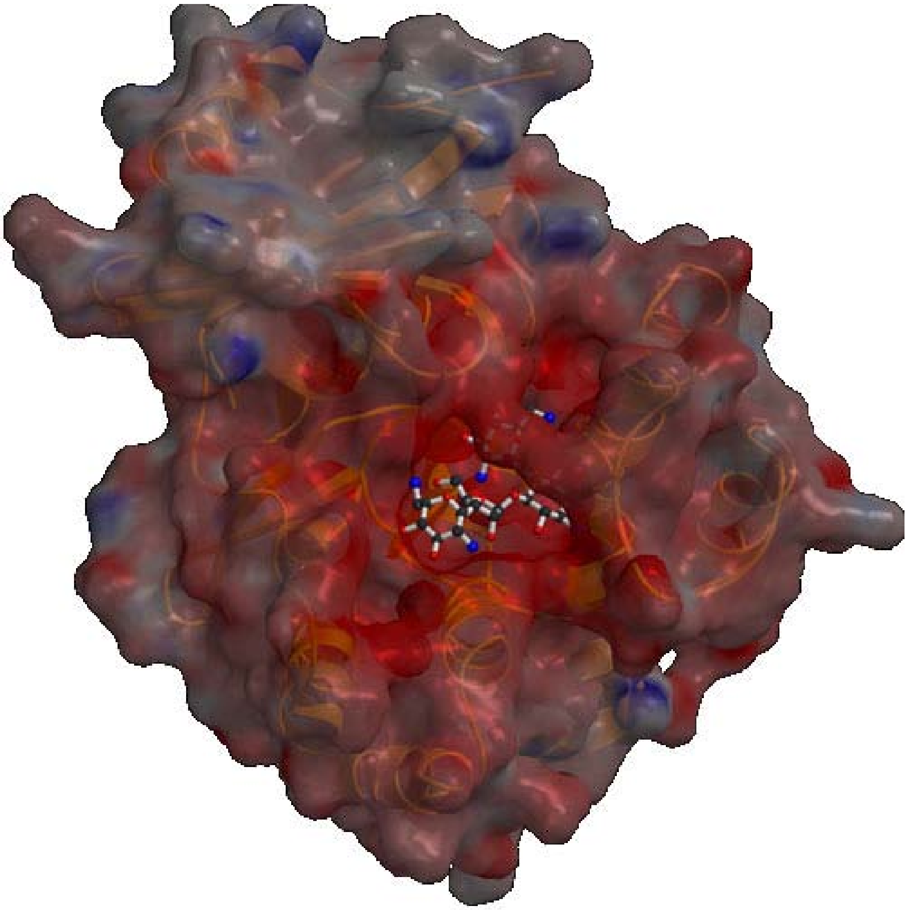
Crystals were obtained by co-crystallization of the enzyme in the presence of ADP and kanamycin or neomycin. We collected 2.9Å resolution data for the kanamycin ternary complex from a rotating copper anode x-ray generator. We also collected 2.4Å and 2.7Å resolution data sets for ternary complexes of a second kanamycin and neomycin, respectively, at the X8C beamline of the National Synchrotron Light Source at Brookhaven National Laboratory.


Albert Berghuis (left) and Desiree H. Fong

Our results reveal how the three-dimensional structure of APH(3′)-IIIa is able to bind to various aminoglycoside substrates. First, since aminoglycosides are invariably positively charged molecules, the substrate-binding pocket is lined with numerous negatively charged residues. Secondly, the APH(3′)-IIIa antibiotic binding pocket is an extended area which has three sub-sites, labeled A, B and C, and different types of aminoglycosides bind to either subsites A and B or A and C. Finally, a flexible loop forms one wall of the antibiotic binding pocket, allowing for additional variability in aminoglycoside binding.

To understand why APH(3′)-IIIa provides resistance to aminoglycosides, we compared the A-site of the bacterial ribosome with APH(3′)-IIIa. We noticed that the binding pocket of APH(3′)-IIIa successfully mimics the aminoglycoside-bound A-site of the ribosome. Although they differ in overall structure and in the nature of the polypeptide (i.e. amino acids vs. ribonucleic acids), they display identical spatial arrangement of hydrogen bond donor and acceptor groups. This means that aminoglycosides that bind to the A-site of the ribosome may also bind to APH(3′)-IIIa, which acts as an efficient decoy target for aminoglycosides.

The close mimicry of the bacterial ribosomal A-site by APH(3′)-IIIa raises concerns about the development of new antibiotics that target the 16S RNA. Fortunately, the antibiotic binding pocket of APH(3′)-IIIa and the ribosomal A-site differ in one crucial aspect: They display significantly different van der Waals interactions with aminoglycosides. This difference suggests possible strategies for the design of novel variant aminoglycosides that can interact with the ribosome but cannot be detoxified by APH(3′)-IIIa and related antibiotic resistance factors.



Ribbon diagram of aminoglycoside kinase (3′) type IIIa [APH(3')-IIIa] in complex with the aminoglycoside antibiotic, Neomycin. The antibiotic is shown in ball and stick style and the molecular surface of APH(3')-IIIa is displayed as a semi-transparent representation colored according to the electrostatic potential (red being negatively charged and blue being positively charged).

A Novel Mechanism for *Clostridium Botulinum* Neurotoxin Inhibition

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BEAMLINE
X12C

PUBLICATION
S. Eswaramoorthy, D. Kumaran and S. Swaminathan, "A Novel Mechanism for *Clostridium botulinum* Neurotoxin Inhibition," *Biochemistry*, **41**, 9795-9802 (2002).

FUNDING
Chemical and Biological Non-proliferation Program of the U.S. DOE
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Treatment of botulism, an acute paralytic disease caused by the bacterium Clostridium botulinum, is currently only preventive. An experimental vaccine is available, but no drug has been developed yet. Biologists at Brookhaven National Laboratory are studying how a chemical called bis(5-amidino-2-benzimidazolyl)methane (BABIM) inhibits one of the toxins produced by the bacterium. The results of this study could help design new drugs against botulism.


Authors of the study (left to right): Subramaniam Eswaramoorthy (lead author), Subramanyam Swaminathan, and Designan Kumaran.

The bacterium *Clostridium botulinum* produces one of the most potent neurotoxins – poisonous proteins acting on the nervous system – to humans. Seven different types of neurotoxins, labeled A to G, are produced by the bacterium.

To infect a neuronal cell, a neurotoxin first binds to the membrane of the cell. Then, the cell membrane curves inward, incorporating the toxin into a vesicle that drifts inside the cell. The toxin escapes the vesicle by creating a channel in the vesicle membrane, inserting its light chain (LC) through the channel, and slipping away completely into the cell's cytosol (fluid portion of the cytoplasm). The toxin is "translocated" from the vesicle to the cytosol. Then, the toxin cleaves specific targets in the cytosol and blocks the release of neurotransmitters – chemicals produced by neurons to communicate with each other – thereby causing muscular paralysis and the eventual death of the patient infected with the bacterium.

The three-dimensional structure of a *botulinum* toxin reveals three domains, called binding, translocation, and catalytic domains, corresponding to the three functions of the toxins.

All seven toxins contain a zinc-based structure in their catalytic domain, but structural details may be different, because each toxin cleaves different types of targets. For example, the catalytic zinc is located in a deep cavity in the active site of all toxins, but the cavity is partially covered by a "belt" region in *botulinum* neurotoxin A (BoNT/A) while it is open in BoNT/B. Treatment of botulism is currently only preventive: An experimental vaccine is available, but no drug has been developed yet. Therapeutic treatment could be effective at any one of the three stages of toxicity – binding of toxin, internalization, or catalytic activity.

Chemicals such as Bis(5-amidino-2-benzimidazolyl)methane (BABIM) are known to combine with the catalytic zinc, thus preventing the toxin from binding to specific targets, also called substrates, in the cytosol. But the effectiveness of BABIM on the toxin needs to be further investigated.

We have determined the crystal structure of a complex of BoNT/B and BABIM, and investigated how BABIM binds to the zinc atom and inhibits the toxin.

The crystal structure of the complex revealed a tunnel that connects a cleft formed between the translocation domain and the catalytic domain to the active site cavity, as shown in **Figure 1**.

Interestingly, two molecules of BABIM bind to the toxin on either side of an aspartic acid molecule, suggesting that the inhibitor can enter and bind to the active site in two different ways. We suggest that one inhibitor molecule has entered through the cleft between the translocation and catalytic domains while the other has entered through the wide opening of the active site cavity.

The inhibitor molecule near the active site perturbs and disrupts the zinc bonds to surrounding molecules in the toxin, as shown in **Figure 2**.

The structure of the BoNT/B-BABIM complex has shown that the active site residues rearrange in the presence of the inhibitor, allowing it to partly occupy the site where the substrate would bind. Also, the zinc atom is progressively removed from the active site and transported to a different site in the protein. We have also shown that it is possible for appropriate inhibitors to enter the active site of the toxin contrary to the belief that the belt surrounding the catalytic domain shields the active site. But the inhibition could be due to either the non-availability of substrate-binding sites, the removal of the zinc atom, or a combination of both.

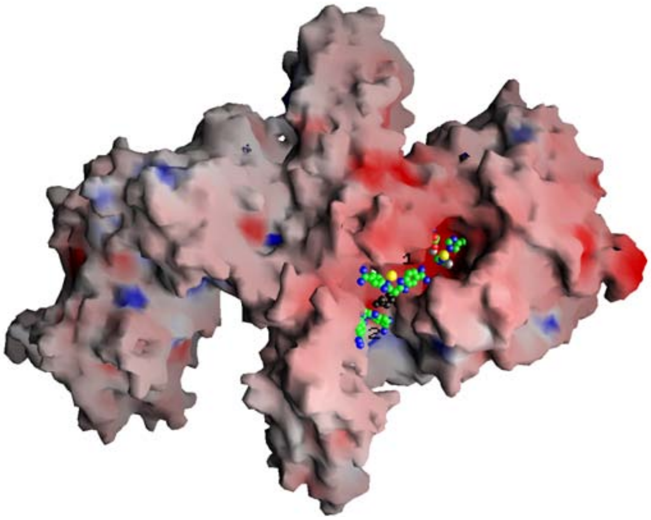

Figure 1. *Representation of the electrostatic potential surface of* botulinum *neurotoxin B. The two molecules of bis(5-amidino-2-benzimidazolyl)methane that are trapped in the tunnel and the residues coordinating the zinc atom are shown as sphere model. Zinc atoms are shown in yellow while water molecules coordinating with the zinc atoms are in silver gray.*

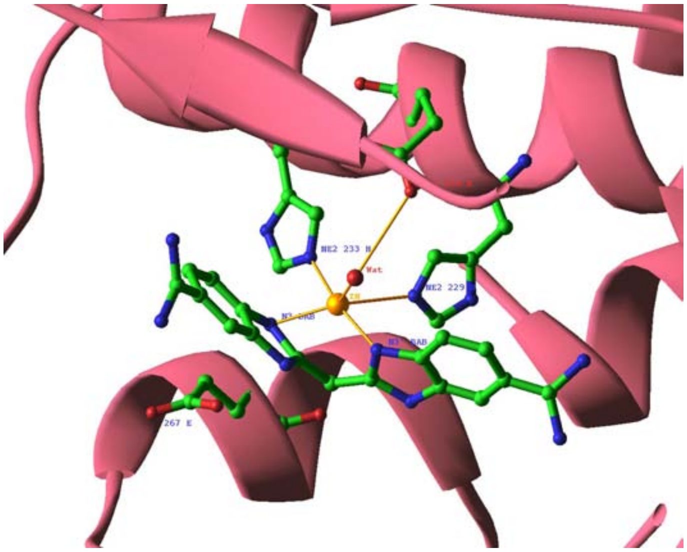

Figure 2*. Representation of bis(5-amidino-2-benzimidazolyl)methane (BABIM) at the neurotoxin's active site, with the light chain and the belt region. Coordination to the zinc atom from protein ligands and BABIM are shown as thin yellow lines.*

BEAMLINE
X4A

PUBLICATION
B. Hao, W. Gong, T. Ferguson, C. James, J. Krzycki, and M. Chan. "A New UAG-Encoded Residue in the Structure of a Methanogen Methyltransferase", *Science*, **296**, 1462-1466 (2002).

FUNDING
National Institutes of Health
National Science Foundation
U.S Department of Energy
Alfred P. Sloan Foundation

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Identification of the 22nd Genetically-Encoded Amino Acid in a Methanogen Methyltransferase

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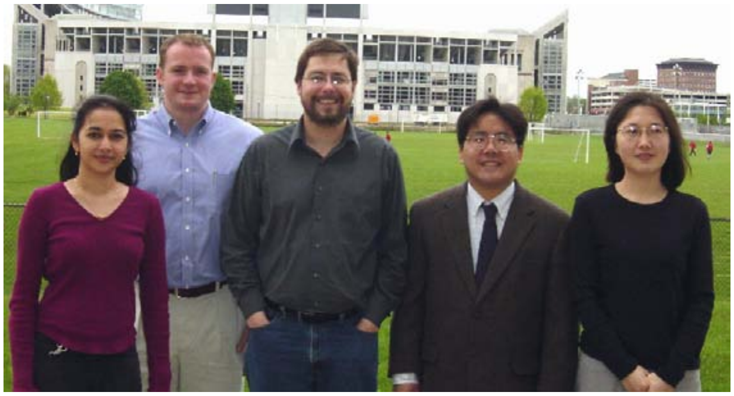
Ohio State University, Columbus Ohio

Proteins have long been known to be composed of only 20 building blocks, called amino acids. But about 25 years ago, scientists discovered an additional, 21st amino acid, called selenocysteine. Now, two groups of researchers led by biochemist Michael Chan and microbiologist Joseph Krzycki, both of The Ohio State University in Columbus, have identified the 22nd amino acid in an enzyme, called methyltransferase, which breaks down methylamine (CH_3NH_2) in methane(CH_4)-producing microbes called methanogens, leading to the production of methane. The scientists call this new amino acid pyrrolysine.



Members of the two teams that conducted the study (from left to right): Gayatrhi Srinivasan, Carey M. James, Joseph A. Krzycki, Michael K. Chan, Bing Hao.

Inside cells, proteins are synthesized by two processes, called transcription and translation. In the first process, the genetic information of DNA is "transcribed" into messenger RNA (mRNA). In the second process, mRNA is "translated" into a series of amino acids, the building blocks of proteins, which self-assemble to form the protein.

During the translation process, a protein/RNA complex called the ribosome attaches to the mRNA, and reads its nucleotides (uracil U, adenine A, guanine G and cytosine C) three by three. Inside the ribosome, transfer RNA (tRNA) molecules attach to each set of three nucleotides, or codon, and provide the corresponding amino acid, which is added to a chain of amino acids that folds and creates, little by little, a protein.

Each of the 64 possible codons has a specific tRNA that recruits one of 20 "standard" amino acids, except for UAA, UAG, and UGA. These three codons are generally used to indicate the end of the amino acid chain. In certain cases, however, a specific tRNA associates UGA to a "nonstandard" amino acid, the 21st amino acid, called selenocysteine.

Over the last few years, two groups of scientists at The Ohio State University in Columbus, led by biochemist Michael Chan and microbiologist Joseph Krzycki, initiated a collaboration to further identify what amino acid is encoded by UAG.

The Krzycki group found that a microbe known to produce methane (CH_4), called *Methanosarcina barkeri*, contain a UAG-decoding tRNA (**Figure 1**), and a unique lysyl-tRNA synthetase (PylS), a protein that helps translating the UAG codon. Krzycki and his colleagues noticed that PylS could charge the UAG-decoding tRNA with lysine, which is then enzymatically modified to form another amino acid.

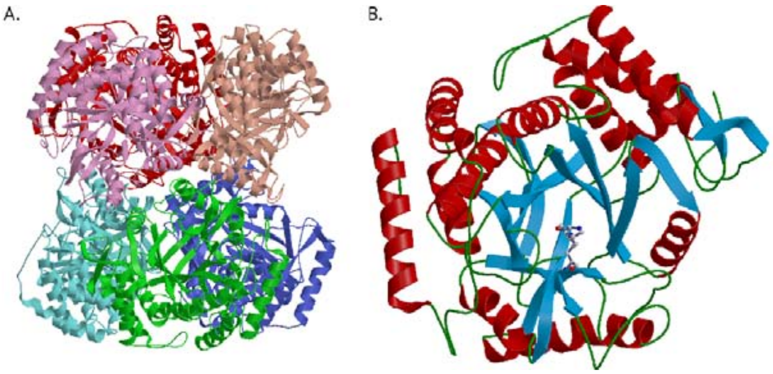
To characterize the identity of the UAG-encoded amino acid, the structure of the monomethylamine methyltransferase was determined by the Chan group. Two forms of the enzyme were obtained from crystallization conditions that differed only in the precipating salt used [NaCl and $(NH_4)_2SO_4$] and were solved to 1.55 angstrom (Å) and 1.7 Å resolution, respectively.

The 1.55 Å resolution methyltransferase structure reveals a hexameric protein, with each subunit adopting a TIM barrel fold – a structure present in 15 enzyme families (**Figure 2**).

The UAG-encoded amino acid, called pyrrolysine, lies at the bottom of a cleft, shown as a ball-and-stick model in **Figure 2**.

Fitting of the electron density from the two different crystal forms suggests that the UAG encoded amino acid is described by the chemical formula: 4-substituted-(4R,5R)-pyrroline-5-carboxylate, with the carboxylate of the modifying group attached to the epsilon nitrogen of lysine (**Figure 3**).

By showing that pyrrolysine corresponds to a UAG codon in some genes, and that an amber decoding tRNA is found in organisms containing these genes, the researchers demonstrate that pyrrolysine is the 22nd genetically encoded amino acid to be identified in nature.



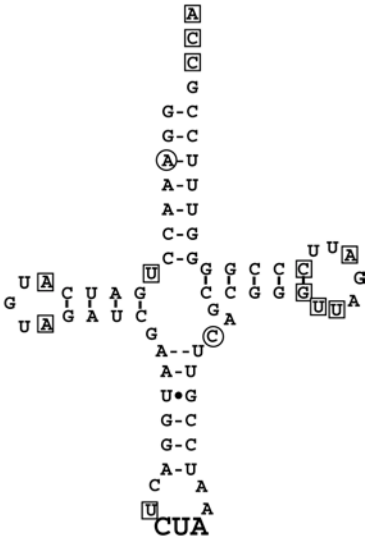


Figure 2. Ribbon diagram of a subunit of the Methanosarcina barkeri monomethylamine methyltransferase hexamer (α helices: green; β sheets: cyan; random coil: yellow). The atoms of the UAG-encoded residue are shown as ball-and-stick models and are colored by their elements, with carbon as gray, nitrogen as blue, and oxygen as red.

Figure 1. Structure of the UAG-decoding tRNA

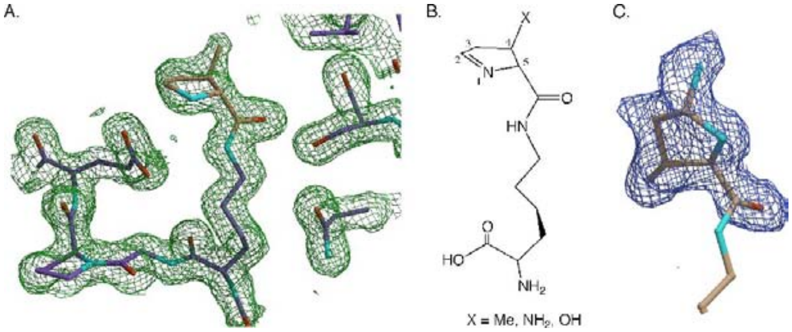


***Figure 3. (A)** Fit of 4-substituted-(4R,5R)-pyrroline-5-carboxylate to the electron density of the NaCl crystal form of pyrrolysine. **(B)** Stick-diagram of proposed pyrrolysine amino acid. The substituent attached to the C-4 carbon could be a methyl (Me), an ammonium (NH_2), or a hydroxyl group.*

BEAMLINE
X12B

PUBLICATION
T. Zhou, *et al.*, "Structure of Human Nicotinamide/Nicotinic Acid Mononucleotide Adenylyltransferase: Basis for the Dual Substrate Specificity and Activation of the Oncolytic Agent Tiazofurin," *J. Biol. Chem.*, **277**, 13148-13154 (2002).

FUNDING
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Structure of Human Nicotinamide/Nicotinic Acid Mononucleotide Adenylyltransferase (NMNAT)

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[1]Univ. of Texas Southwestern Medical Center at Dallas; [2]Integrated Genomics, Inc.; [3]Frederick Cancer Research and Development Center, National Cancer Institute

Nicotinamide adenine dinucleotide (NAD) is a coenzyme (nonprotein part of an enzyme) involved in many metabolic reactions inside the cell, as well as DNA repair and calcium signaling. NAD results from the addition of adenylate (one of RNA's building blocks) to the molecule nicotinamide mononucleotide (NMN), a process catalyzed by the enzyme NMN adenylyltransferase (NMNAT). The enzyme is also involved in activating the anticancer agent, tiazofurin. To understand better the role of NMNAT in NAD biosynthesis and tiazofurin conversion, scientists have used x-rays produced at the National Synchrotron Light Source and the Advanced Photon Source to determine the structure of NMNAT with different ligands, providing insight into the molecular mechanisms of the enzyme's active site.

Nicotinamide adenine dinucleotide (NAD) is a coenzyme (nonprotein part of an enzyme) that has been known for decades as the major hydrogen donor or acceptor in many metabolic reactions inside the cell, as well as the modification of nuclear proteins by ADP ribosylation, a process involved in DNA repair and the regulation of genomic instability.

Scientists have recently found that NAD is a substrate or a co-factor in the SIR2-like histone deacetylase, responsible for gene silencing and the increase of lifespan of many species, including yeast, worms, and mammals. Also, several derivatives of NAD are intracellular calcium mobilizing agents in various calcium signaling pathways.

NAD results from the addition of adenylate (one of RNA's building blocks) to the molecule nicotinamide mononucleotide (NMN), a process catalyzed by the enzyme NMN adenylyltransferase (NMNAT). NAD can also be synthesized by the following two successive processes: addition of adenylate to the molecule nicotinate mononucleotide (NaMN), leading to nicotinate adenine dinucleotide (NaAD), and addition of an amide group (organic compound containing the $CONH_2$ radical) to NaAD, leading to NAD. The first process is catalyzed by NMNAT and the second by NAD synthetase. (A synthetase is an enzyme that catalyzes the union of two molecules.) The processes are illustrated at: http://hhmi.swmed.edu/Labs/hz/nad1.htm

Human NMNAT also catalyzes part of the metabolic conversion of the anti-cancer agent, tiazofurin, to its active form, tiazofurin adenine dinucleotide (TAD), an NAD analogue. The development of tiazofurin resistance has been shown to relate mainly to a decrease in NMNAT activity. The process is illustrated at: http://hhmi.swmed.edu/Labs/hz/tiazofurin.htm

Human NMNAT, located within the cell nucleus, recognizes both NMN and NaMN substrates. To understand the enzymatic properties of NMNAT and how its activity is regulated, we have solved the crystal structures of human NMNAT attached to several ligands, including NAD, NaAD, and TAD, to 2.2-angstrom (Å) resolution.

Hong Zhang (lead author, right) and two members of her team: Subramanian Karthikeyan (seated) and Xuejun Zhang, at the University of Texas Southwestern Medical Center in Dallas.

The data were produced and collected by using x-rays at beamline X12B of the National Synchrotron Light Source and beamline 19ID of the Advanced Photon Source at Argonne National Laboratory.

Our results show that NMNAT bind to NMN and NaMN with high affinity. In particular, a water molecule in the active site appears to play a critical role in the recognition of NaMN (**Figure 1**).

By looking at the structure of NMNAT attached to TAD, we found that TAD molecule adopts a similar conformation as the native ligand NAD, and that TAD and NAD form essentially the same interactions with NMNAT. Additional functional groups on tiazofurin molecule also may enable more favorable interactions between tiazofurin and NMNAT. Further kinetic measurement will be needed to fully characterize the molecular interactions between human NMNAT and tiazofurin.

The results presented here represent a first step in our effort to decipher the metabolic pathways of NAD biosynthesis and how it is regulated in humans. We have recently identified an additional cytosolic form (inside the cell cytoplasm instead of the cell nucleus) of human NMNAT; its structural determination is well underway.

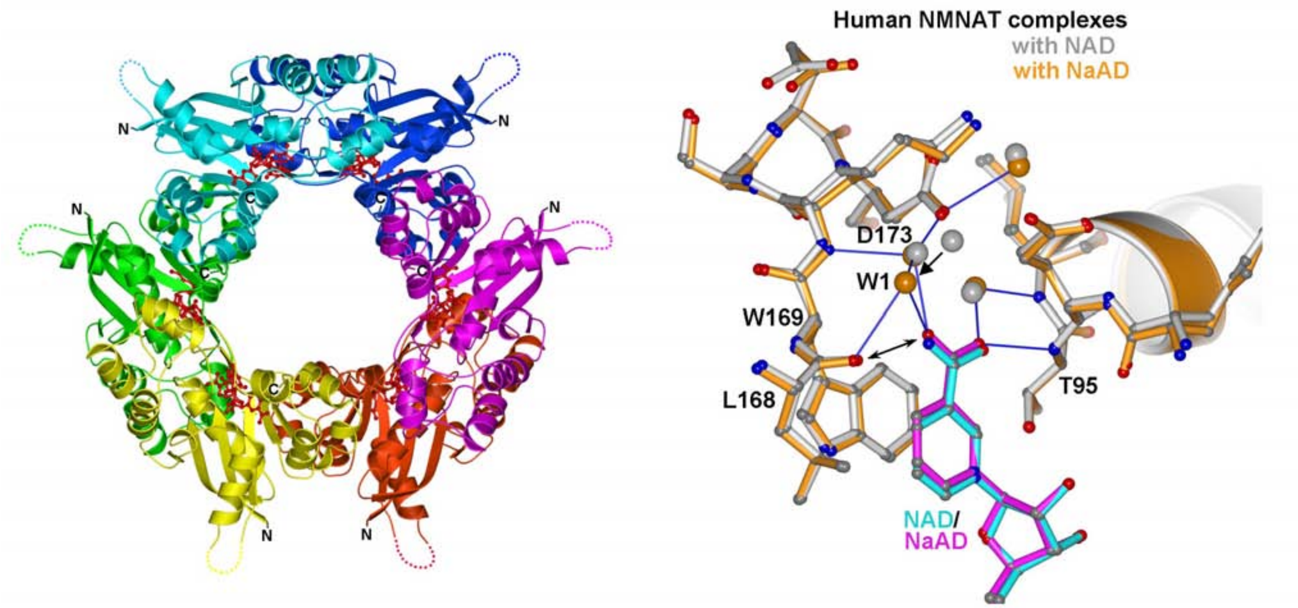

Figure 1. Left*: Ribbon diagram of human nicotinamide mononucleotide adenylyltransferase (NMNAT) hexamer.* ***Right****: The active site of human NMNAT with nicotinamide adenine dinucleotide (NAD) and nicotinate adenine dinucleotide (NaAD). A water molecule (denoted w1) in the active site of NMNAT changes location upon binding to different substrates.*

BEAMLINE
X4A

PUBLICATION
K.P. Locher, A.T. Lee, D.C. Rees, "The *E. coli* BtuCD Structure: A Framework for ABC Transporter Architecture and Mechanism," *Science*, **296**, 1091-1098, (2002).

FUNDING
Howard Hughes Medical Institute

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Structure of a Bacterial ATP-Binding Cassette Transporter

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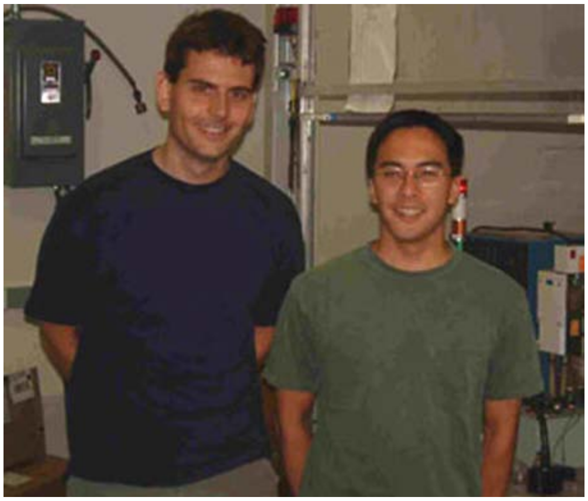
Howard Hughes Medical Institute and Division of Chemistry and Chemical Engineering, California Institute of Technology, Pasadena

Adenosine triphosphate (ATP)-binding cassette (ABC) transporters are ubiquitous membrane proteins that couple ATP hydrolysis to the transport of diverse substrates across cell membranes. Clinically relevant examples are associated with cystic fibrosis and with multidrug resistance of pathogenic bacteria and cancer cells. Using x-rays produced at the National Synchrotron Light Source and other light sources, scientists at the California Institute of Technology in Pasadena have determined the crystal structure at 3.2 angstrom resolution of the Escherichia coli BtuCD protein, an ABC transporter mediating vitamin B_{12} *uptake.*



Kasper Locher (left) and Allen Lee

To survive, cells import nutrients from the surrounding environment and pump toxic substances out of the cytoplasm. These functions are carried out by transport proteins, called transporters, embedded in cell membranes. The largest family of these proteins, called ATP-binding cassette (ABC) transporters, is ubiquitous in all branches of life. (ATP, or adenosine triphosphate, is the primary source of energy in all living cells.) ABC transporters power the transport of substrates across membranes by using the energy released by the hydrolysis, or water-induced decomposition, of ATP into ADP (adenosine diphosphate) and inorganic phosphate.

Several human ABC transporters are medically relevant. For example, mutations in the cystic fibrosis transmembrane conductance regulator (CFTR) protein cause cystic fibrosis. Other ABC transporters are associated with multidrug resistance of tumor cells against cytotoxic substances used in chemotherapy.

In bacteria, ABC transporters are predominantly involved in nutrient uptake, although they also participate in the export of bacterial toxins and harmful substances, contributing to bacterial multidrug resistance.

Despite the immense amount of biochemical studies, and recent advances in the visualization of ABC transporters, their transport mechanisms have remained elusive. To understand better how these transporters operate, the crystal structure of the *Escherichia coli* vitamin B_{12} importer BtuCD protein has been determined, with all critical parts ordered and resolved.

To solve the structure of BtuCD, close to one thousand crystals were screened and data were collected at various synchrotron light sources, including the National Synchrotron Light Source at Brookhaven National Laboratory.

The structure of BtuCD has revealed three key elements to the transport of vitamin B_{12} into the cytoplasm (**Figure 1**):

(1) A transport pathway through the membrane-spanning BtuC subunits. In the absence of ATP, this pathway is accessible from the outside, but is sealed to the cytoplasm by a gate region.

(2) The ATP-binding cassettes (ABCs): Located beneath the BtuC subunits, these proteins present binding sites for two ATP molecules at the interface between the ABCs. As the ABCs bind and hydrolyze ATP, mechanical energy is generated and transmitted to the membrane-spanning domains BtuC, where it induces rearrangements that open the BtuC gate and allow the substrate to cross the membrane.

(3) A cytoplasmic loop of BtuC makes extensive contact with BtuD. Mutations in these critical interface residues severely affect the function and assembly of ABC transporters. For example, 70% of cystic fibrosis patients have a single residue deleted at a position that corresponds to this contact region of BtuD.

The present structure suggests the design of further biochemical studies that could probe the conformational changes of BtuCD during vitamin B_{12} transport. This structure also provides a framework for understanding the structure and mechanism of other ABC transporters.

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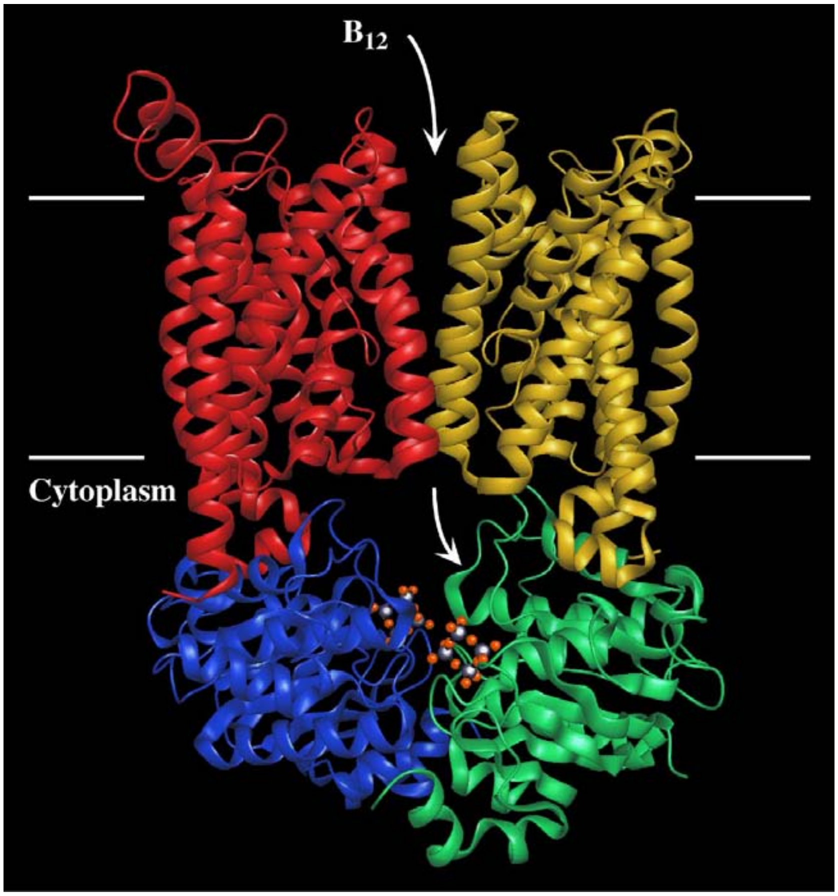

Figure 1. *Ribbon diagram of the BtuCD protein structure. The ATP-binding cassette (ABC) transporter is assembled from two membrane-spanning domains (BtuC, red and yellow) and two ABCs (BtuD, green and blue). At the ATP binding sites, cyclotetravanadate molecules are bound to the transporter (ball and stick models at the BtuD interface). Vitamin* B_{12} *is delivered to the transporter by a binding protein (not shown), likely transported through a pathway provided at the interface of the two membrane-spanning BtuC subunits, and then released into the cytoplasm at the large gap between the four subunits (arrows).* B_{12} *transport is powered by the hydrolysis of ATP by BtuD.*

BEAMLINE
X8C

PUBLICATION
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X-ray Structure of the Rotavirus Protein Involved in Genome Replication and Packaging

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Using x-rays at beamline X8C of the National Synchrotron Light Source, scientists have determined, to 2.6 angstrom (tenth of a billionth of a meter) resolution, the first x-ray structure of a key protein, called non-structural protein 2 (NSP2) of a rotavirus, the major cause of life-threatening infantile gastroenteritis. The structure provides molecular details of the protein's doughnut-shaped functional part, and reveals how it may be involved in the replication and packaging of the viral RNA during infection.

Rotavirus, the leading cause of infantile gastroenteritis, with nearly a million deaths worldwide every year, has a spherical shape with many spikes that bind firmly to a host cell. The virus is composed of six structural proteins arranged in three concentric layers, which enclose the viral genome consisting of 11 double-stranded RNA (dsRNA) segments. Each segment codes for at least one protein, except for one segment that codes for two proteins.

Every rotavirus particle is a fully contained unit that transcribes genome segments from the inside, and then releases the resulting messenger RNA (mRNA) molecules. When a rotavirus fully replicates its genome, several copies of the 11 segments are made (genome replication), and each progeny virus gets one copy of each segment (genome packaging).

One of the least understood, yet critically important, processes in the replication process of a dsRNA virus is the genome replication and packaging. The process by which the correct set of dsRNA segments is assigned to the capsids of each progeny virus remains a mystery.

Several *in vivo* and *in vitro* studies on rotavirus have provided insight into the role of proteins that are not incorporated into the virus but merely assist in genome replication and packaging. These proteins, referred to as non-structural proteins 2 and 5 (NSP2 and NSP5), are co-localized, along with the viral RNA polymerase, called VP1, in granular cytoplasmic inclusions, or viroplasms, in the infected host cell. (The RNA polymerase is the main protein involved in RNA replication.)


H. Jayaram (left) and B.V.V. Prasad


J. Patton (left) and Z. Tarporewala

Recent biochemical studies have shown that NSP2's functional part, a doughnut-shaped octamer – containing eight repeating units, or monomers – binds to RNA, has a nucleoside triphosphatase (NTPase) activity and has nucleic acid destabilizing activity. Based on these studies, NSP2 is thought to work as a molecular motor involved in genome packaging, by using the energy derived from the hydrolysis of nucleoside triphosphates.

We have determined the crystal structure of the functional octamer of NSP2 to a resolution of 2.6 angstrom (tenth of a billionth of a meter) resolution (**Figure 1**). Our studies have provided a firm ground to develop a mechanistic understanding of how NSP2, in concert with NSP5 and VP1, may facilitate genome replication and packaging.

The structure of the NSP2 monomer (**Figure 2**) displays two distinct domains: an amino-terminal domain and a carboxyl-terminal domain. A characteristic feature of the monomer is a 25-angstrom deep cleft between both domains.

While the amino-terminal domain exhibits a fold not seen in any other protein as yet, the carboxyl-terminal domain surprisingly, despite any noticeable sequence homology, exhibits a fold that is observed in the Histidine Triad proteins, a family of ubiquitous cellular proteins that hydrolyze nucleotides. This suggests that the cleft in NSP2 might correspond to the active site for NTP hydrolysis.

The RNA binding and nucleic acid helix destabilizing activities of NSP2 may require the formation of the octamer. In particular, prominent grooves, lined by positively-charged residues, at the sides of the octamer are ideal for binding to single-stranded RNA. The proposed NTP binding sites are located on either side of the groove.

Although NTP hydrolysis is not directly linked to either the RNA binding or the helix destabilizing activity of NSP2, nucleotide binding into the cleft may alter the conformation of the monomer and affect octamer-RNA interactions. Such changes may facilitate transfer of the bound RNA through the polymerase during replication, resulting in concurrent synthesis of duplex RNA and its packaging into assembling progenitor viruses.

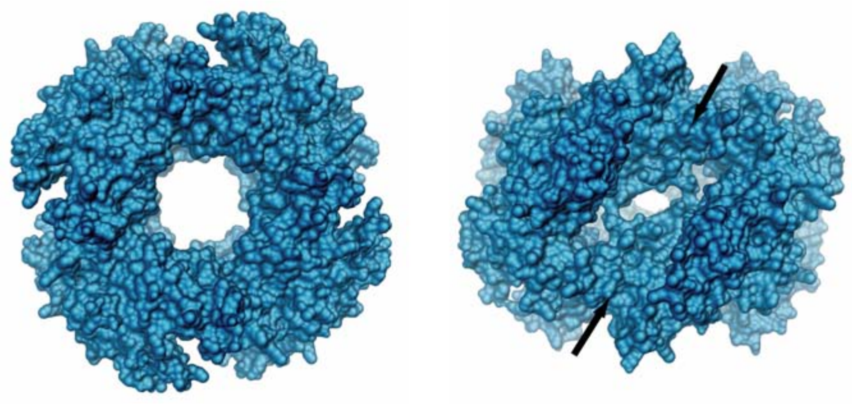


Figure 1. *Surface representations of the octameric part of the non-structural protein 2 (NSP2). (Left): View along the 4-fold axis of the octamer's 4-2-2 symmetry. (Right): View along one of the 2-fold axes. The groove in the octamer is shown by a pair of arrows.*

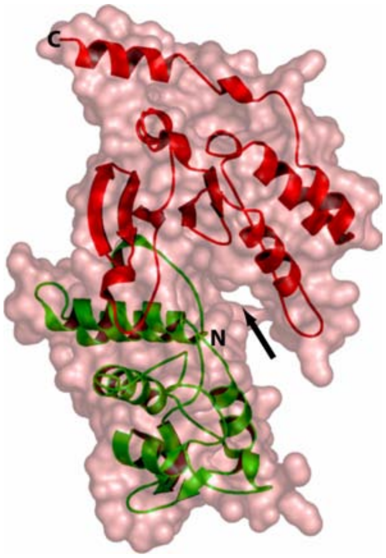
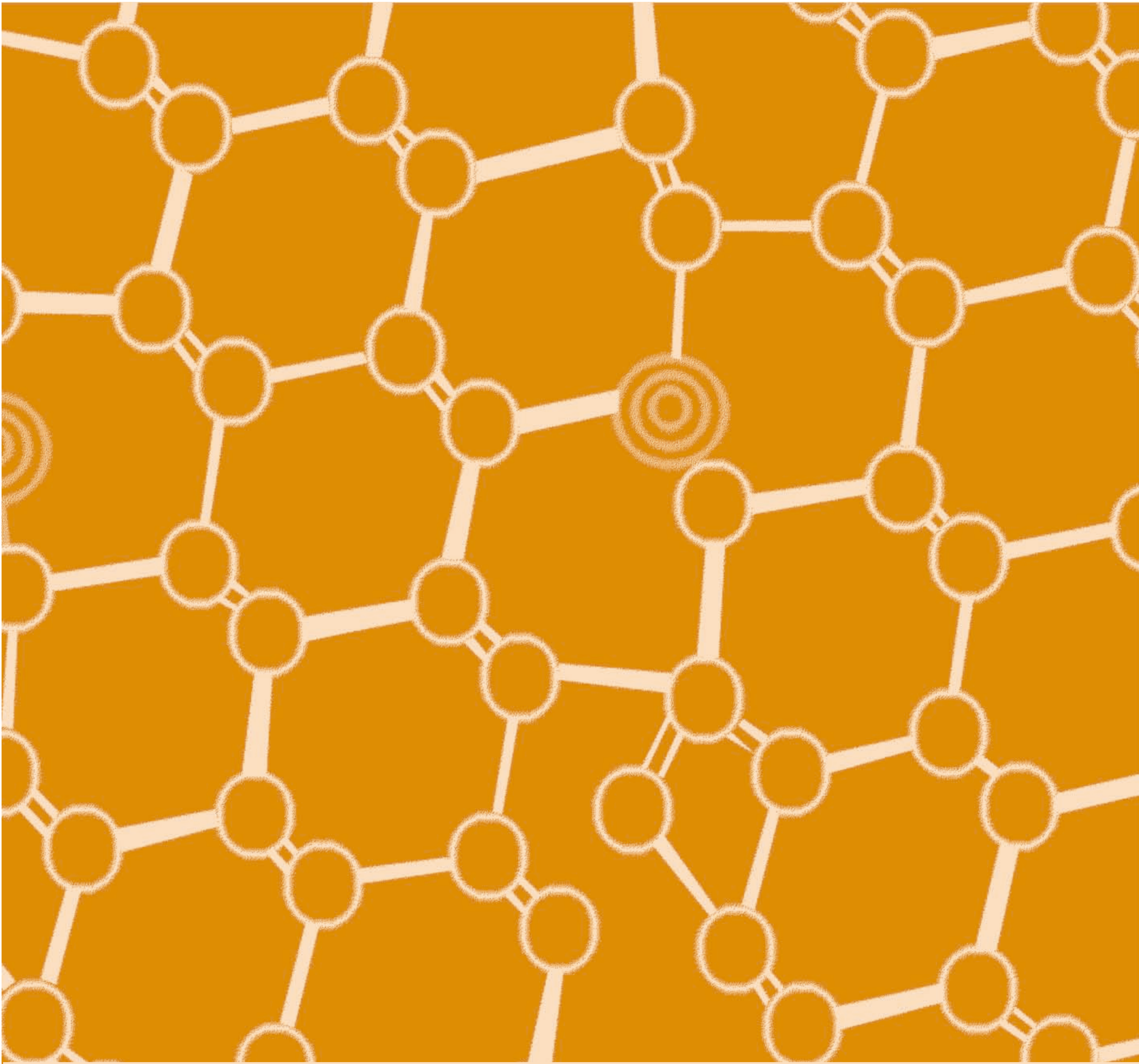


Figure 2. *X-ray structure of the monomeric subunit of the non-structural protein 2 (NSP2). The amino- and carboxyl-terminal domains are shown in green and red, respectively. The cleft between the domains is indicated by an arrow.*



Surfaces, Interfaces and Nanomaterials

At the NSLS, BNL Scientists Create Stripes, Islands, and Towers

Watching someone align bricks on top of each other to build a wall or a flat surface can get routine. But watching as atoms align themselves like bricks - which can be done by using the intense x-ray light at the NSLS - can lead to seeing intriguing new structures, such as stripes and towers, that may result in improved catalysts, compounds used to accelerate chemical reactions.

Doon Gibbs

The "bricklayers" at the NSLS are BNL physicists Doon Gibbs, Hubert Zajonz and their colleagues. While looking at the properties of bimetallic catalysts - compounds made of two metals that are known to be better catalysts than simple metals are - they were building copper layers, atom by atom, on ruthenium. Suddenly, they noticed that the copper atoms were not simply piling up but had started to make new structures.

At first, they saw that when a single copper layer is deposited, it does not settle as it would in bulk copper. Rather, the atoms adopt the same structure as the ruthenium underneath. "The copper atoms go exactly where the ruthenium atoms would have gone if you were depositing ruthenium instead of copper on the ruthenium surface," Gibbs says.

The surprise came when the scientists added more copper atoms, forming a second layer. This time, the atoms rearranged in domains, each taking one of two possible atomic configurations.

"We found that the atoms switch back and forth in two possible configurations in a regular way," says Gibbs. "We see a periodic distribution of stripes, each stripe corresponding to one configuration or the other."

So the researchers kept adding more copper to see what happens when more layers form on ruthenium.

"We started to see something really bizarre and fun: we started to get islands," Gibbs says.

No matter how much copper was deposited on top of the two layers of copper, no new layers formed.

"Little by little, the copper atoms create columns of copper, and, when you add more copper atoms, they run across the surface, jump up on one of the columns, and somehow climb up to the top," Gibbs says. "We end up with a structure in which there is a two-layer film that covers much of the surface, together with these columns of bulk copper sticking up like big buildings."

Gibbs and his colleagues then decided to grow layers of silver instead of copper to see if stripes and towers appeared again. This time, two layers of silver produced more convoluted structures, still with two different configurations.

"Instead of nice stripes following one another, we get little patches of one configuration or the other," Zajonz says. "It may be possible to grow new kinds of nanostructures on these patches, which would change the catalytic properties of the silver-ruthenium compound." The scientists are developing models to understand the origin of such unusual structures. An important parameter is the difference between the interatomic distances in copper or silver and ruthenium.

"The distances between bulk copper atoms is smaller than that between ruthenium atoms," explains Gibbs. "So, when you put a single

Hubert Zajonz

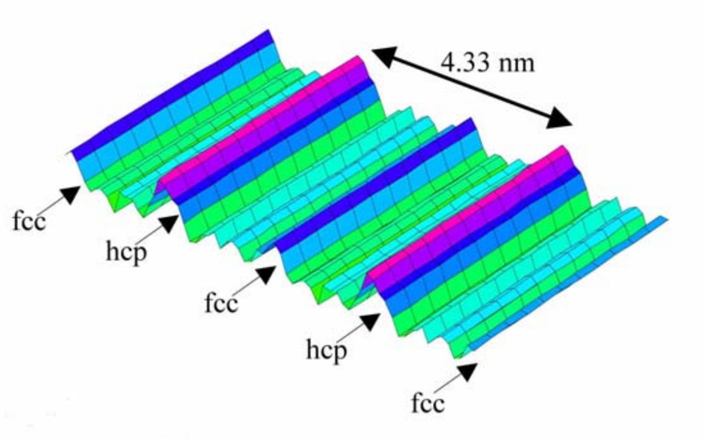
layer of copper onto a ruthenium surface, the distance between the copper atoms is expanded because they are pulled apart. But they want to get closer together, which leads to stripes.

"For silver atoms, it is the opposite," he adds. "The distance between bulk silver atoms is larger than that between ruthenium atoms. So, when you put a single layer of silver on ruthenium, the silver atoms are squeezed and they tend to move apart from each other, creating different kinds of structures."

Gibbs finds the structures "fascinating and beautiful." The scientists are now growing other metals on ruthenium and refining their models for a better understanding of the behavior and catalytic properties of these intriguing structures.

- Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - March 1, 2002.]



When two layers of copper atoms are deposited on ruthenium, the relative distances between the copper atoms are larger than if they were part of bulk copper. The figure shows the relative strain undergone by the copper atoms, which creates two kinds of structures alternately distributed in parallel with each other to form "stripes." One structure is called hcp (hexagonal close packed) and fcc (face centered cubic).

Scientists Create New Material With Varying Densities of Gold Nanoparticles

Material could be used to make better filters, more efficient sensors, and faster catalysts

Jan Genzer

Rajendra Bhat

Dan Fischer

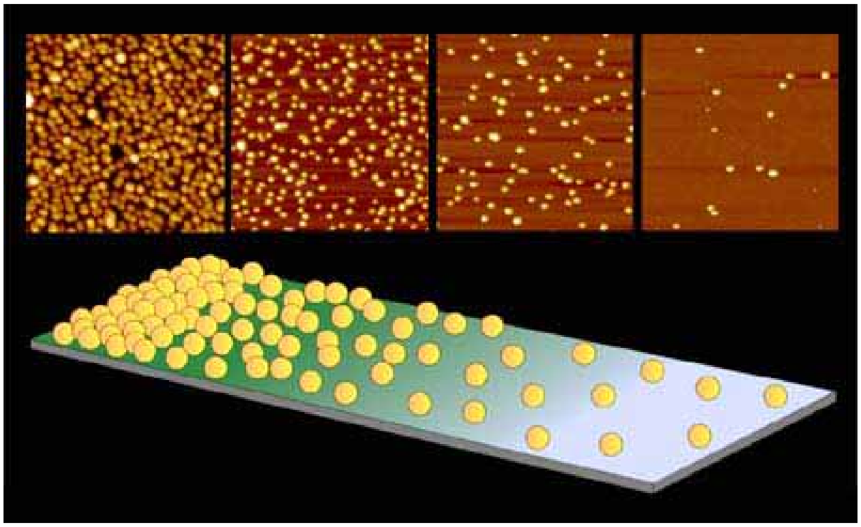
For the first time, scientists have created a material with a gradient of gold nanoparticles on a silica covered silicon surface using a molecular template. The material, which was developed at North Carolina State University (NCSU) and tested at the NSLS, provides the first evidence that nanoparticles — each about one thousand times smaller than the diameter of a human hair — can form a gradient of decreasing concentration along a surface. A description of the material appears as the cover story in the July 23, 2002 issue of *Langmuir*.

"This material promises to be the first in a series with many applications in electronics, chemistry, and the life sciences," said Rajendra Bhat, a Ph.D. student from NCSU and the lead author of the study. Bhat worked with Jan Genzer, a chemical engineering professor at NCSU, and Daniel Fischer, a physicist from the National Institute of Standards and Technology (NIST). To build the material, the scientists first prepared a very thin layer of organosilanes, sticky molecules with a head and a tail, on a rectangular surface of silica.

"The head glues to the surface, while the tail sticks out, acting like a hook waiting for a gold nanoparticle to attach to it," explained Genzer, leader of the NCSU team. The molecules, emitted vertically in the form of a vapor by a source close to one side of the surface, slowly fell on it with decreasing concentration as the distance from the source increased, thus creating a gradient to serve as a molecular template.

The next step was to dip the material in a solution containing the gold nanoparticles, each of which was coated with a negatively charged chemical. In the solution, the tails of the organosilane molecules took on a positive charge, so the negatively charged gold particles attached to the oppositely charged tails underneath.

To visualize the gradient of gold particles, Bhat and his colleagues used an atomic force microscope, in which a tiny needle moves along the surface, following its bumps



Images of gold nanoparticles attached to the silica surface at different distances from the most populated end of the substrate. As the distance increases, the number of particles decreases, revealing a particle gradient. Bottom: Simplified representation of the material showing particles in decreasing concentration along the surface.

and valleys to reveal its topography. To look at the gradient of the organosilane molecules, the scientists used a technique called near-edge x-ray absorption fine structure (NEXAFS). In NEXAFS, extremely intense x-ray light is sent toward the material, and the electrons emitted by the material and collected with a sensitive detector provide information about the concentration of the organosilane molecules on the surface.

"We needed to confirm that both the gold particles and the sticky groups followed the same underlying gradient template," Bhat said. "The results from both techniques were expected to coincide if the particles were attaching to the underlying layer of sticky molecules. Our results show exactly that."

"The distinguishing feature of our approach is that the particles follow a pre-designed chemical template provided by the organosilane sticky groups," said Genzer. "The ability to manipulate the underlying template allows us to prepare gradient structures of nanoparticles with varying characteristics."

The main advantage of the gradient structure is that large numbers of structures can be combined on a single substrate and used for high-throughput processing. It might, for example, save time for chemists testing clusters of nanoparticles used as catalysts — chemicals actively sought by the chemical industry to create new, less polluting sources of energy. "Clusters made of different numbers of nanoparticles could be put on a single surface, and scientists could test this surface only once in a chemical reaction, instead of having to run each cluster separately through the reaction," Fischer said. The material could also be used as a sensor to detect species that have specific affinities for nanoparticles, or as a filter to select particles of given sizes.

Bhat and his colleagues are now exploring the properties of similar materials, with different "sticky" substances and nanoparticles. "This research is so new that we are still thinking of potential applications for these materials," he said.

-Patrice Pages

Nanoscale Crystallography Reveals Structural Details

Understanding the properties of nanoscale materials may allow scientists to manipulate these properties to produce new nanomagnets, nanocatalysts, and composites with better optical properties. But such applications require detailed knowledge of the materials' atomic level structure.

"Without a structure, you are without a road map," said Thomas Vogt, BNL Physics Department. Funded by DOE's Office of Basic Energy Science and the National Science Foundation, Vogt and scientists from Michigan State University (MSU), led by MSU physicist Valeri Petkov, have demonstrated a technique that allows them to decipher such fine-level nanostructures.

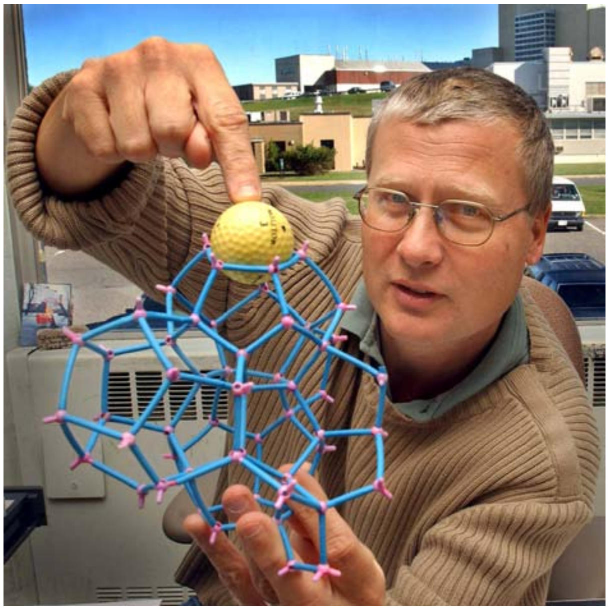
The researchers' analysis of a material composed of cesium ions trapped inside nano-sized pores of the silicon-oxide zeolite $Si_{32}O_{64}$ is described in *Physical Review Letters* of August 12, 2002. This material is also the first example of a room-temperature inorganic "electride," a stable separation of positively charged cations and electrons, with properties determined by the topology of the pores in the host matrix.

Nanoscale structures — made of particles 1,000 times smaller than the diameter of a human hair — are so difficult to decipher because they lack the long-range order and symmetry of perfectly crystalline materials.

"This work shows that, even with a very low degree of order, at synchrotrons such as the NSLS, and using the right techniques, we can determine nanoscale structures. And with structural understanding, we can begin to predict properties, and perhaps begin to manipulate them for useful applications," said Vogt.

-Karen McNulty Walsh

[Editor's note: Reprinted with permission from the BNL Bulletin - October 18, 2002.]



Tom Vogt of BNL's Physics Department illustrates the location of a cesium atom (golf ball) in the nanopores of a zeolite — a structure determined using methods of nanocrystallography

New Technique Reveals Structure of Films With High Resolution

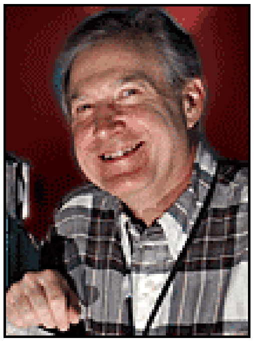
Scientists have developed and tested a new imaging technique that reveals the atomic structure of thin films with unprecedented resolution. For the first time, the technique has shown very precisely how the atoms of the first layers of a film rearrange under the action of the substrate on which the film is grown. The results of the study are reported as the cover story of the October, 2002 issue of *Nature Materials*.

"This technique directly provides a very precise image of atomic positions within a film and at the interface between a film and a substrate," says Ron Pindak, a physicist at the NSLS and one of the authors of the study. "With the current growing interest in the study of nanomaterials, which are the size of a few atoms, this technique will probably be key in devising such materials and understanding their properties."

Thin films are currently used in many technologies, including electronic chips, coatings, and magnetic recording heads. To improve the properties of these materials and create even thinner structures – such as smaller electronic chips – scientists are now trying to understand how the films interact with the substrate on which they are grown.

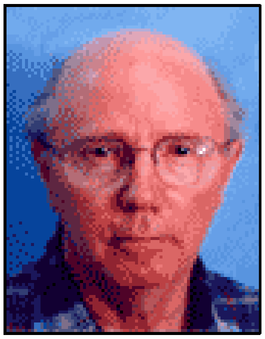
"When you build a film on a substrate, the positions of the atoms of the film are slightly shifted, and some of these shifts can be very small," says Roy Clarke, a physicist at the University of Michigan in Ann Arbor and another author of the study. "So it is important to be able to explain how these films behave at the atomic level."

By building upon traditional x-ray diffraction, the newly devised technique provides such information. In this technique, x-rays are projected onto the film and the substrate pattern, which is then used to determine the positions of the atoms inside the film. The diffraction pattern of thin films is composed of ridge-like features called "Bragg rods," hence the name of the technique: coherent Bragg rod analysis (COBRA).



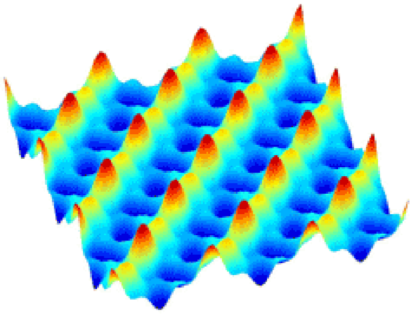
Ron Pindak

Roy Clarke



Yizhak Yacoby

The COBRA technique determines two key properties of the diffracted x-ray waves: their intensity and their phase, which describes the shift in position between the incident and diffracted x-ray waves. Though the amplitude is easily determined from the diffraction pattern, the phase is usually more difficult to determine, which is just what COBRA does.



Electron density map of gadolinium atoms (located around the peaks) in the gadolinium oxide film, obtained by using the COBRA imaging technique.

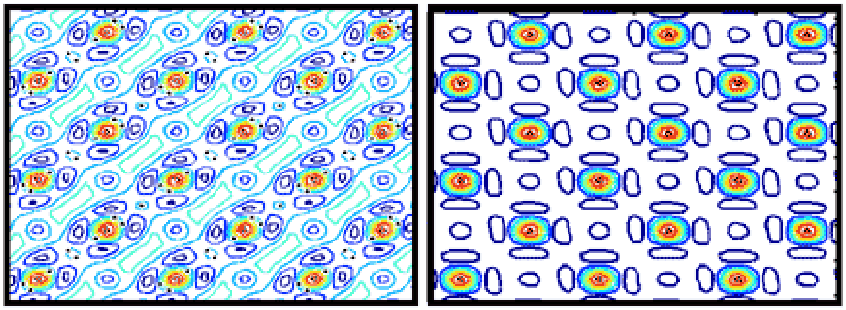
"Key to the COBRA technique is a new approach to determining the phase of the diffracted x-ray waves," says Yizhak Yacoby, physicist at the Hebrew University in Jerusalem and lead author of the study. "Unlike traditional x-ray diffraction techniques, COBRA does not rely on a priori guesses about the structure of the film and the substrate, and we do not need to prepare the sample in a special way – as with a transmission electron microscope."

Yacoby, who started developing the technique four years ago, first applied it to known structures by using x-rays produced at the NSLS, which allowed him to refine the technique. In their recent study, Yacoby and his collaborators applied the technique to a film made of gadolinium oxide grown on a gallium arsenide substrate using brighter x-rays at the Advanced Photon Source (APS) at Argonne National Laboratory in Illinois. Key to the successful data collection were two APS beam lines that Clarke and Edward Stern, a physicist at the University of Washington in Seattle, perfected for the past five years.

The researchers made unexpected observations. They noticed that two thirds of the gadolinium atoms in the first few layers of the film adjust to match the positions of the atoms in the substrate. The researchers also discovered that the structure of the first layers of the film mimics very closely the substrate's structure, while the atoms in the layers farther away from the substrate are arranged more like those in the bulk form of gadolinium oxide. The layer stacking of the film also appeared to mimic very closely the substrate's structure.

The scientists now intend to investigate the properties of various other films. Yacoby, who has already submitted patents for the COBRA technique, is confident that it will have many applications in the design of electronic devices based on thin films, the self-assembly of layers made of metal oxides used in catalysis, and the study of films made of large organic molecules, such as proteins.

-Patrice Pages



Electron density map of one of the layers of the gadolinium oxide film close to the gallium arsenide substrate (left) and a layer in the substrate (right), by using the COBRA imaging technique. A comparison of both maps shows that the gadolinium atoms (around the yellow-red peaks) rearrange so that the maps mimic each other.

Molecular Film on Liquid Mercury Reveals New Properties

A team of scientists from BNL, Harvard University, and Bar-Ilan University in Israel have grown ultrathin films made of organic molecules on the surface of liquid mercury. The results, reported in the November 15, 2002 issue of *Science*, reveal a series of new molecular structures that could lead to novel applications in nanotechnology, which involves manipulating materials at the atomic scale.

Growing molecular films on liquid surfaces is part of an ongoing activity by Brookhaven scientists to create nanomaterials, which are a few billionths of a meter in thickness. Ultrathin films are becoming increasingly important for fast-developing applications, such as faster and smaller electronic and magnetic devices, advanced biotechnological membranes, and controlled drug release in the human body. The Brookhaven team is a leader in the field of liquid surface-supported film growth, with expertise gained over the past 20 years.

"When you grow a film on a solid surface, the molecules of the film tend to interlock with those of the underlying support," says Benjamin Ocko, the Brookhaven physicist who participated in the study. "But an underlying liquid surface is not ordered and provides an ideal setting for studying ultrathin states of matter without the complications of the solid support."

Ocko and his colleagues first filled a small tray with liquid mercury and then deposited on the surface a nanometer-thin film of stearic acid, an organic waxlike material that is a common component of cell membranes. Since stearic acid is not soluble in mercury, it floats on the surface.

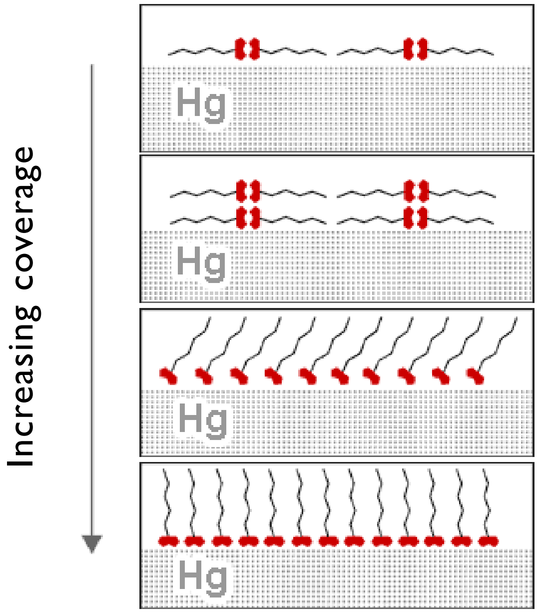
Ben Ocko

To see how the molecules of the film organize on the surface, the scientists measured how x-rays produced by the NSLS scattered off the ultrathin molecular film. Key to the study was a unique instrument used for tilting the x-rays downward onto the liquid mercury surface, which was developed by Peter Pershan, a physicist at Harvard and one of the study's authors, along with the Brookhaven team.

The scientists discovered that, as the number of molecules deposited on the surface increased, they formed four distinct patterns. "First, when a few molecules are deposited, they tend to take as much space as they can, by lying on the surface," explains Henning Kraack, a physics Ph.D. student from Bar-Ilan and the study's lead author. "When more molecules are added, a second layer of molecules lies on top of the first one.

"Then, as even more molecules are deposited," Kraack continues, "they 'stand up' to leave more space to neighboring molecules, allowing them to densely pack in one layer. But even then, before standing up straight, the molecules are first tilted to the side, and stand up completely only when they are 'squeezed' by other molecules that 'elbow their way through.'"

These observations came as a surprise, since previous studies have shown that, when stearic acid molecules are deposited on water — the only other liquid support stud-



This schematic drawing shows how the stearic acid molecules of the film rearrange as they are added onto the surface of the liquid mercury support.

ied so far — they only stand up on the surface. "Patterns in which molecules lie flat on a liquid surface have never been observed before," Kraack says.

Moshe Deutsch, a physicist at Bar-Ilan and one of the authors of the study, notes that because the liquid mercury does not seem to influence too much the way the stearic acid molecules assemble, "growing films on a liquid surface is like growing them without support at all." It might be possible to choose a film pattern, he adds, simply by selecting the appropriate molecular coverage.

"This work shows that without an underlying lattice, we can control film growth," Deutsch says. "By growing other molecules on a liquid support, we will be able to control the size and properties of other films, and thus tailor them for different applications, in particular their use in nanoelectronics and nanosensor technology."

-Patrice Pages

BEAMLINE
X27C

PUBLICATION
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Tailor-Made Polymer Nanocrystals

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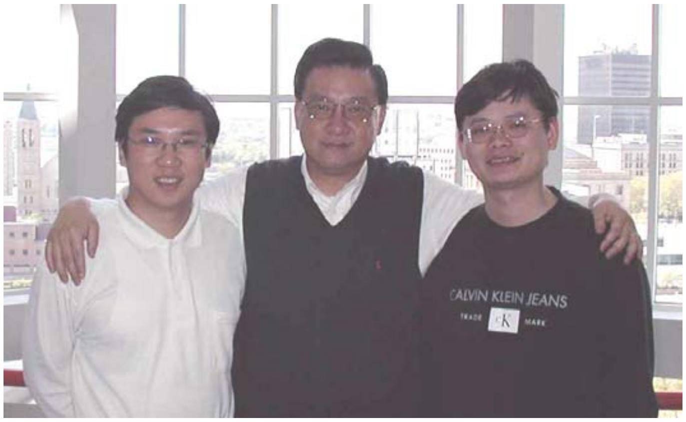
[1]University of Akron; [2]Massachusetts Institute of Technology; [3]Charles Sadron Institute, Strasbourg, France; and [4]State University of New York at Stony Brook

Chemicals made of chain molecules called block copolymers can self-assemble into various ordered structures, such as lamellae, cylinders, and spheres. Using self-assembled crystalline-amorphous block copolymers, scientists have grown ordered polymer nanocrystals, which are expected to be used in advanced optical applications. X-ray scattering studies at the NSLS revealed that, simply by varying the crystallization temperature, the tiny crystals can orient in different directions with respect to the interface between the crystalline and amorphous domains in the copolymer. Crystal growth can also be tailored in specific directions by conforming to the geometry of the block copolymer nanophases at high temperature.

Polymers in a confined space of a few nanometers, or billionths of a meter, are expected to exhibit a different structure, stability and morphology than ordinary bulk polymers. For instance, the polymers can be confined in mesoporous inorganic materials, which are made of pores that are 2 to 50 nanometers in diameter. But if no specific interactions occur between the inorganic material interfaces and the crystalline polymers, it is very difficult to drive polymers into the nanopores.

To avoid this constraint, a team of scientists from the University of Akron in Ohio, the Massachusetts Institute of Technology, the Charles Sadron Institute in Strasbourg, France, and the State University of New York in Stony Brook has been successfully using mesophases (typically ordered phases with submicron sizes) of a crystalline-amorphous diblock copolymer – which form by simultaneous self-assembly process – to study confined polymer crystallization in nanospaces.



Authors (from left): Lei Zhu, Stephen Z.D. Cheng and Ping Huang.

The copolymer, called polystyrene-b-polyethylene oxide (PS-b-PEO), is made of polystyrene (PS), an amorphous polymer with a glass transition temperature of 100 degrees Celsius, and polyethylene oxide (PEO), a crystalline polymer with an equilibrium melting temperature of 69 degrees Celsius. Because of their distinct chemical nature, PS and PEO strongly segregate from each other, so after the PS matrix glassifies, the crystallized PEO blocks are completely confined within the nanospaces of the copolymer.

The PEO nanocrystals were investigated on macroscopically-oriented (a single domain in microns or even milimeters) PS-*b*-PEO single crystals using simultaneous small and wide-angle X-ray scattering (SAXS and WAXS) techniques at the NSLS's Advanced Polymer beamline X27C. The detailed experimental setup is displayed in **Figure 1**.

High-resolution SAXS revealed that the PS-*b*-PEO sample with 0.39 percent of PEO in volume exhibits a hexagonally perforated layer (HPL) phase, where the PS and PEO layers are alternating and each PEO layer is made of two-dimensionally packed PS perforations, as shown in **Figure 2**.

The researchers also studied how the nanolayers and nano-PS cylinders in the PEO layers affect the crystallization behavior of the PEO blocks. The WAXS results showed that the PEO crystal orientation in the HPL phase changes with the crystallization temperature. At low temperatures, the PEO crystals preferentially arrange parallel to the layer plane (**Figure 2a top**), while above zero degree Celsius, the crystals become inclined with respect to the layer plane (**Figure 2b top**). The team also noticed that the ribbon-like PEO crystal grows along the arrays of the hexagonal PS perforations (**Figure 2b bottom**).

Surprisingly, the WAXS experiments showed that crystal orientation forms in an early stage of crystallization with a crystallinity of ~ 7 weight percent. The early stage crystal orientation clearly reflects the confined effect on polymer crystallization in nanospaces.

These results not only show the scientists' ability to manipulate polymer crystalline morphologies confined in nanospaces, but also reveal the details of polymer crystallization on the nanoscale. Further study will investigate the crystal orientation mechanism and explore polymer crystallization in compounds with more elaborate mesophases, such as double gyroids.

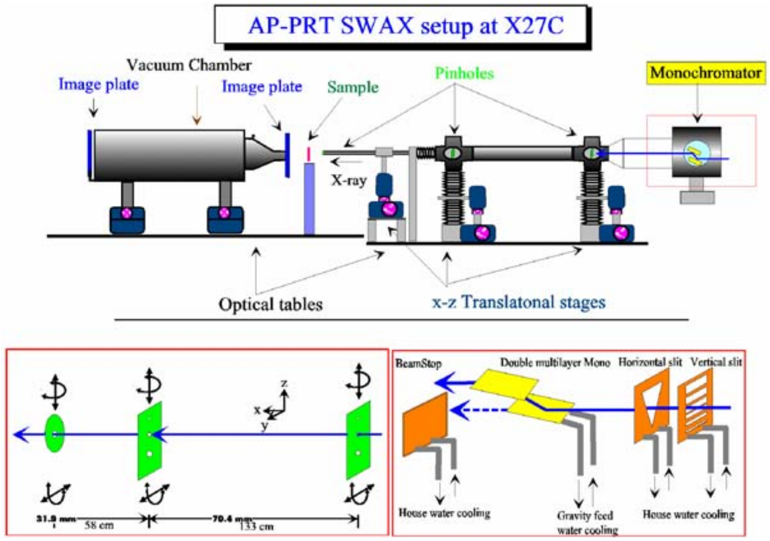
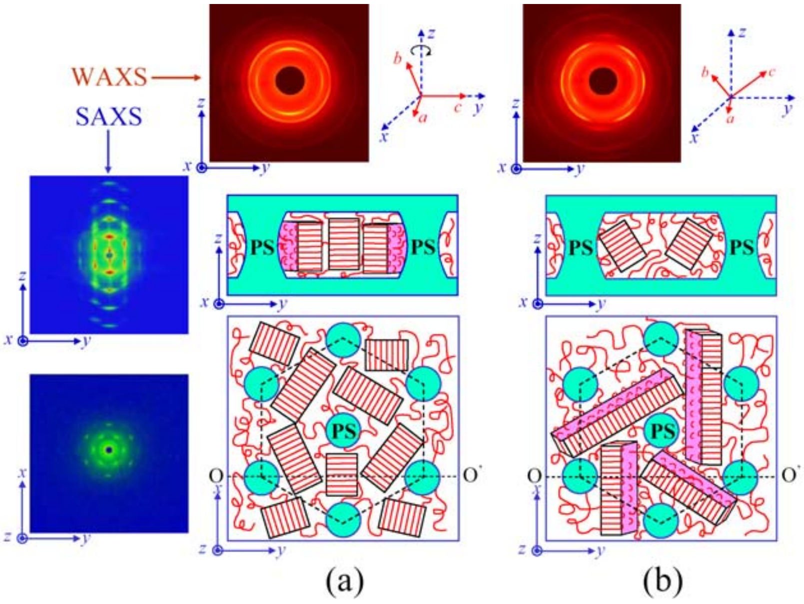


__Figure 1__. Experimental setup for simultaneous SAXS and WAXS at beamline X27C at NSLS.

__Figure 2__. Nano-tailored PEO crystalline morphologies at (a) -50 °C ≤ T_c ≤ -10 °C and (b) T_c ≥ 0 °C. A nanophase is defined by x, y, and z, and crystal unit cell axes are a, b, and c. The top view of the HPL phase is shown at the bottom, and the side view at the top.



BEAMLINE
X23B

PUBLICATION
M.E.Van der Boom, P. Zhu, G. Evmenenko, J.E. Malinsky, W. Lin, P. Dutta, T.J. Marks, *Langmuir* **18**, 3704 (2002).

FUNDING
National Science Foundation (NSF), ARO/DARPA, Nanovation Technologies, U.S. Department of Energy, Helen and Milton Kimmel Center for Molecular Design.

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Nanoscale Self-Assembly of Thin-Film Molecular Materials for Electro-Optic Switching

M.E. van der Boom[1], P. Zhu[2], G. Evmenenko[2], J.E. Malinsky[2], W. Lin[2], P. Dutta[2], and T. J. Marks[2]

[1]Weizmann Institute of Science in Rehovot, Israel; [2]Northwestern University in Evanston, Illinois

Scientists from Northwestern University in Evanston, Illinois and the Weizmann Institute of Science, Rehovot, Israel, have devised a two-step assembly technique to make highly ordered, intrinsically acentric organic materials which can be integrated into electro-optic (EO) and related devices, such as light modulators and switches. The scientists have shown that the self-assembled photonically/electronically functional materials are competitive in terms of EO responses with the highest efficient polar films reported to date, and are more efficient than inorganic systems, such as $LiNbO_3$.

Milko E. van der Boom

Tobin J. Marks

Forming nanoscale organic films and integrating them into semiconductor electronics and all-organic microphotonic circuits has stimulated intense academic and industrial research, but progress is hampered by the lack of device-quality functional molecule-based thin films, driving the need for new reliable film-growth methods.

A general applicable method has been developed generating thermally robust multilayered materials. This new synthetic approach involves two alternating deposition steps, as shown in **Figure 1**. First, monolayers (one-molecule-sized layers) of chromophores are covalently bound on hydrophilic substrates (step (i)). The siloxy removal step (ii) renders the surface hydrophilic, thus allowing the rapid build-up of a covalently-bound siloxane-based capping layer. The resulting films are intrinsically acentric, so no post-deposition steps such as high-voltage poling to align the molecular building blocks are necessary, as in other film growth techniques.

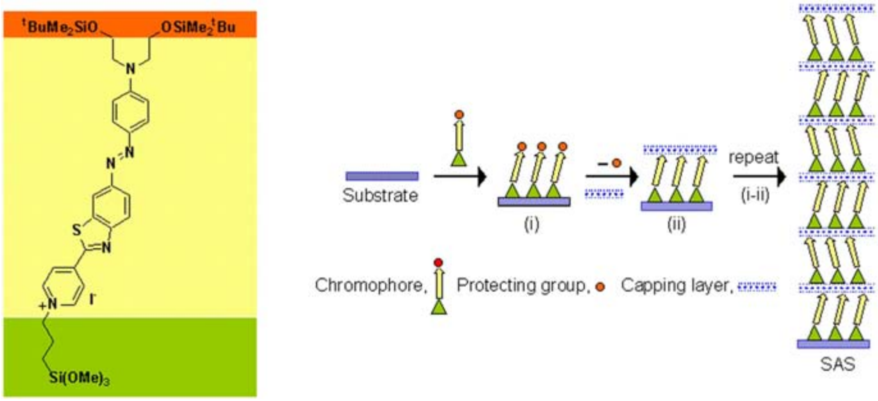

***Figure 1**. Two-step layer-by-layer self-assembly process generating intrinsically acentric superlattices.*

We have developed a film growth process based on chemically reliable steps, amenable to automation-by using a single reaction vessel or dip-coating - and allowing an excellent control of material properties - which is of great interest for optical telecommunications and electronic applications. The high degree of control over film dimensions, texture, and properties has been unambiguously demonstrated using various physicochemical analytical tools, including second harmonic generation measurements and synchrotron x-ray reflectivity measurements (XRR) performed at NSLS beamline X23B (**Figure 2**).

The XRR experiments afforded crystal-clear structural information on the chromophore density (~50 Å²/chromophore), film thickness (~2.8 nm for each chromophore + siloxane-based capping layer), and surface morphology. The robust capping layer is ~8 Å thick. The streamlined two-step assembly process shown in **Figure 1** could be extended to a wide range of molecular building blocks, and become a major synthetic route for the formation of various functional sub-micrometer-sized solids with superb control of material characteristics at the nanoscale level. This assembly process is also part of an ongoing investigation aimed at creating "all-organic" electro-optical modulators (**Figure 3**).

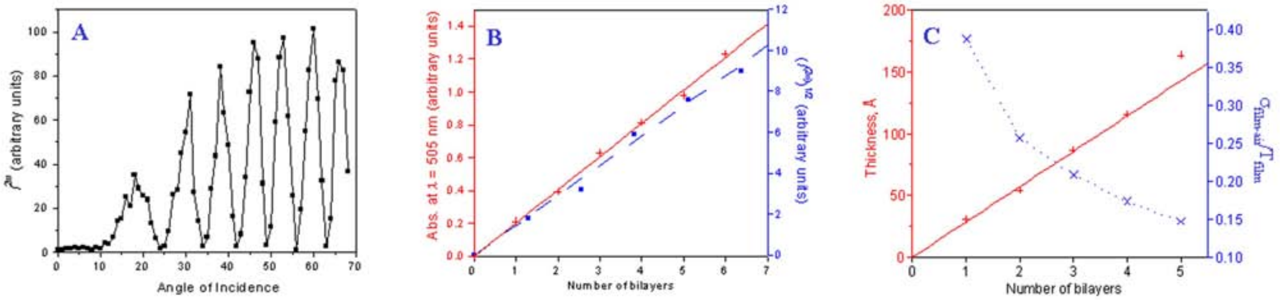

***Figure 2**. (A) Second harmonic generation response at λ$_o$ = 1.06 m as a function of fundamental beam incident angle from a float glass slide having a polar monolayer on either side. (B) Optical transmission and second harmonic generation as a function of the number of bilayers. Left y-axis: absorption at λ$_{max}$ = 505 nm (+). Right y-axis: square root of the SH intensity (•). (C) Specular X-ray reflectivity measurements. Left y-axis: film thickness (Å) as a function of the number of bilayers (+). The solid line is the fit by linear regression for 1-4 bilayers, indicating T = 28.6 ± 0.6 Å × n. Right y-axis: relative film roughness, $\sigma_{film-air}$/T film, as a function of the number of bilayers (×). The dotted line is drawn as a guide to the eye.*

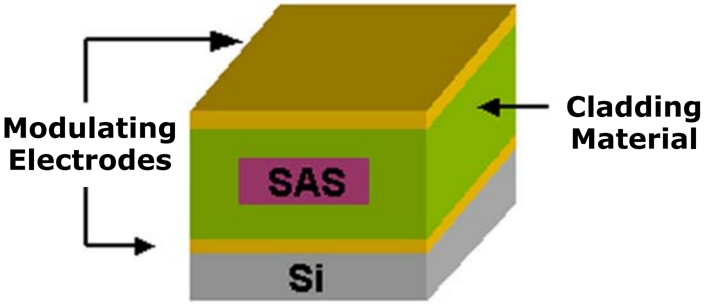

***Figure 3**. Schematic view of a prototype "all-organic" electro-optical modulator based on intrinsically acentric self-assembled superlattices (SAS). Commercially available polymers such as Cyclotene™ and/or Cytop™ can be used as cladding layers.*

BEAMLINE
X16A

PUBLICATION
I. K. Robinson, P.A. Bennett and F. J. Himpsel, "Structure of Quantum Wires in Au/Si(557)," *Physical Review Letters*, **88**, 096104-1 (2002).

FUNDING
U.S. Department of Energy
National Science Foundation

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Structures of Quantum Wires in Gold/Silicon(557)

I. Robinson[1], P. Bennett[2] and F. Himpsel[3]

[1]University of Illinois; [2]Arizona State University; and [3]University of Wisconsin

Scientists working at NSLS beamline X16A have revealed the full three-dimensional structure of nanowires formed by deposition of gold on a stepped silicon surface using surface X-ray diffraction. The result is important because of the unusual band-dispersion properties previously reported for this system. The result is interesting because the gold atoms are neither imaged by STM, nor are involved directly in the electronic states responsible for the metallic behavior of the surface.

Peter Bennett (left) and Ian Robinson standing in front of the X16A beam station at the NSLS.

A "quantum wire" is a one-dimensional wire created by arranging metal atoms that touch each other in an exact straight line. The wave functions describing the electrons present in such a wire are localized in the directions perpendicular to its axis but can form solitons – waves that propagate with little loss of energy and retain their shape and speed after colliding other such waves – in the direction along the wire. The Pauli exclusion principle forbids the free propagation of these solitons along the wire and leads to a one-dimensional metal with highly unusual properties.

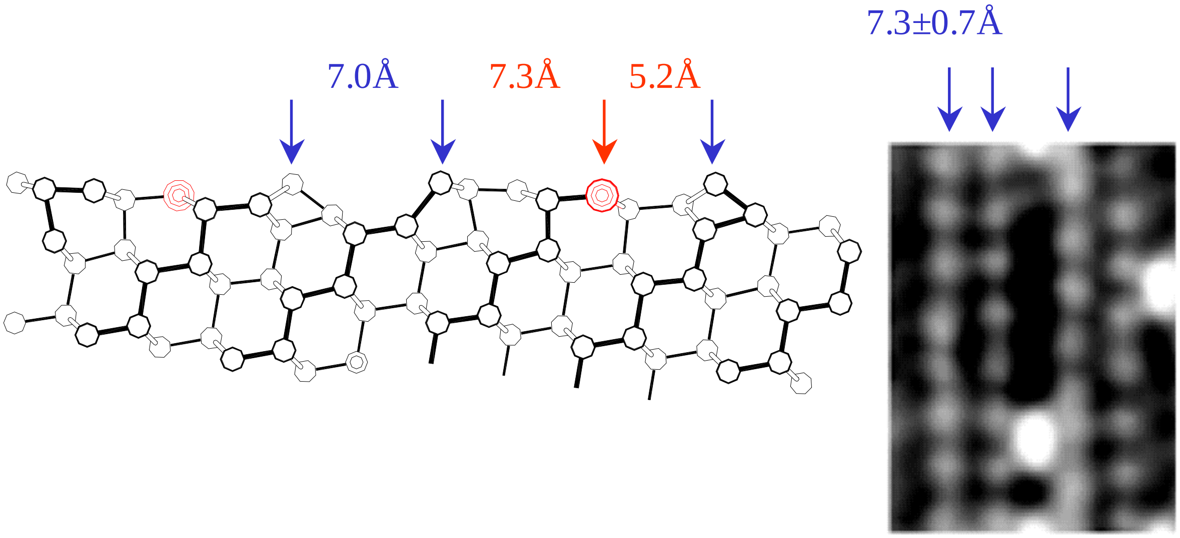
We have investigated a quantum wire in a candidate system for spin-charge separation, due to the formation of a Luttinger liquid, in which electrons have long-range interactions between them.

We used two techniques. The first is the scanning tunneling microscope (STM), which uses electron tunneling to map the positions of individual atoms and defects in a surface. Typical defects are vacancies, steps and kinks. We also used surface x-ray diffraction, a traditional structure-determining method that is less useful for looking at defects, but provides accurate three-dimensional structures.

We measured a large set of crystal truncation rods to solve a crystalline structure formed by evaporation of 20 percent of a monolayer of gold onto a silicon(557) surface using the surface x-ray diffraction facility at NSLS beamline X16A.

The derived structure, shown in the figure, reveals four main features. First, the row of gold atoms sits in the middle of the terrace between adjacent steps in the substrate, while we expected that the metal would have directly lined the step edge instead. Second, an almost ideal substitution takes place in the upper bilayer, resulting in bond lengths between gold and silicon of 2.47 angstrom and 2.35 angstrom, which are indistinguishable from the bond length between two silicon atoms, which is 2.35 angstrom. A gold atom appears to substitute in the silicon lattice with relatively little strain. The third feature is that the step edge reconstructs to form a five-membered ring, but removes a dangling bond. Finally, the figure shows a row of adatoms attached to dangling bonds.

The STM micrograph reveals two lines per unit cell that might be mistaken for wires. The X-ray structure has only a single gold per unit cell. Analysis of the inter-row distances shows that neither of the rows can be gold atoms, which are therefore not visible to STM. The rows correspond instead to the step-edge silicon and the adatom silicon features. Because STM is surface-sensitive, it detects only the outermost electron shells of the topmost layer, whereas surface x-ray diffraction sees the full three-dimensional arrangement of the atom cores.



(Left) Side view of the three dimensional structure of gold nanowires grown on a silicon(557) surface. (Right) The STM image shows two rows of atoms, neither of which corresponds to the single gold atom in the unit cell structure.

Formation of Alkylsilane-Based Monolayers on Gold

T.M. Owens[1], K.T. Nicholson[1], M.M. Banaszak Holl[1], and S. Süzer[2]

[1]University of Michigan; [2]Bilkent University, Ankara, Turkey

A team of chemists from the University of Michigan in Ann Arbor have made a surprising discovery: At room temperature, the chemical reaction of compounds with silicon-hydrogen bonds with gold surfaces yields a new class of alkylsilane($CH_3(CH_2)_nSi$)-based monolayers. The monolayers are reminiscent of the self-assembled monolayers (SAMs) formed by alkanethiols ($CH_3(CH_2)_nSH$). However, they exhibit a different chemical behavior that may prove advantageous in microelectronics and micro-contact printing. The alkylsilane monolayers have been characterized using soft X-ray photoelectron spectroscopy (SXPS) and reflection absorption infrared spectroscopy (RAIRS).

BEAMLINE
U8B

PUBLICATION
T. M. Owens, K. T. Nicholson, M. M. Banaszak Holl, S. Süzer *J. Am. Chem. Soc.* **124**, 6800 (2002).

FUNDING
Dow Corning Corporation; National Science Foundation; Fulbright Fellowship for Sefik Süzer; Alfred P. Sloan Fellowship for M. M. Banaszak Holl; Division of Materials Sciences and Division of Chemical Sciences, U.S. Department of Energy.

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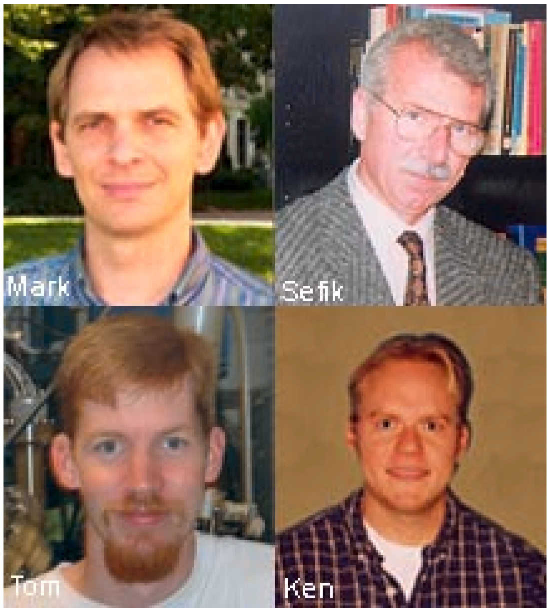
The physical properties of metal surfaces can be modified through the adsorption of small molecules. For example, hydrocarbons modified with carboxylic acid (RCOOH) or thiol (RSH) functional groups have been particularly well studied. In recent years, scientists have shown that the adsorption of alkanethiols ($CH_3(CH_2)_nSH$) on gold surfaces may have many applications, including molecular electronics and micro-contact printing. But these applications have met some limitations due to surface defects introduced by the adsorption process and to degradation caused by atmospheric ozone. We have explored the use of alkylsilanes ($CH_3(CH_2)_nSiH_3$) as a replacement for the thiol groups.

We synthesized monolayers of hexylsilane ($C_6H_{13}SiH_3$), octylsilane ($C_8H_{17}SiH_3$), and octadecylsilane ($C_{18}H_{37}SiH_3$) by exposing gold surfaces to one of the alkylsilanes (**Figure 1**) in an ultrahigh vacuum (UHV) chamber. The gold-alkylsilane samples have been characterized in UHV by reflection absorption infrared spectroscopy (RAIRS) at the University of Michigan in Ann Arbor, and soft x-ray photoelectron spectroscopy (SXPS) at beamline U8B at the NSLS.

The silicon 2p core levels were measured to have a binding energy of -99.8 electronvolts (eV), as shown in **Figure 2A**, suggesting that silicon is bound to the gold surface and that no silicon-hydrogen bonds remain. We measured a full width at half-maximum of 0.4 eV, which is smaller than that observed for bulk, crystalline silicon using the same beamline and monochromator, thus indicating that silicon atoms are in a chemically homogenous environment.

The valence band spectra of the alkylsilane-based monolayers (**Figure 2**) bear a striking resemblance to valence band data of frozen alkanes (C_nH_{2n+2}) of the same length, indicating that the hexyl, octyl, and octadecyl chains have remained intact.

We have not observed a silicon-hydrogen stretch in the RAIRS spectra for alkylsilane monolayers on gold (**Figure 3**). The silicon-hydrogen stretch at 2150 cm^{-1} is the most intense feature observed for liquid alkylsilane (**Figure 3D**). The absence of this feature in the monolayers suggests that no silicon-hydrogen bonds remain after chemical adsorption. The observed carbon-hydrogen stretching modes between 2850 and 3000 cm^{-1}, are consistent with the alkyl (C_nH_{2n+1}) chains being angled away from the surface.



Clockwise from top left: Mark Banaszak Holl, Sefik Süzer, Ken Nicholson, and Tom Owens (lead author).

The combination of SXPS and RAIRS data suggests that when the alkylsilane monolayers bind to the gold surface, all three silicon-hydrogen bonds react with the gold surface and silicon forms bonds to three surface gold atoms, which is unlike single gold-sulfur bonds in structures made of thiol bound to gold.

Also, the alkanethiol and alkylsilane layers exhibit different oxidation behaviors. Oxidation of sulfur atoms causes alkanethiol monolayers to fall apart, while oxidation of the alkylsilane layers knits the silicon atoms together by forming a siloxane network and maintaining the integrity of the alkyl chains and the surface monolayer.

The unique properties of the alkylsilane layers may lend them to applications requiring durable layers and may provide an interesting route for the formation of patterned structures.

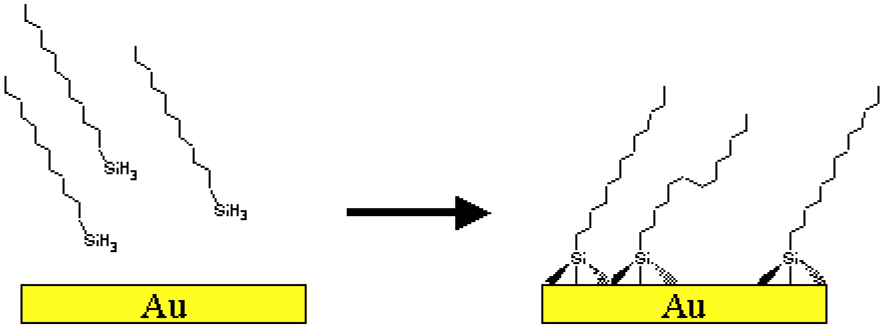
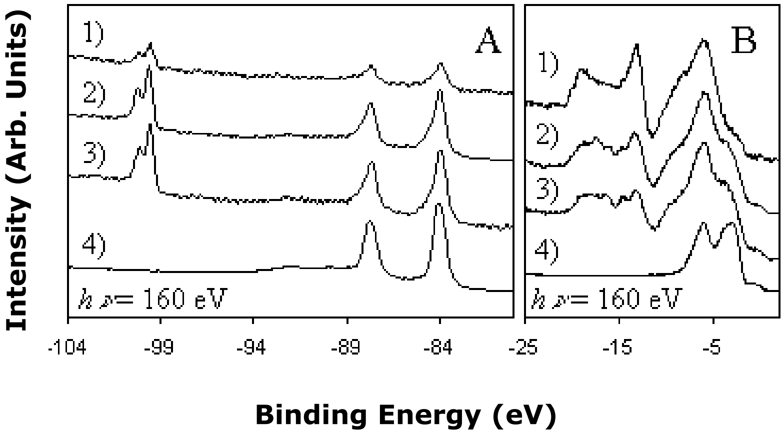


Figure 1. *Schematic representation of alkylsilane (CH$_3$(CH$_2$)$_n$Si) bonding to gold surface.*

Figure 2. *X-ray photoemission spectra (photon energy = 160 eV) of (1) octadecylsilane, (2) octylsilane, and (3) hexylsilane monolayers on (4) gold. (A) Silicon 2p and gold 4f core levels, and (B) valence band.*



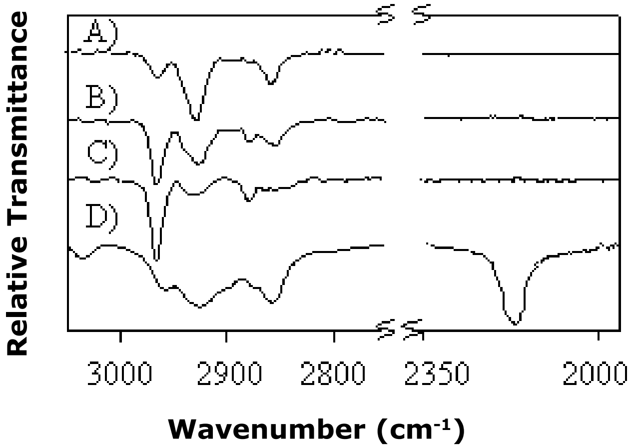


Figure 3. *Reflection-absorption infrared spectroscopy (RAIRS) spectra (3050-2750 and 2350-2000 cm^{-1}) for monolayers of (A) octadecylsilane, (B) octylsilane, and (C) hexylsilane on gold. A solution infrared spectrum of octylsilane (D) is included for comparison.*

BEAMLINE
X10A

PUBLICATION
B.N. Thomas, R.C. Corcoran, C.L. Cotant, C.M. Lindemann, J.E. Kirsch, P.J. Persichini, "Phosphonate Tubules II", *J. Am. Chem. Soc.*, **124**, 1227-1233, (2002).

FUNDING
National Science Foundation

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Phosphonate Tubules

B. Thomas[1], R.C. Corcoran[2], C. Cotant[2], J. E. Kirsch[2], C.M. Lindemann[3], and P. J. Persichini[4]

[1]Louisiana State University, Baton Rouge; [2]University of Wyoming; Laramie; [3]Merck & Company, Whitehouse Station, New Jersey; [4]Allegheny College, Meadville, Pennsylvania

Tubules are stable, hollow, cylindrical crystalline tube-like structures a few micrometers (millionths of a meter) in size, with potential applications in nanofabrication and medical encapsulation. Stringent control of the kinetics of the process by which these tubules self-assemble is necessary to control the dimensions and qualities of the tubules. An alternative control mechanism involves subtle chemical modifications made to the self-assembling molecules. By using x-rays produced at the National Synchrotron Light Source, scientists have studied both control pathways to understand better how subtle changes made to lecithin, a molecule found in egg yolks and the plasma membrane of plant and animal cells, causes rigid, hollow tubules to form.

Scientists are currently studying how to build novel materials the size of a few nanometers (a few billionths of a meter, or the size of a few atoms), and the conditions under which these tiny materials could self-assemble spontaneously into specific configurations. Understanding the self-assembly process is one of the most important objectives of nanotechnology, because this process could lead to materials with completely novel properties.

Perhaps the most familiar example of self-assembly is the formation of vesicles from phospholipids (lipids typically found in cellular plasma membrane) placed in water. The vesicles form under the action of divergent forces on the phospholipid's long, water-insoluble (hydrophobic) hydrocarbon tails, and its compact, water-soluble (hydrophilic) "headgroup." These divergent forces drive self-assembly. For example, the hydrophilic headgroups lie exposed upon the inner and outer surfaces of spontaneously formed spherical vesicles, while their hydrophobic tails lie sheltered in between the two hydrated surfaces.

We are now learning that factors far more subtle than the simple "hydrophobic" and "hydrophilic" forces can have profound consequences upon the morphology of a self-assembled material. For example, small, inflexible sections can be inserted in the phospholipid's otherwise flexible hydrocarbon tails by creating triple bonds in the tails. When this change to the tail's shape is made, then cooling the resulting spherical vesicles leads to the creation of rigid, hollow 30-by-0.5 micrometer (millionth of a meter) vesicles of an astonishing cylindrical symmetry (**Figure 1**).

Close examination of these cylinders, called tubules, reveals two unusual features: the tubules are coaxially nested sets of cylinders, also called lamellae, and the tubules possess a helical, hence chiral, substructure.


Authors (clockwise from left): Phillip Persichini, Christopher Lindemann, and Britt Thomas.

Tubules form through the helical winding of a uniform-width phopholipid bilayer ribbon, which creates a cylinder that serves as a "nucleus" for subsequent coaxial windings. Interestingly, the tubule's helical sense of handedness depends upon the chirality of the tubule-forming molecule: molecules of one chirality produce tubules possessing a right-handed helical structure, while molecules of the opposing chirality always produce left-handed helices.

To understand better tubule formation, we have made synthetic perturbations to tubule-forming molecules and characterized the new products. While it is clear that the tails' diynes are required for tubule formation, the remarkable helical handedness-molecular chirality correspondence suggests that by altering the molecule near its chiral center, significant changes to tubule morphology could result. Indeed, we have found that removing the phosphoryl oxygen that links the headgroup to the tails of the tubule-forming molecule called 1,2-bis(10,12-tricosadiynoyl)-sn-glycero-3-phosphocholine, we have doubled the tubule diameter (**Figure 2**). This change in tubule diameter is significant, since previous studies have succeeded in changing tubule length, but not its diameter.

Tubule gross morphology can be quickly measured by microscopy, but such measurements are both static and insensitive to the tubule's *interior* structure. The high intensity of the x-rays produced at beam line X10A of the NSLS has allowed us to perform kinetic small-angle x-ray scattering (SAXS) studies of the transition between spheres and tubules. These studies revealed that: (1) the transition is of the first order (spheres and tubules are in equilibrium during the transition) and is reversible; (2) tubular interlamellar spacing is tightly conserved when tubule diameter is doubled; and (3) the number of lamellae is *halved* when the tubule diameter doubles. These surprising results about tubule interior structure give important clues about intrinsic membrane curvatures and bending moduli, which are unobtainable by any other means.

Tubule dimensions and morphology suggest a host of potential technological applications, ranging from nanofabrication and purification to medical encapsulation. Our work is one step into realizing this technological potential, which will ultimately require optimizing the tubule's morphology for a given application, and understanding not only its "gross" structure but its interior structure as well.

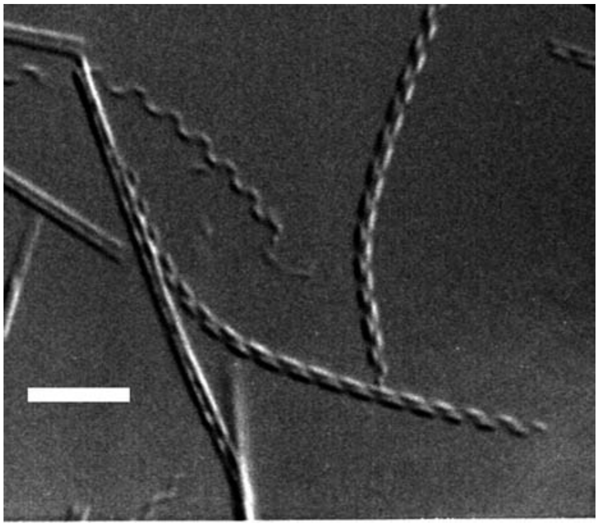

Figure 1. *Some phosphonate tubules observed with an optical microscope.*

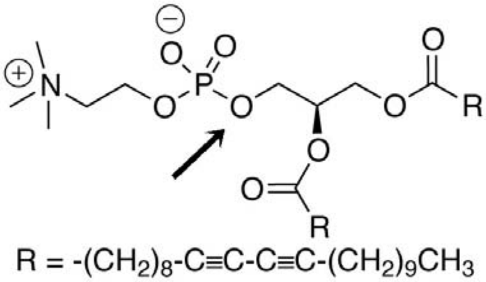

Figure 2. *The tubule-forming 1,2-bis(10,12-tricosadiynoyl)-sn-glycero-3-phosphocholine. The arrow indicates the oxygen atom removed to make the tubule derivative investigated in this study. The molecule's chiral center lies two atoms at the right of that of this oxygen.*

Formation of Self-Assembled Monolayers with Liquid Carbon Dioxide

K. Efimenko[1], B. Novick[1], R. Carbonell[1], J. Genzer[1], and J. DeSimone[2]

[1]North Carolina State University in Raleigh; [2]University of North Carolina in Chapel Hill and North Carolina State University in Raleigh

Self-assembled monolayers (SAMs) are surface-grown materials with a thickness of a few billionths of a meter, and structurally well-defined physico-chemical characteristics. Scientists at North Carolina State University in Raleigh, and the University of North Carolina in Chapel Hill have studied the formation and properties of SAMs prepared by depositing organosilane molecules from their vapor phase or a liquid phase in which they are dissolved in an organic solvent or liquid carbon dioxide, thus exploring the possibility of replacing environmentally harmful vapor and organic solvents by liquid carbon dioxide.

BEAMLINE
U7A

PUBLICATION
K. Efimenko, B. J. Novick, R. G. Carbonell, J. M. DeSimone and J. Genzer, "Formation of Self-assembled Monolayers of Semi-fluorinated and Hydrocarbon Chlorosilane Precursors on Silica Surfaces from Liquid Carbon dioxide", *Langmuir* **18**, 6170 (2002).

FUNDING
Kenan Center for the Utilization of Carbon Dioxide in Manufacturing, National Science Foundation, U.S. Department of Energy

FOR MORE INFORMATION
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Scientists can create thin films with sizes comparable to the dimension of a single molecule, which could be used in electronic components the size of a few nanometers, or billionths of a meter. Such nanometer-thick layers can be prepared by depositing self-assembled monolayers (SAMs) on a substrate.

Some of the most commonly used molecules forming SAMs are alkyls bearing either a mercapto [-SH] group or organosilanes that are terminated with a chlorosilane [-Si-Cl] or alkoxysilane [-Si-OR, R being either methyl or ethyl] moieties. Organosilanes are particularly useful for modifying the surfaces of silicon and other semiconductor surfaces that are covered with a nanometer-thick oxide layer, making them suitable for biosensors, the reduction of corrosion rates, pattern creation by lithographic processes, and friction reduction.

Current techniques involve either vapor- or liquid-phase deposition of organosilane-based SAMs on materials surfaces. The environmental impact of both vapor and liquid deposition processes and the rather slow deposition rates (hours to days) call for exploring alternative media from which SAMs could be deposited. Compressed carbon dioxide (CO_2), either in the liquid or supercritical state (temperature of 31 degrees Celsius and pressure of 72 times the atmospheric pressure), is a benign solvent that should be considered as a viable substitute for current SAM deposition media.

We studied the formation and properties of SAMs based on semifluorinated and hydrocarbon trichlorosilane precursors, $F(CF_2)_8(CH_2)_2SiCl_3$ (F8H2) and $H(CH_2)_{18}SiCl_3$ (H18), respectively. The organosilanes were deposited onto flat silica surfaces either from their vapor phase or a liquid phase in which they are dissolved in an organic solvent or liquid CO_2 (**Figure 1**). To get insight into the formation mechanisms of the F8H2- and H18-SAMs, we determined the thickness, wetting characteristics, and the average tilt angle of the molecules on the surfaces.

Jan Genzer

The SAM thickness was measured by using a technique called ellipsometry, which uses polarized light to probe the dielectric (electric polarization) properties of the sample (**Figure 2**, top). The wettability of the SAM-covered substrates was determined by depositing various liquids on the SAMs and by measuring the angle between the SAM surface and the tangent to the liquid drop, called the contact angle. The ellipsometry and wettability results revealed that the primary mechanism controlling the SAM formation was the adsorption of the molecules to the surface and not the diffusion of the molecules to the substrate. We have also established that the SAM formation kinetics from CO_2 is slower than that from the vapor solvent, but faster when using an organic medium, and the overall SAM formation was shown to be fastest when using CO_2 because of higher precursor concentrations.

We also studied the ordering kinetics of the F8H2 and H18 molecules in their corresponding SAMs by using near-edge x-ray absorption fine structure (NEXAFS) spectroscopy at the U7A beamline of the National Synchrotron Light Source. In NEXAFS spectroscopy, incoming x-rays are absorbed in the atoms of the material by exciting core electrons into unoccupied electronic states. In a given atom, an outer shell electron then occupies the core electronic state, and the energy released by this electron is transmitted to another electron that is ejected. By detecting these so-called Auger electrons, we can determine the molecular orientation and the binding of the SAMs to the substrate's surface.

The NEXAFS results (**Figure 2**, bottom) revealed that F8H2 molecules deposited from liquid CO_2 initially adsorbed without any molecular order. As more F8H2 molecules are accumulated on the silicon oxide substrate, they started to organize and orient. A complete monolayer order was achieved after about 30 minutes. We found that the order in the H18 SAMs increased with increasing time, and that a complete order was achieved faster than with F8H2 SAMs, after about five minutes of exposure to the H18 and liquid CO_2 solution.

We attribute the difference in the orientation kinetics to the different solubilities of F8H2 and H18 in liquid CO_2. While a higher solubility of the fluorinated part of the F8H2 molecule in liquid CO_2 slows down the complete monolayer organization, the high chemical incompatibility between the H18 molecules and liquid CO_2 leads to rapid reorientation of the H18 SAMs on the substrate. Also, possible intermolecular interactions between neighboring F8H2 chains may also be responsible for the slower organization kinetics in the F8H2 SAM.

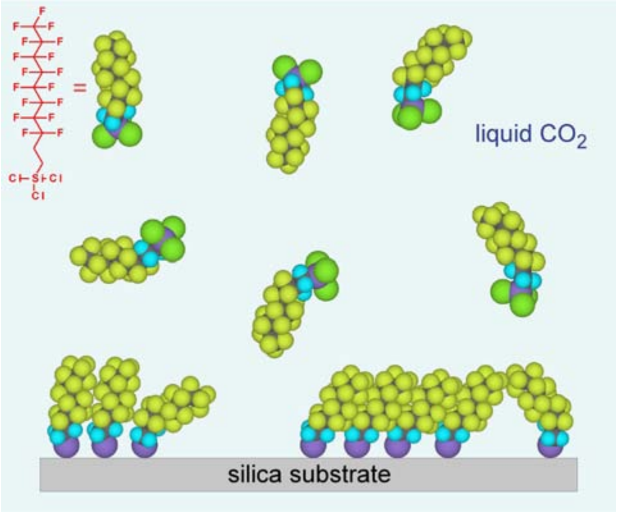


Figure 1. *Schematic representation of the way semifluorinated trichlorosilanes $(CF_2)_8(CH_2)_2SiCl_3$ (F8H2) diluted in liquid carbon dioxide (CO_2) self-assemble onto a flat silica substrate. The precursor molecules react with a trace amount of moisture, thus giving up three chlorine atoms (green spheres) that make hydrogen chloride (HCl) molecules, forming $(CF_2)_8(CH_2)_2Si(OH)_3$, which self-organize on the substrate and bind to the surface's hydroxyl groups via a condensation reaction.*

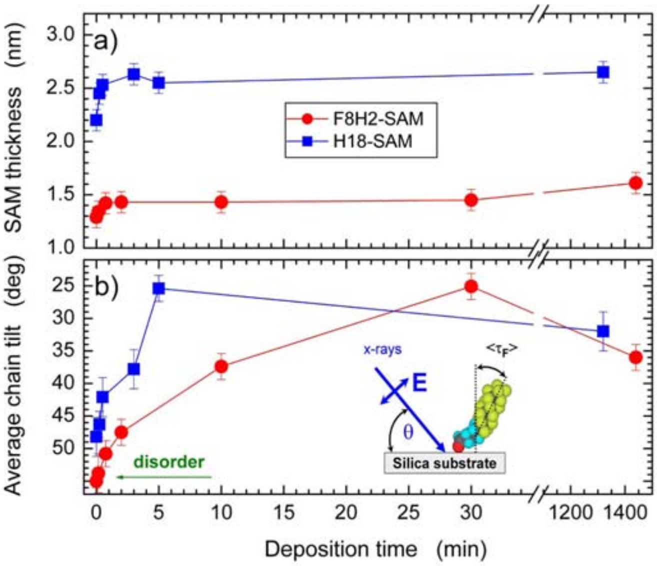


Figure 2. *Thickness (top) and average tilt angle (bottom) of the molecules in self-assembled monolayers of F8H2 (circles) and H18 (squares) molecules as a function of the deposition time from liquid carbon dioxide mixtures. The lines are meant to guide the eye. The inset shows schematically the geometry of the near-edge x-ray absorption fine structure (NEXAFS) experiment.*

Year In Review

Highlights From the March 2002 American Physical Society Meeting

March 18 - 22, 2002

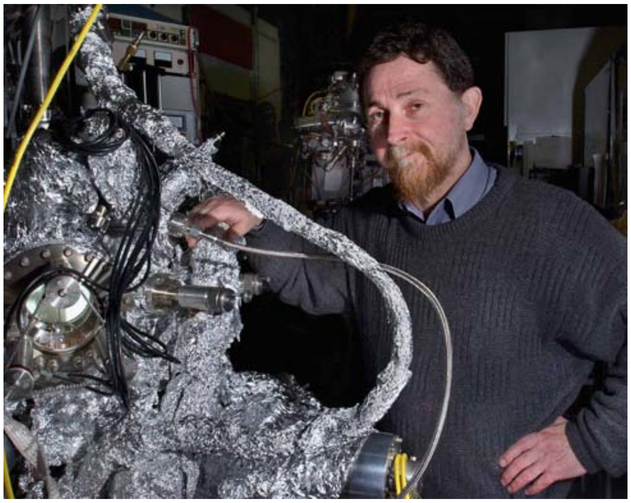
The stories below describe five of the more than 50 scientific presentations given by BNL scientists at the American Physical Society (APS) March meeting in Indianapolis, Indiana.

New Spin on High-Temperature Superconductors

Understanding what holds electron pairs together in high-temperature (high-T_c) superconductors is one of the biggest problems in condensed matter physics, says BNL physicist Peter Johnson, who is searching for the explanation.

Like traditional superconductors, high-T_c superconductors can carry electrical current with no resistance, or loss. But high-T_c superconductors operate at temperatures where liquid nitrogen, rather than expensive liquid helium, can do the cooling. This difference would decrease the cost of and increase potential applications for superconducting materials. But the first step is to understand the mechanism.

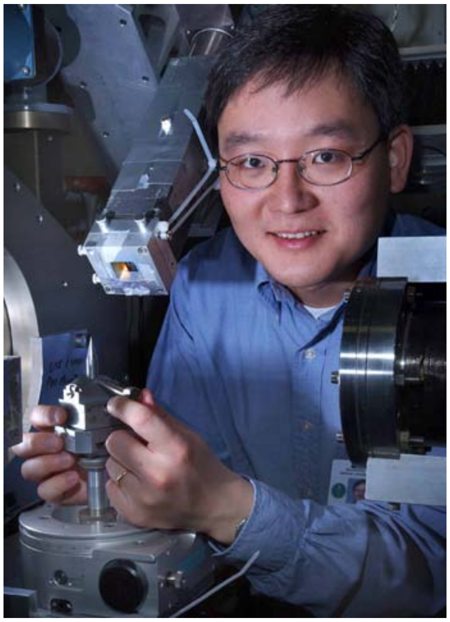
Johnson's National Synchrotron Light Source (NSLS) research indicates that electron "spin" plays an important role. Rather than exchanging vibrations with the crystal lattice (the mechanism for electron pairing in traditional superconductors), electron pairs in high-T_c materials interact by affecting the spin of atoms in the lattice. Understanding the role of spin has the potential to revolutionize our thinking about the transfer of electrical current, Johnson says.



Peter Johnson

Electron Excitations in High-Temperature Superconductors

BNL physicist Young-June Kim is studying the collective behavior of electrons in materials closely related to high-T_c superconductors. He uses resonant inelastic x-ray scattering (RIXS), a technique developed at the NSLS, to understand how electrons are moving around in the system. By comparing the energy of x-rays beamed into a sample and those coming out, this sensitive technique measures how much energy is transferred to the electrons in the material.



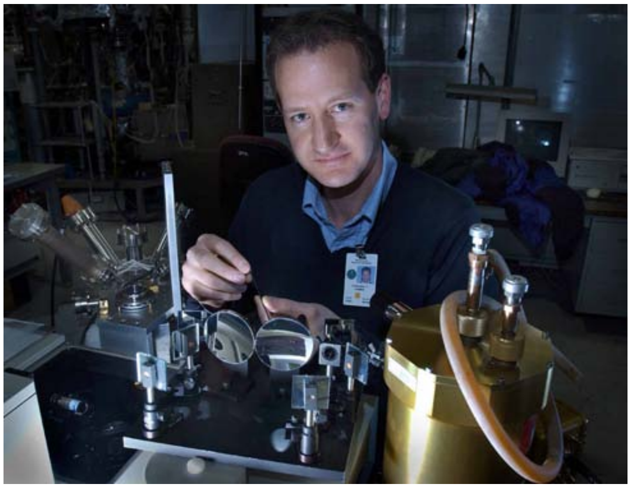
Young-June Kim

The absorbed energy can result in a variety of excitations, which can be distinguished by RIXS. Kim is using the technique to study lanthanium copper oxide, an insulating material, and looking at how the excitations change as the material is transformed to a high-T_c superconductor by gradually substituting strontium atoms for lanthanum atoms. With improved sensitivity, the technique may help reveal the mechanisms behind high-T_c superconductivity.

Material with Unusual Electrical Properties Holds Up to Scrutiny

An unusual material with an extremely high dielectric constant, which may have applications in high-performance capacitors and miniaturized electronics, first described last July by BNL physicist Christopher Homes has stood up to further scrutiny. The material's enormous dielectric constant - a property that determines it's ability to separate positive and negative electrical charges, or become polarized - is apparent even in thin films of the material, which are the form necessary for applications in microelectronics.

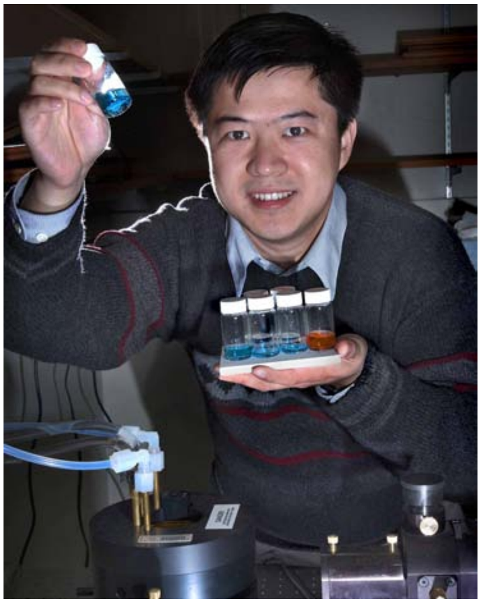
The scientists are now studying how the material changes as the chemical composition is altered. This will allow them to separate intrinsic from extrinsic effects and to zero in on the mechanism responsible for the large dielectric constant, Homes says.



Christopher Homes

Giant Nanomolecule Discovered by Accident

"Giant nanomolecules" may sound like an oxymoron, but these relatively large inorganic structures may provide big benefits for nanoscience, says BNL physicist Tianbo Liu. Measuring 5.1 billionths of a meter in diameter and covered with large pores, the spherical, cagelike molecules may be useful as "containers" for studying chemical reactions at the nanoscale. The molecules are themselves magnetically active, and can be used to create even stronger magnetic materials by loading other compounds inside. One possible application: contrast agents in magnetic resonance imaging (MRI).

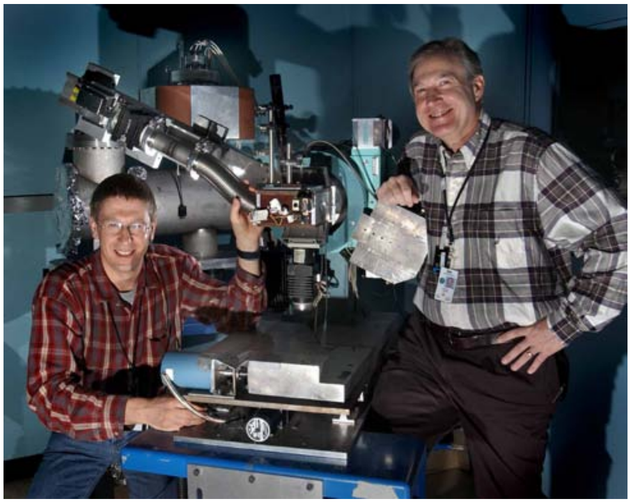


Tianbo Liu

Liu and his collaborators discovered the giant nanomolecules at the NSLS and using transmission electron microscopy. The particles' orderly arrangement tipped the scientists off that they were seeing something unique: nanoparticles with a uniform size. Ordinary nanoparticles vary in size. But these particles are molecules, with a definite molecular structure, so they are made the exact same way every time. Having one-size particles is an advantage when studying and finding applications for nanoscale properties, which are generally dependent on particle size.

Seeing Hidden Structure in Liquid Crystals

Liquid crystals, the materials used in laptop computer screen displays, optical networking devices, and other applications, are composed of rod-shaped mol-



Wolfgang Caliebe and Ron Pindak

ecules with the ability to change their orientation (the direction in which they "point") in response to an electric field. In many cases, these oriented molecules also form layers. The arrangements of these layers and orientations of molecules within them determine the materials' optical properties, says BNL physicist Ron Pindak.

Until now, some of the structural details of liquid crystals have been "hidden," because conventional x-ray scattering can detect the layers, but not the orientation of individual molecules within the layers. Moreover, often the orientation changes over very short distances, making it invisible to an optical microscope. But Pindak's "resonant polarized x-ray diffraction" experiments at the NSLS are starting to reveal these hidden structural details. Understanding these details will allow scientists to improve the design of liquid crystal materials, possibly leading to higher-definition video displays or faster optical conditioning devices. - Karen McNulty Walsh

[Editor's note: Reprinted with permission from the BNL Bulletin - March 22, 2002.]

Synchrotron Science Highlighted for Capitol Hill Policymakers

April 17 - 18, 2002

In an effort to increase the visibility of the NSLS and that of the three other DOE light sources, the chair and vice-chair of the NSLS users' committee, along with their counterparts from the other light sources, met with officials on Capitol Hill on April 17 and 18.

"The overall aim of these visits was to raise the awareness of the synchrotrons and to lobby for increased budgets for the Office of Science, and for the physical sciences overall," says Simon Bare, chair of the users' committee of Brookhaven's National Synchrotron Light Source (NSLS), organizer and lead spokesperson of the delegation.

On the first day of the visit, the delegation met with senior members of the Senate and House Energy & Water Appropriations Committees, the House Science Committee's staff, and key members of the Office of Management and Budget and the Office of Science and Technology Policy. Pat Fulton, Science Lobbyist for Stanford University and Jack Bagley, Battelle's Vice President for External Affairs, had set up all the necessary appointments for the six one-hour-long meetings.

On the second day, Bare and Leemor Joshua-Tor, vice-chair of NSLS users' committee, met with legislative assistants in the offices of Senators Charles Schumer (D-NY) and Hillary Rodham Clinton (D-NY), and in the offices of Congressmen Gary Ackerman (D-NY), Felix Grucci (R-NY), and Steve Israel (D-NY).

The delegation members, representing the synchrotrons located at Brookhaven, Argonne, and Lawrence Berkeley National Laboratories, as well as the Stanford Linear Accelerator Center, explained the various scientific contributions of these facilities in basic and applied science, the economy, health, and security. The light source representatives also highlighted the critical role played by DOE in funding scientific research at the synchrotron sources, and asked for an increase in the FY03 budget for synchrotron science.

"We tried to demonstrate as much as we could to Capitol Hill policymakers that the light sources provide tangible benefits to industry," Bare says, "We particularly stressed the fact that these facilities have led to many scientific breakthroughs during the past decades and are now helping scientists to pursue forefront research, particularly in the life sciences."

In general, Capitol Hill officials recognized the scientific importance of the light sources, Bare says, but they also “strongly encouraged us to communicate our message to a much larger audience, including other members of Congress and stakeholders beyond Capitol Hill.”

In an effort to increase the FY03 budget beyond the President’s request for DOE’s Office of Science, Representatives Biggert (R-IL) and Tauscher (D-CA), and Senators Bingaman (D-NM) and Warner (R-VA) are now circulating letters to provide a broad bipartisan support for science, and to encourage funding increases in the Office of Science. So far, at least a dozen Representatives and 35 Senators have signed these letters, Bare says.

“Our efforts to inform senior staff members about the importance of synchrotron facilities in many areas of science and technology are just bearing fruit,” Bare says. “We clearly need to increase our outreach efforts to broaden the policymakers’ commitments to synchrotron science in particular, and science in general.”

- Patrice Pages

NSLS's Podobedov Wins APS Award For Outstanding Thesis Research

April 21, 2002

Boris Podobedov of the National Synchrotron Light Source (NSLS) Department has won the American Physical Society's (APS) 2002 Award for Outstanding Doctoral Thesis Research in Beam Physics & Engineering.

Boris Podobedov

The annual award was established in 1990 by the APS Division of Physics and Beams, and it is currently supported by Brookhaven Science Associates, Southeastern Universities Research Association, and Universities Research Association.

"I am honored to receive this award, which is related to my studies at the Stanford Linear Accelerator Center," said Podobedov. "Since I came to BNL in 1999, I've been applying similar techniques to study beam dynamics at the NSLS rings."

The award, which consists of $2,500 and a certificate, was announced at an APS meeting in Albuquerque, New Mexico, on April 21.

Podobedov was cited "For an experimental study of the microwave instability in the Stanford Linear Collider (SLC) damping rings using a streak camera to correlate each event to the RF. The development of this sophisticated technique provides a powerful tool for the study of non-linear instabilities above threshold."

Most storage rings, whether used as light sources or high-energy physics accelerators such as SLC, require high beam current. When the current is increased, electrons or other types of stored subatomic particles reach a critical density and various kinds of instabilities tend to occur, which degrade beam quality.

For example, microwave instability affects internal bunch structure - the way in which particles are grouped together in a beam - which is often hard to measure. Using some novel diagnostics as well as an instrument called a streak camera, Podobedov measured the bunch shape evolution of unstable electron and positron beams at the SLC rings. In his award-winning thesis, Podobedov was able to show understanding of the detailed structure of the instability and to identify the mechanism that caused it.

At the NSLS, Podobedov has led a team of NSLS staff in designing and implementing the digital orbit-feedback system. This system stabilizes electron beam motion, which in turn results in more stable synchrotron light for experiments.

Installed in the vacuum ultraviolet (VUV) ring since August 2000, the system measures the displacement of electron orbit due to various factors such as floor vibrations or electrical noise, and then uses small magnets to correct for it. Currently, Podobedov is working on adapting this system for the NSLS x-ray ring. His other projects include work on possible upgrade options for the NSLS, and experimental studies of collective beam dynamics in the VUV ring.

Podobedov received his M.S. in physics from the Moscow Institute of Physics and Technology, Russia, in 1993, and his Ph.D. in applied physics from Stanford University in 2000. He joined BNL in October 1999 as an assistant scientist at the NSLS and was promoted to associate physicist in 2001.

-Diane Greenberg

[Editor’s note: Reprinted with permission from the BNL Bulletin - May 17, 2002.]

NSLS Annual RapiData Course in X-Ray Crystallography Again Successful

April 21 - 26, 2002

Brookhaven's offering of RapiData 2002, the fourth in an annual series of crash courses in x-ray crystallography, was once again a huge success. Forty-eight students from around the world participated from April 21 to 26.

More formally titled "Rapid Data Collection and Structure Solving at the NSLS: A Practical Course in Macromolecular X-Ray Diffraction Measurement," the program was developed by BNL's Biology and National Synchrotron Light Source (NSLS) departments to introduce students to the best people, newest equipment, and latest techniques in the field of macromolecular x-ray crystallography.

Workshop Participants

Half the students came to observe; the other half came with their own specimens with the goal of solving the structure of a particular enzyme. About four structures were solved during the course, and each of these is likely a publishable result.

Designed and run by Bob Sweet and Denise Kranz of BNL's Biology Department, the program is funded by a range of agencies. Key support comes from the National Institutes of Health National Center for Research Resources, and significant support from DOE's Office of Biological and Environmental Research.

The course starts with two days of lectures and tutorials taught by BNL scientists and scientists from industry, academia, and other national labs - a total of 38 instructors, tutors, or helpers, many of them volunteers.

These mentors then serve as hands-on scientific supervisors when the students move to the NSLS beam lines to begin collecting data on their crystal samples. There are also plenty of occasions for stimulating conversation over drinks and meals.

"Everyone, teachers and students alike, finds the experience absolutely riveting. The 60 hours of data collection are near chaos on the NSLS floor. But each of us ends up exhausted but happy, having learned a little from each of the others," Sweet said.

-Karen McNulty Walsh

[Editor's note: Reprinted with permission from the BNL Bulletin - May 17, 2002.]

Frontiers in Synchrotron Research on Soft Matter and Biomaterials Workshop

April 24 - 27, 2002

A workshop on "Frontiers in Synchrotron Research on Soft Matter and Biomaterials" was held from April 24-27 at the Mary Duke Biddle Estate in Tarrytown, NY. The workshop was co-organized by John Flanagan (BNL Biology), Ben Ocko (BNL Physics), and Ron Pindak (NSLS). Seventy-seven attendees from government, university, and industrial labs met at this magnificent site overlooking the Hudson River.

The workshop participants related exciting advances and identified open issues in a number of soft matter and biomaterials research areas including: phase transitions in confined geometries and at surfaces, directed self-assembly of soft matter systems into functional nano-structures, controlled inorganic growth in an organic matrix, new behavior in complexes such as those formed by lipids and proteins or liquid-crystals and polymers, and the dynamics of protein-folding.

The measurements described in these presentations were mainly carried out at the NSLS and SSRL synchrotrons. Measurements carried out at the APS and the ESRF synchrotrons were also presented. The latter measurements focused on the application of radiation from high brightness sources to study dynamics such as membrane fluctuations and polymer reptations.

In order for all the synchrotron facilities to more effectively impact these problems, the participants indicated that faster detectors with energy resolution, flexible geometry, and wide dynamic range were essential, that barriers needed to be reduced to improve the accessibility of synchrotrons to new users, that better on-site facilities were required for sample preparation, and that existing information on mitigating radiation damage needed to be assembled into a report.

Finally, a glimpse into potential new superconducting undulator insertion devices and energy-recovery schemes for high-brightness x-ray sources was provided. The interactions between the soft matter and biomaterials researchers were lively and productive in nature. A follow-up meeting to track progress and further encourage cooperative ventures is under consideration.

- Ronald Pindak

Workshop Participants

Take our Daughters to Work Day: Mysteries of Light and Extreme Cold Uncovered

April 25, 2002

On April 25, over 20 girls and their parents learned about some of the scientific programs at the National Synchrotron Light Source (NSLS), and even performed their own scientific experiments. The one-day visit was part of the national "Take our Daughters to Work Day."

At the NSLS, the girls learned that light can take many forms, from microwaves to x-rays, and have many applications in electronics, lasers, microscopes, and medicine.

NSLS scientist Lisa Miller offered a tour of the experimental floor to the girls, who discovered how synchrotron infrared light is used to study diseases like osteoporosis, osteoarthritis, and Alzheimer's disease. NSLS scientist Vivian Stojanoff also showed the girls how the study of protein crystals is used to develop new drugs. Later, the girls toured the Control Room, where Machine Operator Gary Weiner told them how synchrotron light is made.

The girls also performed their own scientific experiments. By suspending an inflated balloon in the cold vapor above liquid nitrogen, they discovered that the air inside of the balloon contracts, and then re-expands when warmed up. In another experiment, the girls found, much to their surprise, that they could

suspend soap bubbles in a gaseous carbon dioxide layer over a block of dry ice.

Perhaps one of the girls' most memorable experiments involved freezing natural and artificial flowers in liquid nitrogen. Both the girls and their parents learned that it is much more fun to freeze and crumble a living flower than to take it home as a souvenir.

-Patrice Pages

Successful Science, Exciting Plans, Record Attendance Characterize This Year's Annual NSLS Users' Meeting

May 20 - 22, 2002

The excellent scientific record of the past year, plans for upgrades of the NSLS, and projects for new light source facilities were all hot topics at this year's National Synchrotron Light Source (NSLS) Annual Users' Meeting, held at BNL on May 20 to 22.

Among the speakers at the 2002 Annual NSLS Users' Meeting were: (from left) Satoshi Ozaki, Richard Osgood, Steven Dierker, all of BNL;Patricia Dehmer, DOE; Leemor Joshua-Tor, Cold Spring Harbor Laboratory, NSLS Users' Executive Committee (UEC) Chair-Elect; Simon Bare, UOP-LLC, NSLS UEC Chair; and John Marburger, Presidential Science Adviser and Office of Science & Technology Policy Director.

The meeting, which drew a record attendance of 380 participants coming from all over the world, consisted of a one-day plenary session and eight one-day-long workshops on the latest results achieved at the NSLS in the physical, biomedical, environmental, and instrumentation sciences.

Speakers included: Steven Dierker, NSLS Chair; Richard Osgood, Associate Laboratory Director for Basic Energy Sciences; Patricia Dehmer, Associate Director of Science for Basic Energy Sciences (BES) at DOE; Judith Vaitukaitis, Director of the National Center for Research Resources, National Institutes of Health; and Satoshi Ozaki, Acting Deputy Laboratory Director for Science & Technology. John Marburger, Presidential Science Adviser and Director of the Office of Science & Technology Policy, was the meeting's keynote speaker.

In the plenary session, Ozaki opened the meeting by welcoming the participants and giving an overview of the major Laboratory programs. Osgood followed with an outline of the re-organization of the material science effort at BNL and the major initiatives within the BES directorate, in particular, a proposed BNL nanoscience center.

U.S. Representative Felix Grucci (R-New York, First District), who was scheduled to speak at the meeting, was detained in Washington. In a message read by meeting chair Leemor Joshua-Tor, Cold Spring Harbor Laboratory, Grucci praised the current and future scientific programs at the NSLS.

In his keynote address, Marburger said, "We are beginning the 21st century with a profound revolution in science based on capabilities in computing and instrumentation. These capabilities have achieved an importance as the foundations of contemporary science that earns them a top priority for support. The National Synchrotron Light Source is one of the key representatives of this new instrumentation."

Dierker highlighted the qualities that make the NSLS such a successful facility. "With more than 2,500 scientists from over 400 institutions per year coming from academic, industrial, and government institutions, the NSLS is a widely used facility," he said. "Not only do we have a large contingent of users, but also, last year, the users produced more than 800 publications based on research performed at the NSLS, 150 of which appeared in premiere science journals."

Lonny Berman, BNL, Leemor Joshua-Tor, Cold Spring Harbor Laboratory; Steven Dierker, BNL, Simon Bare, UOP-LLC.

Users' Executive Committee Chair Simon Bare (right), UOP, LLC, presents a photo of the NSLS to Office of Science & Technology Policy Director John Marburger.

In terms of budget, Dierker announced that DOE had recently added $600,000 to the NSLS budget for FY02. An additional $1.6 million is also contained in the President's FY03 budget request. "Altogether, we are looking at an increase of $2.2 million next year," Dierker said. "We are very grateful to DOE and Congress, and we are excited about the opportunities that this additional funding will allow."

Patricia Dehmer, Associate Director of Science, Office of Basic Energy Sciences, DOE.

The 2002 Annual NSLS Users' Meeting Organizing Committee includes:(from left) Lisa Miller, BNL; Mary Ann Corwin, BNL; Caroline Kisker, BNL; Nancye Wright, BNL; Dan Fischer, National Institute of Standards & Technology; Lydia Rogers, BNL; Leemor Joshua-Tor, Cold Spring Harbor Laboratory; Lonny Berman, BNL; Anatoly Frenkel, Yeshiva University.

Approximately 40 of the 55 posters at the NSLS Annual Users' Meeting poster session, organized by Lisa Miller (NSLS) were the work of students and postdocs.

Dierker also discussed some changes under way at the NSLS that will improve support for current and future users. One major change has been an administrative reorganization, to provide better coordination for project management and to place greater emphasis on support for user science. "We expect these changes to increase productivity and to enable our users to obtain better research results," he said.

In announcing longer-term plans, Dierker first hailed a recent achievement by one of the near-term future light sources, the deep ultraviolet free electron laser (DUV-FEL). Last February, the DUV-FEL generated light at 400 nanometers (billionths of a meter) by a process called self-amplified spontaneous emission process.

By fall, Dierker added, the DUV-FEL is expected to provide radiation at 88 nanometers, to be used in pioneering chemistry experiments. To advance beyond current NSLS performance by increasing beam brightness while reducing pulse length, Dierker announced plans for a new facility, which would benefit from two technological approaches.

One, based on an ultra-low emittance storage ring, would provide about a factor of 10,000 increase in brightness. The other, called Photoinjected Energy Recovery Linac (PERL), would produce high brightness and very short pulse lengths.

"The design would start with an ultra-low emittance storage ring and evolve toward a PERL approach as that technology develops," Dierker said. The new facility would be located adjacent to the x-ray ring.

During her presentation on the current programs managed by DOE-BES, Dehmer was enthusiastic about the current and future projects at the NSLS. "Steve Dierker has done a tremendous job over the

(From left:) Mark Chance, Albert Einstein College of Medicine; Judith Vaitukaitis, Director, National Center for Research Resources, National Institutes of Health; Patricia Dehmer, DOE; and Richard Osgood, BNL.

(From left:) Stefan Kycia, LNLS, Campinas, Brazil; Cam Hubbard, Oak Ridge National Laboratory; and Peter Stephens, Stony Brook University.

past year in rethinking the challenges that face the NSLS," she said.

Another new project discussed at the meeting was a center dedicated to the study of the infinitesimally small. Called the Center for Functional Nanomaterials, or Nanocenter for short, this new BNL facility has received a "very strong thumbs up" from initial reviews by DOE-BES, according to Ozaki.

The Nanocenter will be organized similarly to the NSLS, including laboratory clusters, user and visitor laboratories, and training and seminar facilities, and is expected to be built close to the NSLS and the Instrumentation Division.


Lunch under the "big top" next to Berkner Hall on Tuesday, May 21.

"The NSLS and the Nanocenter will be portals to each other," said Osgood, who masterminded the Nanocenter project. "We envision both of these facilities as helping each other maintain a strong and vigorous user base."

"Brookhaven's Nanocenter will be a bold departure from business as usual in the research communities of materials sciences and chemistry," Dehmer commented. "We are trying to change the face of small science by locating a lot of disciplines in one place, so that, if scientists want to pursue a research program that mixes chemistry, biology and materials science, then they can do it."

Marburger, in his keynote address, outlined some of the new changes facing science in the 21st century.

"Scientists are opening the doors to a new kind of science," Marburger said. "Never before had we been able to relate properties of large-scale matter and big things made of atoms to the detailed atomic structure. Now, we can, and the prospects are truly exhilarating.

"I look forward to seeing what comes out of this Laboratory, as well as others that I fully expect society to continue to support," he added.

During the workshops and part of the plenary session, scientists presented current research and future light source projects. They addressed topics as diverse as the use of synchrotrons in the environmental sciences, the study of ultra-fast processes with x-rays, nanoscale materials, membrane protein crystallography, catalysis, materials processing, synchrotron micro-spectroscopy and imaging, and the development of more advanced light source detectors.

— Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - June 14, 2002.]


Hiroshi Takahashi (left), BNL, and Rick Bradanick, VAT Inc.


(From left:) Janos Kirz, Stony Brook University; Thomas Vogt, BNL; Richard Osgood, BNL; and Peter Paul, BNL Interim Director.

UEC Community Service Award Presented to Gary Nintzel

May 21, 2002

Congratulations go out to Gary Nintzel, a member of the NSLS User Science Division Technical Specialists staff. Gary is the first recipient of the NSLS Users' Executive Committee (UEC) Community Service Award. This award is for service, innovation and dedication to users of the NSLS.

Gary Nintzel working on beamline U5UA.

Gary was nominated for this award by members of the NSLS user community. Many of the comments on the nomination form speak volumes for his contributions over the years:

- "Gary's support has made the difference between survival and defeat for several PRTs."

- "With his extensive knowledge of nearly all the beamlines on the VUV floor, he is able to diagnose problems very efficiently, minimizing lost beamtime."

- "In addition to his regular and untiring support for the growing number of beamlines under the control of the NSLS User Science Division, he has made time to provide significant support to nearly all the other beamlines on the VUV floor, plus X1 and X13."

Simon Bare, Chair of the NSLS UEC, presented the award to Gary at the NSLS Users' Meeting banquet on the evening of May 21. Gary received a $250 gift certificate and his name was engraved on a plaque that is displayed in the atrium of the NSLS.

- Simon Bare

Gary Nintzel (right), NSLS, is the first winner of the NSLS Users' Executive Committee (UEC) Community Service Award for service, innovation, and dedication to users of the NSLS. Nintzel was presented a with $250 gift certificate by Simon Bare, UEC Chair, at the Users' Meeting banquet on Tuesday, May 21, and his name was engraved on a plaque that is displayed in the NSLS atrium.

The Environmental Sciences: Synchrotrons Providing Powerful Tools for a New Science Workshop

May 20, 2002

A workshop on applications of synchrotron radiation in the environmental sciences has become a tradition at the NSLS Users' Meeting.

Two talks on soft X-ray applications were presented: Physicist Juergen Thieme, of the University of Georgia Augusta in Göttingen, Germany, outlined soft X-ray spectro-microscopy in studies of geochemical and environmental processes, and geoscientist Satish Myneni, of Princeton University, described the structure of water in controlling environmental processes. Two talks were given on powder diffraction. Jeffery Post, the Curator of the National Gem and Mineral Collection of the Smithsonian Institution in Washington, DC, demonstrated his joy in basic research by discussing real-time powder diffraction studies of the dehydration behaviors of clays. Geoscientist Peter Heaney, of Pennsylvania State University in University Park, showed how real time powder diffraction studies of hydrotalcites could elucidate the mechanism of rapid cation exchange in this class of clay minerals.

Chemist Hoi-Ying Holman, of Lawrence Berkeley National Laboratory, introduced the possibilities for synchrotron radiation FTIR spectro-microscopy to study microbial degradation of poly-aromatic hydrocarbon on mineral surfaces.

Environmental engineer Lisa Axe, of the New Jersey Institute of Technology in Newark, explained metal sorption on amorphous iron and manganese oxides by X-ray Absorption Spectroscopy.

Chemist Martine Duff, of the Savannah River Technology Center in South Carolina, discussed applications of hard X-ray spectroscopy to the field of high-level radioactive waste treatment and characterization.

BNL geochemist Mark Fuhrmann outlined the applications of synchrotron techniques to phytoremediation.

The workshop concluded with a roundtable discussion on environmental science at NSLS: Chi Chang Kao, Associate Chair of NSLS, outlined the NSLS perspective and support for environmental research; Geoscientist Rich Reeder, of the State University of New York in Stony Brook, and environmental scientist Paul Kalb, of BNL, outlined the EnviroSuite initiative; BNL environmental scientist Keith Jones discussed the laboratory for Synchrotron-Based Earth and Environmental Research currently located at X26A and X27A; Steve Sutton, director of Consortium for Advanced Radiation Sources (CARS) - which goal is to develop X-ray beamlines at the Advanced Photon Source at Argonne National Laboratory - outlined the concept for EnviroSync to define needs of environmental science to synchrotron users.

-Douglas Hunter

Workshop Participants

X-Ray Sources for Studies of Ultrafast Processes Workshop

May 20, 2002

Many regions of the electromagnetic spectrum have provided critical information on the structure and dynamics of matter, but x-rays have been especially useful because their wavelengths are similar to atomic dimensions. So, x-rays have proven indispensable in determining the structure of materials at the atomic level.

In addition to a material's structure, scientists can also look at the dynamics of atoms inside the material. Over the past several decades, physicists have learned how to produce short pulses of light, which have provided valuable insights into the motions of various forms of matter, including biological systems. This workshop focused on the use of "X-ray Sources for Studies of Ultrafast Processes."

Lasers have played a key role in producing short pulses of visible, ultraviolet and infrared radiation. These pulses can last between a few billionths of a second (nanoseconds) to a few billionths of a billionth of a second (attoseconds). Using various combinations of laser and accelerator technologies, scientists are now on the verge of conducting studies of ultrafast processes at the atomic level using pulsed x-rays.

The most direct means of generating ultrashort pulses of x-rays is to focus a laser with a corresponding temporal structure onto a target, hence generating a plasma that emits a flash of x-rays. Chemist Christoph Rose-Petruck, of Brown University in Providence, Rhode Island, described such a system developed in his laboratory for the study of chemical processes. This system is relatively inexpensive, costing only about half a million dollars and offers all of the advantages of having a dedicated system.

Two speakers at the workshop described the generation of ultrafast X-ray pulses by Thompson scattering of a laser pulse off relativistic electrons generated by a linear accelerator. Physicist Gwyn Williams, of the Thomas Jefferson National Accelerator Facility (TJNAF) in Newport News, Virginia, described experiments performed at TJNAF's Free Electron Laser. BNL physicist Igor Pogorelsky described the generation of short X-ray pulses at BNL's Accelerator Test Facility.

Beyond the available or nearly available approaches outlined above, three speakers described accelerator based X-ray sources in various stages of development that offer the prospects of far greater brightness. Physicist John Arthur, of Stanford Linear Accelerator Facility (SLAC), described plans for SLAC's Sub-Picosecond Pulsed Source (SPPS), which will use that portion of the SLAC linac not required in the B-factory experiment.

The planned successor to the SPPS at SLAC is the Linac Coherent Light Source (LCLS), which was described by physicist Stephen Milton, of the Advanced

Workshop Speakers (from left): Christoph Rose-Petruck, (Brown University in Providence, Rhode Island), John Arthur, of Stanford Linear Accelerator Facility (SLAC), Stephen Milton, (Advanced Photon Source (APS) at Argonne National Laboratory), John Sutherland (BNL, Biology), Joel Brock, (Cornell University in Ithaca, New York), and Gwyn Williams (Thomas Jefferson National Laboratory).

Photon Source (APS) at Argonne National Laboratory. Development of the LCLS is being undertaken by a collaboration of six research organizations including SLAC, Argonne National Laboratory (ANL), BNL, Los Alamos National Laboratory (LANL), Lawrence Livermore National Laboratory (LBNL), and the University of California at Los Angeles. It is designed to generate sub-picosecond pulsed X-rays using Self-Amplified Spontaneous Emission (SASE), hence eliminating the need for normal incidence mirrors to reflect the laser as used in conventional laser operating in the ultraviolet, visible and infrared regions of the spectrum or the use of seeded beams.

A parallel approach to generating ultrashort pulses of x-rays based on high energy pulsed electron beams generated in an Energy Recovering Linear (ERL) accelerator was described by physicist Joel Brock, of Cornell University in Ithaca, New York. Unlike the SPPS and LCLS, an ERL can function much in the fashion of existing synchrotron radiation sources supporting many experimental stations simultaneously, only some of which would be devoted to ultrafast timing.

The formal presentations were followed by a round table discussion on the present sources and future prospects for ultrafast X-rays. One point made by several of the discussants is that developments in accelerator physics have greatly outpaced development of detectors and beam transport optics. The scientists agreed that investment in needed in these areas to fully exploit current and future sources.

-John Sutherland

EXAFS Studies of Nanoscale and Nanostructured Materials Workshop

May 20, 2003

Motivated by the recent excitement of scientists in the field of nanoscience, which is the study of materials at a scale of a few billionths of a meter, physicist Vincent Harris of the Naval Research Laboratory (NRL) in Washington, DC, organized a workshop titled: "Extended X-ray Absorption Fine Structure (EXAFS) Studies of Nanoscale and Nanostructured Materials."

Although the workshop was tightly focused on the use of EXAFS to probe the nanostructure of materials, international speakers also covered magnetism, biology, geochemistry, catalysis, and electronics.

Physicist Scott Calvin, of NRL, chemist Josef Hormes, the director of the Center for Advanced Microstructures and Devices (CAMD), a synchrotron radiation research center at Louisiana State University in Baton Rouge, and chemist Robert Schloegl, of Fritz-Haber-Institute in Berlin, Germany, addressed the difficulties of quantitatively describing freestanding nanoparticles.

Physicist Anatoly Frenkel, of Yeshiva University in New York, made a presentation on nanoparticles embedded within a common amorphous host. Physicist Maria Grazia Proietti, of the University of Zaragoza in Spain, and environmental scientist Ken Kemner, of Argonne National Laboratory, presented their progress in the study of semiconductor quantum wires and nanoparticles formed via biogeochemical interactions, respectively.

Calvin and Hormes both presented talks focused on the use of EXAFS, specifically the use of FEFF-generated theoretical data together with FEFFIT fitting codes, to describe the local element-specific structure of nanoparticles and core shell nanoparticles. FEFF is a computer program created at the University of Washington that generates x-ray absorption fine structure spectra for clusters of atoms. This data is used by another computer program called FEFFIT to allow rapid fitting of unknown atomic structures.

Calvin's work includes the multi-edge fitting of mixed spinel ferrites and the passivation of metallic iron in multi-shelled nanoparticles. Calvin is particularly interested in the stabilization of high magnetic moment particles for applications ranging from high frequency electronics to magnetic targeted drug delivery. He described his nanoparticles as having multiple layers of amorphous metal and oxide surrounding a close packed Fe core.

In contrast, Hormes relied heavily upon XANES spectra to establish that the local structure of nanoparticles are indeed quite different from their bulk counterparts and that XANES spectra can be used to track the evolution of valency as the particle

Workshop Participants

size is increased during processing.

Schloegl presented a review of his work in the use of *in-situ* time resolved EXAFS to understand the local structure fingerprinting of the phase transformations of molybdates; a class of materials that hold potential for catalytic applications.

Proietti described the use of Grazing Incidence Diffraction Anomalous Fine Structure (GI-DAFS) to reveal the role of phosphorous in InAs/InP self assembled quantum wires. This talk was a balance of high quality semiconductor materials processing and extremely difficult techniques that left the audience thoroughly impressed.

Finally, Kemner showed the value of the APS as a state-of-the-technology synchrotron capable of probing the dynamics of ground water contamination in the burgeoning field of biogeochemistry.

Each speaker generated excitement and active question-and-answer periods that often spilled out into the corridors. Materials scientist Joseph Woicik, of the National Institute of Standards and Technology (NIST) reminded the attendees of the value and importance of presenting raw data for inspection by the audience.

In summary, the key findings of the workshop were that EXAFS is a near-ideal tool for the characterization of nanoscale systems, but that it is best used with other techniques to provide scientists with a clearer picture of the material under study.

-Vincent Harris

Practical Aspects of Membrane Protein Crystallography: From Overexpression to Crystallization Workshop

May 20, 2002

Integral membrane proteins perform some of the most important functions of living cells, yet understanding their molecular mechanisms through structural studies presents unique challenges. The aim of the workshop entitled "Practical Aspects of Membrane Protein Crystallography: From Overexpression to Crystallization" was to discuss the nuts-and-bolts of over-expressing, purifying, and crystallizing membrane proteins for structural studies by x-ray crystallography.

Biologist Susan Buchanan, of the National Institute of Diabetes and Digestive and Kidney Diseases (NIDDK) at the National Institute of Health (NIH), made a presentation on the production, purification, and characterization of bacterial outer membrane proteins both refolded from inclusion bodies and also as membrane-inserted functional proteins.

Cell biologist Da Neng Wang, of New York University, described the production of bacterial inner membrane transporters as functional, affinity-tagged proteins, and methods for optimization of each step in their production and purification.

Biologist Martine Cadene, of Rockefeller University in New York, explained the use of the Matrix Assisted Laser Desorption Ionization (MALDI) mass spectrometry in defining the covalent state of purified membrane proteins, and using this information as an aid in designing mutant constructs specifically amenable to crystallographic analysis.

Biologist Miro Venturi, of NIH, described methods for the production and screening of monoclonal antibodies specifically reactive against the folded conformations of integral membrane proteins. He described recombinant methods for production and purification of Fab and Fv fragments of these antibodies, and their use as crystallization reagents, giving work with the transporter protein NhaA as a specific example.

Structural biologist Reinhard Grisshammer, of NIH, "Expression and Purification of G-Protein-Coupled Receptors (GPCRs) for Structure Determination", gave a detailed presentation on the expression and purification of functional G-protein coupled receptors as fusion proteins in E. coli. He gave examples of success with this approach for several different GPCRs.

Barry Springer, director of Molecular Biology and Protein Biochemistry and project team leader at 3-Dimensional Pharmaceuticals Inc. (3DP), presented the expression of functional GPCRs in mammalian (HEK-293) cells, and biophysical and fluorimetric high-throughput assays to assess their functionality and stability in detergent-containing solutions.

Biophysicist Ehud Landau, University of Texas Medical Branch in Galveston, described the use of cubic lipidic matrices as a medium for the stable incorporation of integral membrane proteins, and their crystallization in this medium. He presented many examples of highly diffracting crystals obtained using this methodology.

BNL biologist Dax Fu described the stabilization of solubilized bacterial transporters by inclusion of their cognate binding ligands, a necessary precursor to their successful crystallization.

Biochemist Patrick Loll, of Drexel University in Philadelphia, presented a rational approach to the design of crystallization screens for membrane proteins, based on the "cloud points" of detergents in different protein-precipitating conditions. Based on this approach, he has developed screens containing over 600 potentially useful conditions, near detergent phase transitions, and demonstrated a high rate of success with this screen.

-Larry Shapiro and Filippo Mancia

Workshop Participants

Recent Advances in the Application of Synchrotron Radiation to Catalysis Workshop

May 22, 2002

In a successful and informative workshop titled "Recent Advances in the Application of Synchrotron Radiation to Catalysis," topics ranged from new developments of using soft x-rays at high pressures to in situ X-Ray Diffraction (XRD) studies.

Chemist Gary Haller, of Yale University, began the workshop with a talk on a detailed characterization of platinum and tin in PtSn-MCM41 catalysts.

Workshop Participants

Simon Bare, from UOP LLC - a leading company in developing and commercializing technology for license to the oil refining, petrochemical and gas processing industries, headquartered in Des Plaines, Illinois - showed how theoretical calculations combined with in situ X-ray Absorption Near-Edge Structure (XANES) is a powerful combination that provided detailed insight into the chemistry of small platinum clusters.

Chemical engineer Enrique Iglesia, from the University of California, Berkeley, showed several examples highlighting both the power and also the drawbacks of X-ray Absorption Fine Structure (XAFS) in catalyst characterization, particularly in supported metal oxide catalysts. He emphasized the need that the characterization must be combined with catalyst activity in order to ensure that meaningful structure-activity relationships are developed.

BNL chemist Jon Hanson highlighted the applicability of in situ X-Ray Diffraction (XRD) for following structural phase transformation in bulk catalysts.

The applicability of Near-Edge X-ray Absorption Fine Structure (NEXAFS) to characterize the electronic structure of both model and real catalysts was demonstrated by Jingguang Chen, of the University of Delaware in Newark.

Chemist Robert Schloegl, of Fritz-Haber-Institute in Berlin, Germany, updated the workshop on recent developments in using soft x-rays at more realistic reaction conditions to probe the electronic structure and chemical form of the surfaces of working catalysts. He showed two examples where the activity of the catalyst was linked to a particular species that was only present under reaction conditions, this highlighting the need for in situ surface sensitive spectroscopy.

Chemical engineer and materials scientist Daniel Resasco, of the University of Oklahoma in Tulsa, talked about a novel catalyst used for producing single-walled carbon nanotubes, and how Extended X-ray Absorption Fine Structure (EXAFS) was critical in developing an understanding the structure of the active form of the catalyst.

John Gland, of the University of Michigan in Ann Arbor, used in situ Fluorescence Yield Near Edge Spectroscopy (FYNES) to not only characterize the intermediates present during the total oxidation of hydrocarbons on single crystals of platinum but also to follow the kinetics of the oxidation.

BNL chemist Jan Hrbek closed out the workshop with a talk on the use of high resolution X-ray Photoelectron Spectroscopy (XPS), combined with Density Functional Theory (DFT) calculations, to follow the sulfidation of a titanium dioxide single crystal surface.

-Simon Bare

Combining Synchrotron Infrared and X-Ray Micro-Spectroscopy and Imaging Workshop

May 22, 2002

One of the most well attended workshops at the meeting was entitled "Combining Synchrotron X-Ray and Infrared Imaging and Microspectroscopy."

The goal of the workshop was to introduce and describe the many micro-spectroscopy and imaging techniques available at the NSLS, including infrared (IR) micro-spectroscopy, hard x-ray microprobe, Scanning Transmission X-ray Microscopy (STXM), x-ray micro-diffraction, diffraction-enhanced imaging (DEI), and x-ray micro-tomography. The workshop provided unique insight into why the NSLS is so well suited for these techniques.

The synchrotron light produced at the NSLS is 1,000 to 10,000 times more intense than that produced at conventional laboratory sources. The light is also very highly collimated and covers a broad spectra range, from x-ray wavelengths as small as 0.1 angstrom to the far-infrared, with wavelengths longer than 10^7 angstrom.

These characteristics mean that smaller, more intense beams can be produced, giving scientists the capability to produce images of extremely small or dilute samples, determine elemental or molecular concentration gradients, and probe the chemical states of elements and compounds in a variety of materials with very high spatial resolution.

The workshop gave the attending NSLS users a practical sense of the individual techniques available to them and specific examples of how such techniques are being used and combined by users to solve complex scientific problems in a variety of fields.

During the morning session, six speakers described the ins and outs of each technique.

Geochemist Antonio Lanzirotti, from the University of Chicago, presented a discussion of the X26A beamline hard x-ray microprobe. With an emphasis on Earth and Environmental Sciences, X26A allows users to quantify elemental abundances of materials in situ with detection limits often better than one part per million (ppm) and at a spatial resolution of 10 micrometers or better.

Physical chemist Jean Jordan-Sweet, from the IBM Research Division's Thomas J. Watson Research Center in Yorktown Heights in New York, introduced the attendees to the uses of micro-diffraction imaging at the X20 beamlines, showing how these instruments can be used to study interfacial strain caused by residual stresses in materials and for real-time characterization of electro-migration effects with a spatial resolution as small as 2 μm.

Physicist Chris Jacobsen, from the State University

Workshop Participants

of New York in Stony Brook, gave a broad and thorough overview of soft x-ray microscopy studies at the X1A beamlines. With an energy range of 100-1000 eV, these beamlines are optimized for imaging and spectroscopic analysis of low-Z elements such as carbon and oxygen with a spatial resolution as small as 30 nm. They are thus very well suited to the study of single biological cells.

John Dunsmuir, from ExxonMobil Research & Engineering Company (EMRE) in Fairfax, Virginia, discussed the uses of x-ray absorption micro-tomography (XMT) and how such studies are conducted at beamline X2B. With image resolutions approaching 1 µm, XMT analysis of medical, industrial, and earth materials gives users the ability to image the internal structure of macroscopic objects without having to physically dissect the object.

NSLS physicist Zhong Zhong highlighted the recent and dramatic advances in Diffraction-Enhanced Imaging (DEI) at the X15A beamline. DEI dramatically enhances the ability to image and contrast soft tissue in ways not currently possible using techniques such as Magnetic Resonance Imaging (MRI) and Computed Tomography (CT) imaging. In the field of clinical imaging, particularly with respect to mammography, it's clear that DEI can provide remarkable sensitivity compared to standard radiographic techniques.

NSLS chemist Lisa Miller gave an informative presentation illustrating most effectively how the 500-1000 times greater brightness of the infrared (IR) source on the VUV-IR ring in comparison to standard laboratory sources allows IR studies, often using commercial infrared microscopes, to be done with smaller spot sizes and higher throughput. Miller showed that these improvements allow for better resolution of chemical components in a wide range of materials, including plant and animal tissue, polymers and laminates, and geological samples.

The six speakers of the afternoon session focused on various applications, emphasizing the complementary information that can be obtained by using multiple techniques.

BNL environmental scientist Keith Jones presented a study of the properties of sediments in the New York/New Jersey harbor using XMT, hard and soft x-ray micro-spectroscopy, and Fourier-Transform Infrared (FTIR) techniques.

Biologist Lila Graham, from the Children's Hospital in Boston, presented the results of a study of the nature of bone mineralization using FTIR, hard x-ray micro-spectroscopy and micro-diffraction techniques.

Physicist George Flynn, of the State University of New York in Plattsburgh, provided insight into the elemental mapping of interplanetary dust particles that he obtained by using hard and soft x-ray micro-spectroscopy, FTIR, and XMT techniques.

Geophysicist Russell Hemley, of the Carnegie Institute in Washington, DC, presented studies of high-pressure materials and compounds using FTIR and x-ray micro-diffraction techniques.

Physicist Marco Di Michiel, of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France, presented results of a study of fast high-energy x-ray tomography and site preferences for metal uptake in minerals, using hard x-ray micro-spectroscopy.

Geologist Richard Reeder, of the State University of New York at Stony Brook, presented his latest results on site preferences for metal uptake in minerals, obtained using hard x-ray micro-spectroscopy.

A "Guidebook of Micro-spectroscopy and Imaging Techniques at the NSLS" was distributed to the attendees at the end of the workshop and can be downloaded online at:
http://nslsweb.nsls.bnl.gov/nsls/pubs/nslspubs/imaging0502/imaging0502.htm

-Lisa Miller and Antonio Lanzirotti

In situ Studies of Materials Processing Workshop

May 22, 2002

A workshop titled “In situ studies of materials processing” included speakers using synchrotron-based x-ray techniques to study materials processes and speakers outside the synchrotron community.

Physicist Randy Headrick, of the University of Vermont in Burlington, kicked off the workshop with an overview of in situ studies of materials processing. He outlined the information that can be obtained with

Workshop Participants

different synchrotron-based surface x-ray techniques and assessed the potential of X21 for such studies in the future.

Physicist Christian Lavoie, from the IBM Research Division's Thomas J. Watson Research Center in Yorktown Heights in New York, showed how the use of real-time synchrotron light can dramatically reduce the time necessary to investigate the physical properties and transformations in a wide range of cobalt alloy silicides.

Materials scientist Kit Umbach, of Cornell University in Ithaca, New York, showed how Grazing-Incidence Small-Angle X-ray Scattering (GISAXS) had enabled him to follow carefully the formation of ripple patterns on silicon dioxide during sputter erosion with argon ion beams. His results suggest that the finite viscosity of the surface plays an important role in determining the surface morphology.

This work contrasted with that of materials scientist George Malliaras, of Cornell University, who is developing pentacene-based optoelectronic materials. His initial Atomic Force Microscopy (AFM) and x-ray scattering results with Headrick suggest that there is an unusual structure in the first layer of pentacene deposited onto silicon, and that 3-d crystallites nucleate on top of that layer. One of the most powerful surface x-ray techniques is surface crystallography, but its effective utilization requires solving the well-known phase problem.

Physicist Paul Lyman, of the University of Wisconsin in Milwaukee, showed that, to some extent, this problem could be overcome by using maximum entropy methods.

The last three talks dealt with very important materials processes for which real-time x-ray scattering may be able to provide vital information.

Physicist R.D. Vispute, of the University of Maryland in College Park, discussed the extensive Pulsed-Laser Deposition (PLD) growth efforts at the University of Maryland, focusing particularly on efforts to develop aluminum nitride dielectric layers for use on silicon carbide-based devices and to develop wide-bandgap semiconducting zinc-oxide/manganese oxide alloys for UV detectors.

Physicist Eleftherios Iliopoulos, of Boston University, discussed Molecular Beam Epitaxy (MBE) growth of III-V nitride films, particularly alloys of gallium-nitride with aluminum-nitride and indium-nitride.

Finally, materials scientist Karl Ludwig, of Boston University, made a brief presentation on behalf of materials scientist Charles Eddy, from the Naval Research Laboratory (NRL) in Washington, DC, who was unable to attend the workshop. Eddy reviewed plasma processing and showed that, while much has been learned about the chemistry of the plasma and the gas above the processed surface, relatively little is known about the actual structure of the surface itself. In this case, as in many examples presented throughout the workshop, the new real-time facilities being planned at the NSLS would provide many opportunities for enhancing our understanding of fundamental materials processes.

-Karl Ludwig, Randy Headrick, and Chi-Chang Kao

Advanced Detectors Development Workshop

May 22, 2002

The workshop on advanced detector development was well attended, with 45 people present. Eight speakers made presentations on pixellated detectors, spectroscopy detectors and other advanced detector developments.

BNL physicist Pavel Rehak presented his ideas and initial results from a 2-D detector built using Complementary Metal-Oxide Semiconductor (CMOS) readout circuits formed directly on a detector-grade silicon. Rehak's ideas could be used to construct efficient direct-detection devices with rapid (about one millisecond) readout.

Physicist Paul Seller, of Rutherford Appleton Laboratory, in Chilton, U.K., gave a summary of European efforts in the design and construction of pixellated detectors. He described several projects in various stages of completeness, and left a definite impression that the European synchrotron community may have been quicker to realize the need for advanced detectors than their American colleagues.

Biophysicist Mark Tate, of Cornell University in Ithaca, New York, described the construction and applications of a high-speed (microsecond) pixellated-integrating detector for real-time imaging, and showed some fascinating data from radiography of vehicle fuel injectors in action.

BNL's Instrumentation Division scientist Gianluigi De Geronimo described the design and initial tests of a 384-element spectroscopy detector for use in Extended X-ray Absorption Fine Structure (EXAFS) experiments of dilute systems.

This theme was continued by engineer Chris Cox, from Princeton Gamma-Tech - a leading supplier of microanalysis systems for x-ray and gamma ray spectroscopy - of experiments to study incomplete charge collection effects in lithium-silicon detectors when used in the soft x-ray range.

Physicist Stephan Friedrich, of the University of California, Davis, described a new class of detectors based on cryogenics. He showed data from experiments using superconducting tunneling junction detectors in the soft x-ray region, demonstrating extraordinarily good energy resolution, in the range 5-10 eV at a few hundred eV.

Physicist Dan Fischer, from the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland, described his application of efficient multilayer-based dispersive optics to the problem of extracting a weak fluorescent signal from a large background of other radiation, without requiring an ultra-high resolution detector.

The final talk of the workshop was given by BNL physicist Graham Smith, who described a range of new ways to perform electron amplification using micropatterned electrodes. This new technology should allow optimizing new devices for synchrotron facilities.

-Peter Siddons

Workshop Participants

BNL Hosts VIPs at RHIC, ITD, PET, NSLS

May 31, 2002

On May 31, Undersecretary of Energy Robert Card and Director of DOE's Office of Science Raymond Orbach, with DOE's Craig Reed and Jeffrey Salmon, visited BNL. After a tour of the STAR detector, the visitors saw BNL's supercomputing facilities in the Information Technology Division, learned about some environmental programs, then visited the Positron Emission Tomography Facility before stopping at the National Synchrotron Light Source. Here, U.S. Representative Felix Grucci also joined in welcoming the group and later hearing a presentation on current and future research.

[Editor's note: Reprinted with permission from the BNL Bulletin - June 14, 2002.]

U.S. Representative Felix Grucci (left) is welcomed by Richard Osgood, Associate Laboratory Director for Basic Energy Sciences, and Marge Lynch, Assistant Laboratory Director for Community, Education, Government & Public Affairs.

Secretary Abraham Gives Go-Ahead for BNL Nanoscience Center

June 14, 2001

"On behalf of the Department of Energy, I am pleased today to officially announce our approval to begin the conceptual design of the $85 million Center for Functional Nanomaterials here at Brookhaven." With this statement, U.S. Secretary of Energy Spencer Abraham declared last Friday, June 14, that DOE will establish a Northeast center for nanoscience research at BNL, as one of five such facilities to be built around the country under the National Nanotechnology Initiative.

The study of materials the scale of billionths of a meter (or 1,000 times smaller than a human hair), nanoscience is expected to allow researchers to design materials tailored to specific needs, such as strong, lightweight materials, new lubricants, and more efficient solar energy cells.

Abraham announced this good news — BNL's first scientific facility construction project since the Relativistic Heavy Ion Collider (RHIC) — at a Lab-wide meeting in Berkner Hall, during the Energy Secretary's first visit to BNL. Abraham delivered his remarks after touring the Lab with BNL Interim Director Peter Paul, U.S. Representative Felix Grucci (R-NY, First District), and DOE Office of Science Director Raymond Orbach.

The design and construction activities of the nanoscience center at BNL are expected to start in fiscal year (FY) 2004, with commissioning planned for FY2007. The project's preliminary cost estimate is $70-85 million. Under DOE's Office of Science, the conceptual design report will be prepared immediately so that construction funding can be requested from Congress.

A standing ovation followed Abraham's announce-

At a press conference held after Secretary of Energy Spencer Abraham announced DOE's support for the nanocenter at BNL: (from left) Director of DOE's Office of Science Raymond Orbach, U.S. Representative Felix Grucci, Secretary Abraham, and BNL Interim Director Peter Paul.

ment as the audience of BNL employees, facility users, and other guests showed its enthusiasm for this new scientific facility, which will allow BNL to become a major player in the new field of nanoscience. "Nanoscience offers the potential for a second industrial revolution," Abraham said. "The implications are enormous. That is why we are so serious about America's leading the way in nanoscience." Grucci, a member of the House Committee on Science and a leader on science issues, introduced Abraham, citing the Energy Secretary's strong commitment to science policy from the time when he was U.S. Senator from Michigan, 1995-2001.

"Abraham was widely viewed by his colleagues and most observers as the senator with the most and the best understanding of high-tech policies and issues," Grucci said. As Abraham elaborated, the new center at BNL will be a user facility open to researchers from across the country and around the world that will gain access to its state-of-the-art instruments and equipment through a peer-review process. "When this center is complete, physicists, chemists, material scientists, and biologists will work with computer scientists and engineers exploring the world atom by atom," Abraham said. "You need major facilities for this kind of work, major facilities with the best new

Spencer Abraham (below, left), Peter Paul, and BNL Associate Director for Basic Energy Sciences Richard Osgood.

Secretary Abraham (left) learns about the U7A beam at the National Synchrotron Light Source (NSLS) from researcher Daniel Fischer, NIST; and NSLS Chair, Steven Dierker (right).

technology and the best minds America has to offer," he continued. "We have all of this here at Brookhaven."

In his welcoming remarks, Paul had emphasized the Lab's role in pursuing forefront scientific research, both fundamental and applied, and reinforcing collaborative efforts with other scientists, from both research institutions and industry. "Our motto is 'a passion for discovery,' " Paul told the audience. "We work with a passion for the science and the science-based applications that derive from it for the good of the nation and industry." Abraham acknowledged that nanoscience is still a basic science with no clear applications yet.

But he expressed his commitment to supporting basic science because of its unknown ultimate applications. "What is so exciting about the work we do [at DOE] is that we produce benefits to America and the world that go well beyond the original scope of our mission," he said. "No one really knows where nanoscience will ultimately lead us, but we do know that we are at the beginning of a science initiative that may change the way we look at, and can use, the world around us."

Before the all-hands meeting, Abraham, Grucci, and Orbach, with the Secretary's wife, Jane Abraham, Frank Crescenzo, Acting Manager of DOE's Brookhaven Area Office, and others had toured some of the Lab's main facilities. After receiving an overview of the Lab from Paul, they listened to a lunchtime presentation by Nora Volkow, Associate Laboratory Director for Life Sciences, on the Lab's positron emission tomography facility and the proposed cyclotron isotope research center. Following lunch, the group toured the RHIC tunnel and STAR detector, accompanied by Thomas Kirk, Associate Laboratory Director for High Energy & Nuclear Physics, and others, and learned about BNL's national security and energy research from Ralph James, Associate Laboratory Director of Energy, Environment & National Security. The visitors were then shown the NSLS by Richard Osgood, Associate Laboratory Director for Basic Energy Sciences, and Steven Dierker, NSLS Chair, including an overview from Osgood of the then-proposed nanoscience center. "With RHIC and with the nanoscience center, which we launch today, Brookhaven is on track to remain one of America's — and the world's — most valuable centers for scientific research," Abraham concluded.

-Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - June 21, 2002.]

Yongjae Lee Wins Award for High-Pressure Research

June 26, 2002

Yongjae Lee, a postdoctoral fellow in the Physics Department, has won the 2002 Alvin Van Valkenburg Award for his work on a newly discovered class of materials that expand under pressure.

This award is given every second year in the name of physicist Alvin Van Valkenburg, co-inventor of the diamond anvil cell, to honor a young scientist who uses this device in his or her scientific research.

Lee was presented with a prize from the Alvin Van Valkenburg Memorial Fund and gave a short talk on his work at a June 26 awards ceremony hosted by William Bassett, Cornell University, during the biannual Gordon Conference on "Research at High Pressure" in Meriden, New Hampshire, June 23-28. The award news will also be featured in the upcoming issue of Physics Today.

"I am very honored to win this international young scientist award in high-pressure sciences," said Lee. "Most of the discoveries and results would not have been possible without support and creative input from my many mentors and collaborators and the optimal instrumentation available at Brookhaven Lab."

Funded by DOE's Division of Materials Sciences and Division of Chemical Sciences, as well as by BNL's Laboratory Directed Research & Development, Lee's research is on the pressure-induced swelling of zeolites. The phenomenon was discovered by an international collaboration between BNL and the School of Chemical Sciences at England's University of Birmingham.

When subjected to great pressures in a diamond anvil cell, these zeolites expand as fluid from the surrounding medium is squeezed into their tiny pores.

This unusual result may lead to applications for these materials as "molecular sponges" for soaking up chemical pollutants or even radioactive waste because, when the pressure is released and the material contracts, the fluid and possibly larger molecules or atoms could be trapped inside.

Using a technique called powder diffraction at the NSLS, Lee and his collaborators were the first to decipher one such zeolite's molecular structure last year.

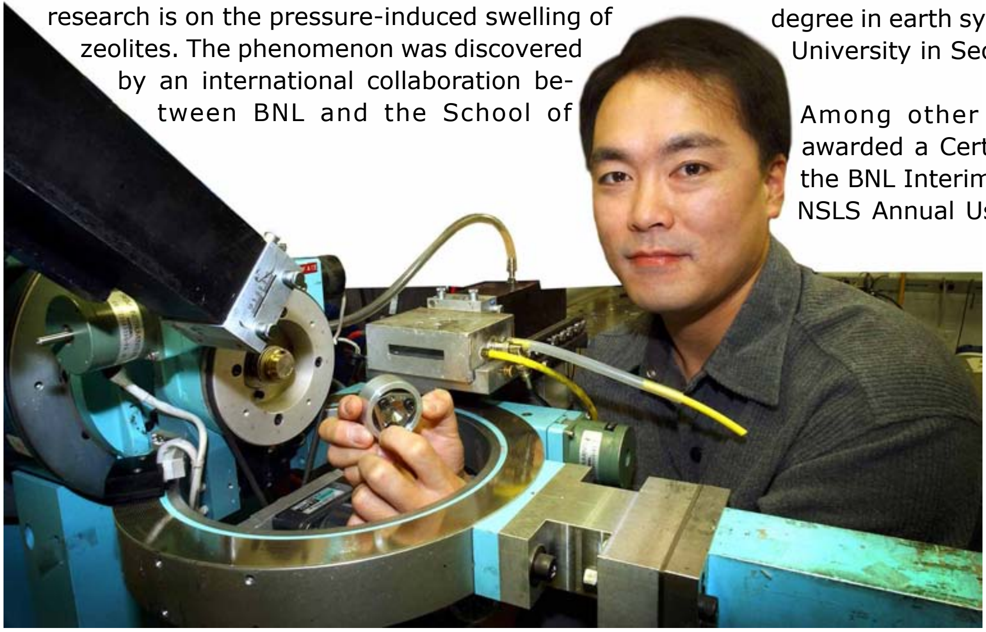
Their findings, published in the *Journal of the American Chemical Society*, for the first time explained the material's unusual ability to absorb excess fluid, and showed where the extra liquid goes.

Lee came to BNL in 2001 as a postdoctoral fellow in the powder diffraction group led by Thomas Vogt of the Physics Department, after completing his doctoral studies in geosciences at Stony Brook University. Lee had received a master's degree in geosciences from Stony Brook in 1998 and a bachelor's degree in earth system sciences from Yonsei University in Seoul, Korea (1996).

Among other honors, Lee has been awarded a Certificate of Excellence from the BNL Interim Director during the 2002 NSLS Annual Users' Meeting, a Sigma Xi Award for Excellence in Research at Stony Brook in 2001, and the Pauling Prize at the 2000 American Crystallographic Association Meeting.

-Karen McNulty

[Editor's note: Reprinted with permission from the BNL Bulletin - August 23, 2002.]



Yongjae Lee is seen with the diamond anvil cell that he used during the experiments on zeolites that expand at high pressure.

NSLS Summer Sunday

August 11, 2002

On Sunday, August 11th, Summer Sunday visitors came to BNL to take a tour of the National Synchrotron Light Source, where more than 2,500 visiting scientists from 350 institutions worldwide come annually to perform experiments. Visitors saw how infrared, ultraviolet, and x-ray synchrotron light produced in the NSLS is used for scientific research by guest scientists and BNL staff in biology, chemistry, medicine, physics, and many other fields, including criminal investigations.

In addition, visitors took guided bus tours of the Lab site that ran continuously throughout the day. A new hands-on exhibit called "Brain Teasers," which is a collection of 20 puzzles ranging from giant jigsaws to rope tricks that challenge both children and adults, was available in Berkner Hall. Also, local high school students demonstrated the robots they built. In addition, the "Whiz Bang Science Show," -popular with both adults and children - was shown at 10:30 a.m., noon, 1:30 p.m., and 3 p.m. every Sunday during the summer program. Tour hours are between 10 a.m. and 3 p.m. Admission is free and no reservations are needed, but, to be admitted on site, all visitors age 16 and over must bring a photo ID.

[Editor's note: Reprinted with permission from the BNL Bulletin - August 9, 2002.]

BNL Community Summer Science Program: Mount Sinai HS Student Studies at the NSLS

August 16, 2002

Despite evidence on walls and mirrors to the contrary, children's fingerprints can disappear faster than those of adults. This little-known fact can hamper investigations of kidnapping cases, which have been so prevalent in the news this summer.

But, this summer, this mystery has been studied at BNL: Lara Hershcovitch, who will be a senior at Mount Sinai High School this September, is using an infrared microscope at the National Synchrotron Light Source (NSLS) in an experiment to determine why adults' fingerprints can last longer on objects than children's prints do.

Hershcovitch is a participant in BNL's Community Summer Science Program, managed by the Lab's Office of Educational Programs (OEP) and funded by Brookhaven Science Associates.

Through this program, 26 high school students have been spending six weeks at BNL this summer in hands-on workshops or, as is Hershcovitch, in a research internship. All students also attend morning lectures on various scientific fields.

In her experiment, Hershcovitch is using the infrared microscope to study fingerprints from fathers and their sons ages 5-8, to determine the differences in the prints' chemical composition. Her data analysis may eventually be published in a scientific journal, and it could lead to more effective forensic investigations.

- Diane Greenberg

[Editor's note: Reprinted with permission from the BNL Bulletin - August 16, 2002.]

Working on the fingerprinting project together are: (from right) Lara Hershcovitch; Lisa Miller, a National Synchrotron Light Source (NSLS) Department scientist who volunteered to mentor Hershcovitch; and Jackie Tetenbaum, an NSLS guest technical collaborator who, as an undergraduate, participated at the Lab in the Energy Research Undergraduate Fellowship, another student program run by BNL's Office of Educational Programs.

UEC Lobbying

September 16 - 17, 2003

The UEC is vigorously continuing its lobbying efforts to increase the budget of the Office of Science at the Department of Energy, which funds the operation of the four DOE-run synchrotrons, and thus increase funding for the light sources. The feedback we are getting is overwhelmingly positive, owing largely to these grassroots efforts. During our spring visit, Richard Mertens, the Chief of the Energy Branch at the Office of Management and Budget (OMB) invited us, representatives from the four synchrotrons, to return in September to update him and make our case once again before they start working (in earnest) on the 2004 budget. Simon Bare and Leemor Joshua-Tor represented the NSLS during the visit on September 16-17. We came to realize that this would be a tough budget year, but we stressed the crucial need for an investment in the physical sciences, and the importance of an efficient and productive operation of the light sources facilities for future growth of the economy. The point was made that nearly 50% of growth of the U.S. economy in the last 50 years was due to Federal investment in scientific and technological innovation and that, now more than ever, we must not let that falter. We also explained that, because of essentially flat funding for operational support for the synchrotrons for more than a decade, the DOE is not getting its full return on its investment since the synchrotrons are not utilized to their full potential. We took this opportunity to meet with other officials, such as Kathie Olsen, the new Associate Director for Science, and Michael Holland, a senior policy analyst, at the Office of Science and Technology Policy (OSTP). We also met with professional staff members of the energy subcommittee of the House Committee on Science. We expressed our enthusiasm for the "Biggert Bill" (HR 5270) in the House, which calls for a substantial increase in funding for the Office of Science of the DOE. Though there appears to be sympathy for our mission, we as a user community cannot be complacent at this time.

We also visited the offices of Senators Hillary Clinton and Charles Schumer and the office of Congressman Felix Grucci. We presented each of them with a framed photograph of the NSLS with a personalized dedication from the NSLS users thanking them for their support.

Our trip ended with a meeting with Ray Orbach, the Director of the Office of Science, and Pat Dehmer, the Associate Director of Science for Basic Energy Sciences at the DOE. We updated them on our visits and discussed other issues important to users such as the PRT/CAT issue and plans for upgrades. We also wanted to ensure that our lobbying efforts would indeed benefit our users.

-Simon Bare

NSLS Annual Awards Ceremony and Picnic

September 21, 2002

On Friday, September 21, the NSLS had its annual Awards Ceremony and Picnic. This year, the festivities were highlighted by a pig roast instead of the traditional barbeque, an excellent suggestion by Boyzie Singh. The picnic was coordinated by Laura Miller and executed by a number of NSLS staff members, including Charlie Nielson, Boyzie Singh, Bob Best, Joe Greco, Paul Humbert, Jim Lacy, Phil Marino, Jim Newburgh, and Tom Seda.

NSLS Chairman, Steve Dierker, presented the Service and Spotlight Awards at the Annual Picnic.

This year, Service Awards were given to 20 NSLS staff members: Charlie Nielson (35 years); John Smith (30 years); Wayne Rambo, Malry Tardd, Peter DeToll, Bob Kiss, Bob Malone (25 years); George Jahnes, Norm Cernyar, Randy Church, Li Hua Yu, Tony Kuczewski, Al Borrelli (20 years); Michael Santana, Ron Beauman, Joe Greco, Andrew Mingino, Yoong Koh, Roger Hubbard, Jr., Tom Seda (10 years).

Spotlight Awards were presented to NSLS staff members for the completion of extraordinary accomplishments that were of significant benefit to the Department, Division, or Laboratory. This year's Spotlight Award winners were: (1) Bob Best, Patrick Moylan, Leonard Santangelo and Michael Schwarz for the reduction in Mini-Myte solenoid valve replacements which decreased machine and beamline downtime, (2) Michael Caruso for the timely design, assembly, and testing of a new joint system for a failed critical vacuum joint, (3) Ken Koebel for leading the NSLS Budget Group during the time period between Budget Group heads, (4) Michael Lehecka for designing and constructing a low-profile multi-axis alignment platform for magnet positioning, and (5) Corinne Messana for coordinating an important NSLS conference.

Photo on left: (from left to right) Jim Lacy and Nick Gmür (NSLS), and Ken Sutter (MSD). Top photo: Staff enjoying the pig roast.

NSLS Course on EXAFS Data Collection and Analysis

September 23 - 25, 2002

The first ever NSLS hands-on EXAFS Data Collection and Analysis Course was a resounding success! Twenty-eight students participated in the course held September 23-25, 2002. The course was co-organized by Simon Bare (UOP LLC), Bruce Ravel (Naval Research Laboratory) and Syed Khalid (NSLS), with administrative support by Lisa Tranquada (SFA, Inc.), and held at the NSLS.

The three-day course was divided into two mornings of lectures, two afternoons of hands-on data collection using six different NSLS spectroscopy beamlines (X9B, X11A, X18B, X19A, X23B, and X26A), and one full day of data analysis. The instructors on the NSLS beamlines were beamline scientists Nebojsa Marinkovic, Kaumudi Pandya, Wolfgang Caliebe, Syed Khalid and Tony Lanzirotti as well as Scott Calvin from NRL.

The morning lectures were given by Matt Newville (University of Chicago), Rob Scarrow (Haverford College), Simon Bare (UOP LLC), Bruce Ravel (NRL), Shelly Kelly (Argonne National Laboratory), Anatoly Frenkel (Yeshiva University), Grant Bunker (Illinois Institute of Technology), and Trevor Tyson (New Jersey Institute of Technology) on topics ranging from sample preparation and data collection to EXAFS data analysis and interpretation of XANES spectra.

The participants attending the course were both diverse in their scientific disciplines and in their geographic location and represented universities, national laboratories and industry. They attended the lectures in the morning, then were divided up into small groups by research discipline to conduct the experimental part of the course. Each student became familiar with beamline operation and collected data on standard materials as well as on samples from their individual research projects. On the final day, the participants learned data analysis techniques in a computer laboratory established specially for the course. The participants also enjoyed ample time for informal discussion over coffee and in the evenings over meals and drinks.

There was a tremendous amount of information disseminated over the three days. All the participants left the course with new friends and armed with the basic tools to apply x-ray absorption spectroscopy to their own research.

The course was sponsored by the NSLS, with partial support by Oxford-Danfysik and Canberra.

It is hoped that the course will be offered again in 2003 - stay tuned for more information!

-Simon Bare

Workshop Participants

Meeting Report: Low Energy Electrodynamics in Solids (LEES '02)

October 13 - 18, 2002

The 5th International Meeting on the Low Energy Electrodynamics in Solids (LEES) was held in Montauk, NY (USA) during the week of October 13-18, 2002. Nearly 100 scientists from the U.S., Canada, Europe and Asia traveled to the eastern end of Long Island to participate in the meeting. Historically, the LEES meetings have provided a forum for infrared (especially the far-infrared) spectroscopy of solids. During the dozen years since the inception of the LEES meeting series, the use of infrared synchrotron radiation for spectroscopy has flourished. At the same time, the energy resolution for angle-resolved photoemission spectroscopy and x-ray inelastic scattering has increased to the point that they are now powerful tools for the study of low energy phenomena. Consequently, it was no surprise that 20% of the presentations directly involved the use of synchrotron facilities.

K. Alex Muller (Zurich), the Nobelist for the discovery of the high T_c superconductors, delivered opening remarks and chaired a session addressing both spectroscopy in high magnetic fields and electron spin resonance (ESR). In this session, Laszlo Mihaly (Stony Brook) described how ESR is enhanced when coupled with very far-infrared synchrotron radiation and a high-resolution spectrometer. Jack Crow (NHMFL) surveyed the capabilities at the National High Magnetic Field Laboratory in Florida and New Mexico, including infrared and microwave spectroscopy, and also described plans for new European facilities. Many of the meeting attendees agreed that the addition of high-field magnets at synchrotron radiation beamlines would significantly advance the study of electronic and magnetic properties of materials.

Several sessions addressed electronic band structure, superconducting energy gaps and quasiparticle lifetimes in correlated electron systems. Walter Hardy (British Columbia) presented microwave spectroscopy results on the low energy electronic scattering in the high-T_c superconductor $YBa_2Cu_3O_{7-\delta}$ (YBCO) that agrees with a d-wave energy gap. High-resolution photoemission was the subject of presentations by Jim Allen (Michigan) and Tony Valla (Brookhaven), who each studied quasiparticle lineshapes in systems having reduced dimensionality. Juan Campuzano (Argonne) and Alessandra Lanzarra (Berkeley) discussed photoemission results for high-T_c superconductors, including electron-phonon interactions. Phonons and their interaction with electrons were also the focus of presentations by the Brookhaven infrared group (Chris Homes and Jiufeng Tu), and a

Some of the LEES 02 conference attendees enjoying a crisp October day in Montauk.

generalized theory for extracting electron self energies from infrared and optical reflectance data was described by Frank Marsiglio (Alberta).

Charge inhomogeneity was another recurring theme of the meeting. Sophie DeBrion (Grenoble) described a study of phase separation in manganites using ESR as a probe. S. Sridhar (Northeastern) explained his group's microwave measurement results for a variety of high-T_c cuprates in terms of charge/stripe ordering and inhomogeneity. Setsuko Tajima (ISTEC-Tokyo) used far-infrared spectroscopy to study the low frequency absorption in $La_{2-x}Sr_xCuO_4$, and suggested that charge inhomogeneity may explain the excess absorption observed at low frequencies in the superconducting state. This excess absorption has consequences for optical sum rules and calculations of the condensate energy for superconductors. Dirk Van der Marel (Groningen) discussed the shift in spectral weight observed in the optical conductivity of systems undergoing a phase transition. The energy scale over which the spectral weight shifts is expected to be a measure of the energetics driving the phase transition itself. Andy Millis (Columbia) presented a more detailed theoretical discussion of the f-sum rule and its application to perovskites. Optical sum rules are also being used by Dimitri Basov (San Diego) to understand ferromagnetism in $Ga_{1-x}Mn_xAs$ and other magnetic III-V semiconductors. In all cases, accurately tracking spectral weight was dependent on precise spectroscopic data to very low frequencies.

John Hill (Brookhaven) and George Sawatzky (British Columbia) each described synchrotron x-ray studies of low-dimensional, correlated electron systems. John Hill used resonant inelastic x-ray scattering to study electronic excitations, achieving 0.3 eV energy resolution, sufficient to observe cross-gap excitations and excitons. In contrast to infrared, the larger momentum of x-rays allows one to probe k-space away from the zone center and track the dispersion of various excitations. George Sawatzky discussed x-ray absorption spectroscopy, using the facilities at the X1B beamline of the NSLS, to probe charge order in highly correlated electron systems. The ideas of stripe order, reduced dimensionality, and high-T_c superconductivity were nicely tied together in a theory presentation by Steve Kivelson (UCLA).

Superconductivity in MgB_2 continues to attract interest, with increasing evidence for multiple energy gaps. Leo Degiorgi (ETH-Zurich) used infrared reflectivity measurements on a mosaic of MgB_2 crystals, and detected a rather small energy gap. Ferenc Simon (Budapest) used ESR and microwave absorption to study electronic scattering and the critical field for MgB_2 powders. Conventional metallic behavior was observed in the normal state, but the magnetic field dependence in the superconducting state suggests a significant field-induced normal component, evidence for critical field anisotropy. Other presentations by Jure Demsar (Los Alamos) and Larry Carr (NSLS - Brookhaven) described time-resolved studies of non-equilibrium dynamics in MgB_2. Photoexcitation drives the electronic system away from equilibrium, and the relaxation process is probed by synchrotron THz spectroscopy. Whether the system was strongly perturbed (Demsar) or weakly perturbed (Carr), the relaxation process pointed to at least two energy gaps.

The main conference room at the Montauk Yacht Club was just sufficient for the nearly 100 participants.

Time-resolved spectroscopy was also discussed in the final session of the meeting. Joe Orenstein (Berkeley) has studied the relaxation of photoexcited excess quasiparticles inYBCO and BiSrCaCuO high-T_c superconductors using 100 fs laser pulses and THz pulses. In contrast to s-wave (isotropic) superconductors, the excess quasiparticles do not experience a phonon bottleneck that typically masks the intrinsic quasiparticle recombination rates. Rick Averitt (Los Alamos) presented a time-resolved study of collosal magneto-resistance manganites using laser-produced THz pulses. Optical excitation leads to a decrease in the far-IR conductivity, corresponding to a shift in spectral weight into the near-IR. The relaxation mechanism was found to depend on the magnetic state of the material (via spins below T_c and phonons above T_c). Time-resolved spectroscopy using pulsed infrared synchrotron radiation was described by David Tanner (Florida), and results for both electron-hole recombination (semiconductors) and excess quasiparticle recombination (superconductors) were presented.

Efforts are underway at many synchrotron facilities to produce shorter duration pulses of light for time-resolved spectroscopy. When the electron bunches are very short, coherent synchrotron radiation (CSR) at THz frequencies results. Gwyn Williams (Jefferson Lab) described measurements of high power THz produced as CSR in their energy recovery linac. A combination of relativitistic effects, plus the high average current of their accelerator, led to an average output power of 20 watts. Gwyn described a variety of applications in solid state physics as well as THz imaging. CSR has been observed in storage rings too, but the average beam current must be kept quite low to avoid bunch-shape instabilities. Still, the resulting sub-THz radiation may prove useful for some experiments, including time-resolved spectroscopy. Mike Martin (ALS) described an idea for a new storage ring dedicated to infrared spectroscopy, including a "coherent mode" for producing sub-picosecond duration pulsed THz radiation.

Despite the stormy weather on Wednesday, many of the conferees attended a winery tour where they were introduced to some local wines and pleasant scenery. Following the conference banquet that evening, the participants were treated to a concert by Zurab Ninua (Baritone) and Natia Astakhishvili (pianist), who performed musical selectrions from the works of Mozart, Tchaikovsky, Verdi and others. The meeting concluded with a Friday afternoon tour of the infrared and photoemission beamlines on the VUV-IR ring at the NSLS, and discussions on spectroscopy instrumentation and techniques. The solid state physics community looks forward to the next LEES meeting which is planned for the summer of 2004 in Germany.

The conference was supported by the National Synchrotron Light Source and its Users Executive Committee, Brookhaven National Lab and the U.S. Department of Energy, Brookhaven Science Associates, Stony Brook University and several equipment vendors. The administrative support of Corinne Messana (NSLS) and Arlene Rementer (BNL) is also gratefully asknowledged.

-G. Lawrence Carr

Learning from Light on "Take Our Sons to Work" Day at the NSLS

October 14, 2002

On October 14, over fifteen sons learned about work done at the NSLS, and even performed their own scientific experiments. The one-day visit was part of the national "Take our Sons to Work Day". At the NSLS, the boys learned that the facility produces light that can take many forms, from infrared to x-rays, and this light is used for applications in many fields of science.

NSLS sons were part of over 50 boys that participated in BNL's annual "Take Our Sons to Work Day".

NSLS scientist Marc Allaire offered a tour of the experimental floor to the boys. They saw close-up how a beam line is designed for macromolecular crystallography, a technique in which light is diffracted from tiny protein crystals in order to determine their structure. Later, the boys toured the Control Room, where Machine Operator Gary Weiner told them how synchrotron light is made. The boys were full of questions and were very interested in operating the controls themselves.

At the end of the program, the boys had the opportunity to perform their own scientific experiments with light. They first observed simple reflection of light from a mirror. They then learned about the process of diffraction by reflecting red laser light from the world's most popular diffraction grating — a CD-ROM. But perhaps the most exciting moment was when the boys created fantastic rainbow patterns by diffracting visible white light from the CD-ROM.

-Marc Allaire

377th Brookhaven Lecture
NSLS's Elio Vescovo Talks On Thin Magnetic Films

October 16, 2002

Over the past few years, scientists have been developing new materials for information storage applications, often in the form of thin-layered films.

The technology has evolved to the point where individual layers may be only a few nanometers - literally just tens of atoms thick. Such multi-layered structures not only promise to go beyond the limits of the memory density of current computers, but also display a variety of complex magnetic properties that scientists are only beginning to understand.

For example, scientists can assemble many layers of ultra-thin films, creating structures with electrical properties that can dramatically change depending on the values of an applied magnetic field, a phenomenon called giant magnetoresistance. Another puzzling property is the co-existence, in some ultra-thin magnetic films called half-metallics, of both metallic and insulating properties.

Elio Vescovo, physicist at the National Synchrotron Light Source (NSLS), has been investigating the properties of these intriguing ultra-thin magnetic films for the last five years. He uses a technique called photoemission, for which x-rays generated by the NSLS are projected toward a sample of thin films, and electrons ejected from the sample are further studied to reveal its properties.

Vescovo is currently responsible for the spin-resolved photoemission program at NSLS beam line U5UA, where he has been working since 1995. Vescovo received his Ph.D. in nuclear engineering and a Ph.D. in physics from the Polytechnic Institute of Milan, Italy.

-Patrice Pages

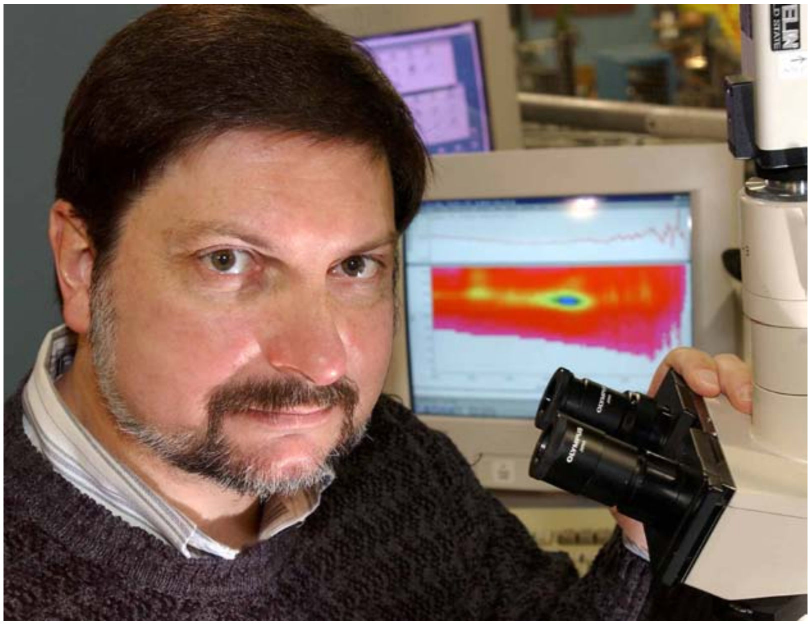
Elio Vescovo

[Editor's note: Reprinted with permission from the BNL Bulletin - October 11, 2002.]

Five BNL Scientists Are Granted Tenure

December 13, 2002

As described in the Scientific Staff Manual, a tenure appointment constitutes "recognition of independent accomplishment of a high order in the performance of original research or of other intellectually creative activity appropriate to the purposes of the Laboratory. Recognition may be earned through significant contributions to knowledge related to the purposes of the Laboratory and/or in furtherance of the aims of the Laboratory, through continuing contributions of outstanding significance to productive uses of the facilities, or through outstanding and creative contributions to their design, development, and improvement."



Larry Carr

Brookhaven Science Associates (BSA) has granted tenure to five more Brookhaven scientists. They are: Lawrence Carr, National Synchrotron Light Source Department; Arokiasamy J. Francis, Environmental Sciences Department (ESD); George Hendrey, ESD; Alexei Tsvelik, Physics Department; and Rajagopal Venugopalan, Physics.

Tenure appointments are granted by the BSA Board of Directors after a rigorous selection procedure overseen by the BSA Science & Technology Steering Committee.

In making tenure decisions, the BSA Board is advised by Brookhaven Council members, who are tenured scientific staff, elected by the scientific staff of their respective departments and divisions. A description of the accomplishments of each of the five newly tenured scientists will appear in The Bulletin over successive weeks, starting this week with Lawrence Carr.

Lawrence Carr, NSLS Department

Larry Carr, a physicist in the National Synchrotron Light Source (NSLS) Department was recommended for tenure for his outstanding research accomplishments in solid-state physics based on far-infrared spectroscopy.

"Larry has either brought to the NSLS or characterized all of the new infrared experimental sources and techniques in use at BNL today," said Steve Dierker, NSLS Chair. "He has assumed the lead role in all aspects of infrared spectroscopy at the NSLS, from its production to multiple fore-front mid- and far-infrared scientific programs. The NSLS infrared beamlines under Larry's control are acknowledged leaders worldwide."

In addition to his infrared beamline development work, Carr has made significant contributions to the advancement of accelerator physics at the NSLS.

In 1998, he made the first observation of coherent emission of very far infrared radiation in the vacuum ultraviolet storage ring, one of the two NSLS rings. While not the first observation worldwide, Dierker notes, it was the second, and it garnered much interest in the accelerator physics community at large.

Larry Carr earned his Ph.D. in physics from Ohio State University in Columbus in 1982. He joined BNL in 1996 as an associate physicist, becoming a physicist in 1998.

— Patrice Pages

[Editor's note: Reprinted with permission from the BNL Bulletin - December 13, 2002.]

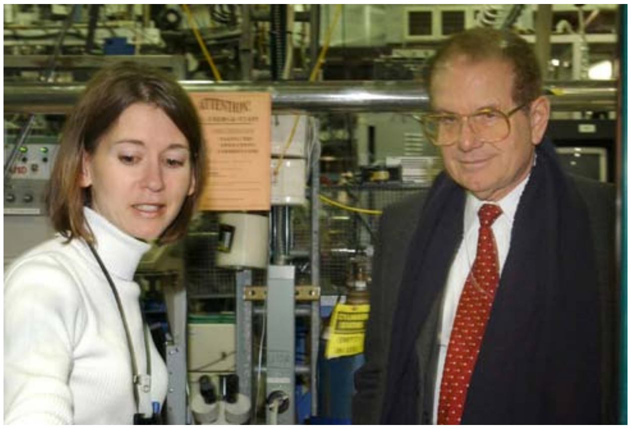
DOE Office of Science Director Orbach, BNL Director Paul Welcome U.S. Representative Timothy Bishop to BNL

December 30, 2002

On Monday, December 30, Raymond Orbach, the Director of DOE's Office of Science, journeyed to BNL to meet with U.S. Representative Timothy Bishop, the newly elected First District Congressman, who was visiting the Lab for the first time. The visitors, who included Office of Science advisor Todd Harding and Congressional staffer Lee Leshen, were welcomed by Peter Paul, BNL Interim Director, and Michael Holland, Manager of DOE's Brookhaven Area Office (BAO).

At the DOE Brookhaven Area Office on December 30 are: (from left) Peter Paul, BNL Interim Director, Timothy Bishop, then U.S. Representative-elect; Raymond Orbach, Director of DOE's Office of Science; and Michael Holland, Manager of DOE Brookhaven Area Office.

Highlights of Bishop's brief tour of the Lab included a stop at the National Synchrotron Light Source (NSLS). Doon Gibbs, Interim Associate Laboratory Director for Basic Energy Sciences, and NSLS Chair Steven Dierker, explained a proposed NSLS upgrade which will dramatically improve the capabilities available to the approximately 2,500 researchers from scientific institutions and industry who use the NSLS for their research each year. They also discussed plans for the new BNL Center for Functional Nanomaterials (CFN), for which Secretary of Energy Spencer Abraham announced DOE approval when he visited the Lab last June 14.



Researcher Lisa Miller, National Synchrotron Light Source Department, and Raymond Orbach, Director of DOE's Office of Science.

Bishop, who was sworn in on Tuesday, January 7, and Orbach then visited the staff on the NSLS experimental floor and saw research results on osteoarthritis and the makeup of newly formed bone. They also met with facility-users whose findings about human papillomavirus may lead to drugs to prevent cervical cancer. At their next stop, the Relativistic Heavy Ion Collider (RHIC), the visitors heard from Tim Hallman of the Physics Department about the world's most energetic heavy-ion reactions which are recreating conditions that existed in the first few microseconds of the universe, and they saw collision events recorded with the huge STAR detector.

Bruce Gibbard, Physics, who directs the RHIC Computing Facility, described the technical effort needed to accept, translate, and store the RHIC collision events with thousands of tracks and make them available to about 1,000 users simultaneously. The tour ended with an overview of BNL's pioneering neuroimaging research on the brain chemistry of addiction, mental illness, and aging, from Nora Volkow, Associate Laboratory Director for Life Sciences. David Schlyer, Chemistry Department, also explained some recent research that includes imaging of awake animals.

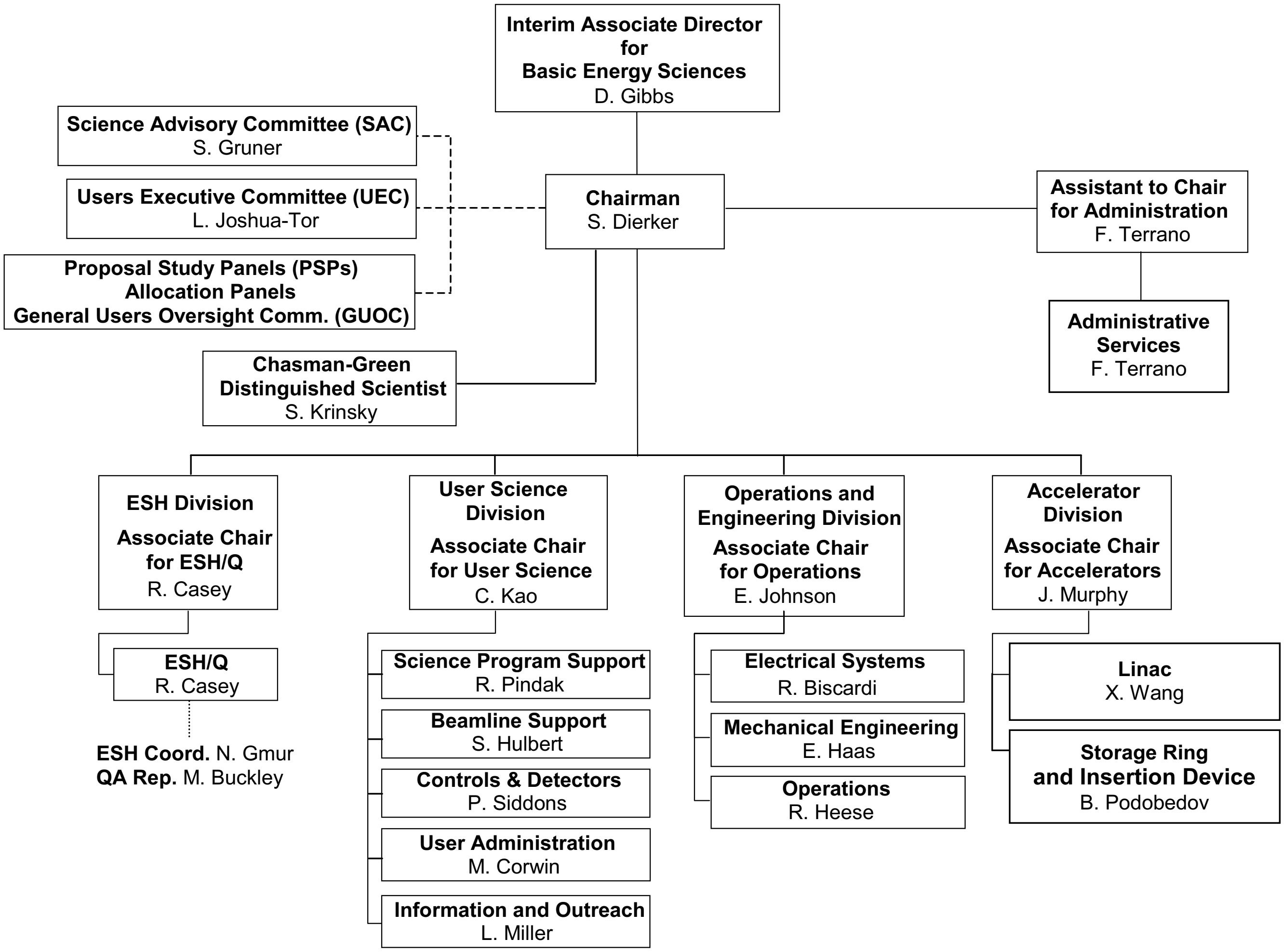
-Liz Seubert

[Editor's note: Reprinted with permission from the BNL Bulletin - January 23, 2003.]

U.S. Representative Timothy Bishop at BNL's National Synchrotron Light Source.

NSLS Organization

NSLS Organization Chart



NSLS Advisory Committees

2001 - 2002 Users' Executive Committee

The Users' Executive Committee (UEC) provides for organized discussions among the user community, NSLS administration, and laboratory directorate. It aims to communicate current and future needs, concerns, trends within the user community to NSLS staff and management, and to disseminate to the users information about NSLS and BNL plans.

Chair
Leemor Joshua-Tor
Cold Spring Harbor Laboratory

Vice-Chair
Antonio Lanzirotti
U. of Chicago

Past Chair
Simon Bare
UOP LLC

Secretary
Bruce Ravel
Naval Research Laboratory

Member
Steven Almo
AECOM

Member
Fred Dyda
NIH

Member
Daniel Fischer
NIST

Member
Anatoly Frenkel
Yeshiva U.

Member
Richard Reeder
SUNY @ Stony Brook

Ex-Officio Member
Mary Anne Corwin
NSLS User Administraton Head

Ex-Officio Member
Chi-Chang Kao
NSLS User Science Head

Ex-Officio Member
Lisa Miller
NSLS Information and
Outreach Head

Special Interest Group Representatives

Special Interest Groups in areas of common concern communicate with NSLS management through the UEC.

Bio. Scatt. & Diffr.	Michael Becker, BNL/Biology
Imaging	Susan Wirick, SUNY @ Stony Brook
Industry	Paul Stevens, Exxon Research & Eng. Co.
Infrared	Laszlo Mihaly, SUNY @ Stony Brook
Nuclear	Mahbubul Khandaker, Thomas Jefferson Nat. Lab.
Scattering	Peter Stephens, SUNY @ Stony Brook
Students/Post Docs	Udupi Ramagopal, BNL/NSLS
Timing	John Sutherland, BNL/Biology
Topography	Michael Dudley, SUNY @ Stony Brook
UV Photoemission	Peter Johnson, BNL/Physics
XAFS	Bruce Ravel, Naval Research Lab

Science Advisory Committee

The Science Advisory committee (SAC) evaluates science programs at the NSLS and makes recommendations to the Chairman.

Mario Amzel, Johns Hopkins U.
Martin Blume, BNL/Physics
Stephen Burley, Rockfeller U.
Sol Gruner, CHESS/Cornell U.
Franz Himpsel, U. of Wisconsin, Madison
Keith Hodgson, SLAC
Leemor Joshua-Tor, Cold Spring Harbor Lab.
Jochen Schneider, HASYLAB/DESY
Albert J. Sievers, Cornell U.
Sunil Sinha, U. of California, San Diego
Doon Gibbs, BNL
Peter Paul, BNL

NSLS Advisory Committees

General User Proposal Study Panel

The Proposal Study Panel (PSP) reviews and rates General User Proposals. Members are drawn from the scientific community and generally serve a two-year term.

VUV Science
- Daniel Fischer, NIST
- Fredrich Hoffman, Sci-Med
- Jan Hrbek, BNL

X-Ray Biology
- Seth Darst, Rockefeller U.
- DaXiong Fu, BNL
- Da-Neng Wang, NYU
- Rui-Ming Xu, Cold Spring Harbor

X-Ray Imaging
- George Flynn, SUNY @ Plattsburgh
- Richard Reeder, SUNY @ Stony Brook

X-Ray Spectroscopy
- Anatoly Frenkel, Yeshiva U.
- Douglas Hunter, U. of Georgia
- Trevor Tyson, NJ Inst. of Tech.

X-Ray Scattering
- Ben Hsiao, SUNY @ Stony Brook
- Rainer Kolb, ExxonMobil
- Karl Ludwig, Boston U.

Allocation Panel

The Allocation Panel allocates General User beam time to both new proposals and Beam Time Requests based on ratings provided by the Proposal Study Panels. Members are drawn from the scientific community and generally serve a two-year term.

VUV	Laszlo Mihaly, SUNY @ Stony Brook Elio Vescovo, BNL/NSLS
X-Ray	Marc Allaire, BNL/NSLS Wolfgang Caliebe, BNL/NSLS Elaine DiMasi, BNL/Physics Jean Jordan-Sweet, IBM Syed Khalid, BNL/NSLS Craig Ogata, HHMI

General User Oversight Committee

The General User Oversight Committee resolves disputes between General Users, PRTs, and NSLS staff.

Simon Bare
UOP LLC

Mark Chance
Albert Einstein College of Medicine

Barbara Illman
U. of Wisconsin

Dale Sayers
North Carolina State U.

Facility Report

Operations, Engineering, and Facility Report

Erik D. Johnson

Associate Chair for Operations & Engineering

Organization and Mission

What is now known as the Operations and Engineering Division was formed in late 2001 through reorganization of the NSLS where the Operations, Electrical, and Mechanical sections were collected into one division. The mission of the division falls into three areas:

- Design, fabrication, and maintenance of the NSLS accelerators and utilities including upgrades and modifications to meet changing needs
- Operation of NSLS complex 24 hours a day, 7 days a week, on average 44 weeks each year
- Engineering and technical support for the other NSLS divisions and the NSLS user community

2002 Activities

The work of our division touches on a great deal of infrastructure that is largely hidden from our user community until something goes awry. The NSLS utilities complex provides 1500 Tons of cooling capacity for HVAC, three accelerator water systems and the experimental (users) water system. It also provides compressed air for the facility. The NSLS uses approximately 10 megawatts (MW) of electric power, much of which is distributed to the accelerator complex and the experimental stations. The power systems group has over 1600 pieces of maintenance-tracked equipment including almost 100 high power systems that require two trained people to service. They also support operations and development of the pulsed power systems in the complex, which includes the linac with its klystrons, and the 14 'kicker' pulsed magnets that shuttle the beam from one machine to the next. The radio frequency (RF) group develops and maintains 9 high power 'sockets', the RF drive systems for the facilities 7 high power RF cavities. The physical infrastructure of our accelerator complex, including vacuum, magnets, diagnostics, and controls, is also managed by our division, with over 25000 drawings on file for our custom components.

Perhaps a bit more visible to the user community is the support for the insertion device program, including the 25 W liquid helium plant for the X-17 superconducting wiggler. Support for the design, fabrication and installation of the new Mini Gap Undulators, (MGU's) in collaboration with the Accelerator and User Science divisions of NSLS, is also provided by the Operations and Engineering Division.

The organization has 80 NSLS staff members and 7 skilled trades from the laboratory, as shown in the table below. We increasingly rely upon a department-wide matrixed management approach; Operations and Engineering draws resources from other divisions to support operations, and we provide special expertise to support development for other divisions.

Operations reliability (operations during scheduled time) and availability (operations compared to total scheduled time) remained high overall during the year, as shown in the 2002 Ring Performance and Usage figures, page 6-11, with most of the down time attributed to a few events. On the VUV ring, there was a failure of the transport line shutter bellows that resulted in 34 hours of downtime in April, and a failure and rebuild in-place of the RF transmitter in September, which resulted in 64 hours of lost operations. All other events were under a day down time, with the vast majority causing disruptions or delays of operations of 15 minutes or less.

Operations and Engineering Division Staffing

	Operations	**Electrical Systems**	**Mechanical Engineering**	
	Operations Mgmt. Operators Coordinators	RF and Power Systems Acc'l and Computer Cnt'ls Controls and Diagnostics Electrical Design Electricians	Engineering Design Mechanical Utilities Vacuum Shop/Trades	**80**
Sci	1			1
Prof		13	8	21
Tech	13	21	24	58
Trade		5	2	**7**

During 2002, the division supported a number of planned projects, as well as the ‘rapid responses’ to equipment failures as noted above. Our planned activities included installation of the new dipole chamber for X29, and support for installation of the X17 hutch and infrastructure to expand their user program. We also installed a third experimental water system pump to improve its reliability, and a third new RF cavity in the X29 straight section of the ring. The modifications of the X29 front end were initiated to pave the way for a new MGU, to be installed in this recovered space in order to add another insertion device to the complement of NSLS facilities. Operations and Engineering provided support to the DUV-FEL program through the installation of the NISUS shielding system, extension of the interlock systems to include the FEL, and the construction and installation of the HGHG energy modulator and its gap separation mechanism.

As an engineering-oriented organization, much effort is focused on projects both for customers within the division, and for people in the larger NSLS community. In vetting projects we apply the following guidelines to allocate our resources:

- Maintain 95% reliability target for user operations
- Deal with critical items for operations (imminent failure anticipated)
- Update equipment for continued ops through up grade (5 to 10 year horizon)
- Update equipment to reduce maintenance
- Provide engineering, technical and operational support for department projects

With these objectives in mind, the following projects were selected for 2002/2003 funding. While all of these projects are in support of the user program, many fall into the realm of hidden infrastructure. Those that will be more directly visible to our user community are highlighted in italics.

Looking further ahead, the division is working hard, along with rest of the NSLS and its user community, to define and develop the next generation of NSLS facility. What form it may take is yet undecided, but the Operations and Engineering Division is ready to provide its collective experience, and has the desire to bring it to fruition for our user community.

Electrical Section

XRF Test 1 Plate Power Supply Upgrade
Back-leg Bump
X17 Refurbish Controls
Replace Linac Electron Gun
Upgrade Spare Modulator
EPICS for NSLS Controls
New Graphical Local Bump Generator
Orbit Correction Program Upgrade
Power Supply Regulator Upgrade
XRF1 Circulator and Switch
X13 EPW Control Upgrade
Iden & Rem of Control room cables
Iden of disconnect means for panels
Corr of X-ray Trench to NEC
VUV Interlock Upgrade
X-ray BPM electronics relocation
X-ray SRU & Hutch logic DC conversion

Operations Section

Klystron Drive Amplifier
Hi Level Controls port to LINUX
New Control System Device Types
New Klystron
Rebuild Modulator 1
X-ray Trim Magnet Upgrade
New Software for Dbase Micros
GPLS Board Upgrade

Mechanical Section

Third Experimental Water Pump
Auto Milling Machine
Ceramic Chamber Transition Cooling
X-29 Beamline Project

Other Divisions projects mg’d by OED

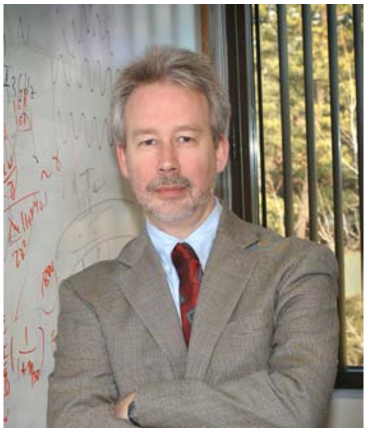
Beamline Electrical Safety Inspection
Minigap Undulator
Superconducting Undulator Devel.
Strain Mapping End Station Devel.

Accelerators Report

James Murphy

Associate Chair for Accelerators

Organization and Mission



The NSLS Accelerator Division (AD) was established in late 2001 through reorganization of the NSLS and is headed by James B. Murphy. The division is organized into two sections: the Linear Accelerator (Linac) Section, headed by Xijie Wang, and the Storage Ring & Insertion Device Section, headed by Boris Podobedov. The staff consists of eight accelerator physicists, two engineers, three technicians and three postdocs.

The NSLS Accelerator Division (AD) has a four-part mission:

- To ensure the quality of the electron beam in the existing NSLS linear accelerator and storage rings: the x-ray and vacuum ultraviolet rings;
- To operate the NSLS Magnet Measurement Laboratory;
- To participate in the NSLS upgrade;
- To perform fundamental research and development in accelerator and free electron laser physics, e.g., the deep ultraviolet free electron laser (DUV-FEL) experiment.

2002 Activities

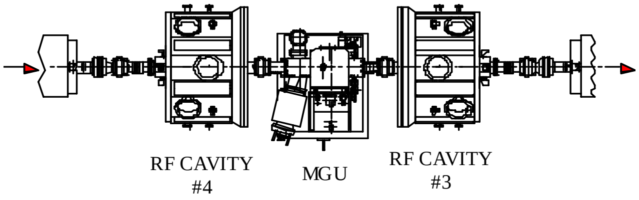
The AD staff maintains and improves the electron beam quality in the NSLS accelerator complex through research and development and weekly machine studies programs. More than a decade ago, the NSLS developed the first global orbit feedback system for a storage ring light source. This early system was based on analog components. In the intervening years, digital technology has exploded and it became clear that continued improvements in the global feedback system could only be realized by making the transition to digital technology. The AD staff, working in close collaboration with the Operations and Engineering Division staff, has developed and implemented digital orbit feedback systems on both storage rings. Key advantages of the digital feedback systems include: a) scalability to a larger number of beam position monitors and correctors; b) more sophisticated correction algorithms; c) potential for machine diagnostics; d) ease of maintenance due to compact system size.

Accelerator Division and Operations & Engineering Division staff designed and built an economical and yet effective digital system using the original architecture based on commercially available virtual machine environment (VME) boards. The system includes all the beam position monitors and most of the correctors, and is running at a sample/update rate of five kilohertz, leading to correction bandwidths in excess of 100 hertz and limited only by the bandwidth of the correctors.

The VUV ring digital global orbit feedback was used in regular operations for over two years. A 60-hertz digital notch filter, which eliminates the power line noise on the vertical orbit, was recently added to the ring, demonstrating the flexibility afforded by the digital design.

The x-ray ring system was recently enhanced to perform both the global and local corrections at some of the beamlines. In particular, the gain-bandwidth product or the residual orbit noise of the system correcting the vertical orbit motion, which was put into operation in September 2002, performed significantly better than the old analog systems. This system has also proved to be very effective in pinpointing the sources of orbit disturbance. Work is ongoing to make the x-ray ring horizontal system operational in fiscal year 2003.

In 2002, the Magnetic Measurement Lab efforts centered on assembling, measuring and shimming the magnetic arrays for the next mini-gap undulator, called MGU-29. The magnet arrays are now ready to be mounted in their support structure and vacuum vessel, which are also nearing completion. MGU-29 will be installed in May 2003 between the two radio-frequency accelerating cavities in the X29 straight section of the x-ray ring, to serve a new protein crystallography beamline funded by the National Institutes of Health.



2nd in-vacuum Mini-Gap Undulator will be installed between pair of RF cavities in X29 straight section to serve a new NIH-funded PX beamline

Other ongoing projects include:

- Design study of a novel, variable polarization undulator with quasi-periodic magnet arrays, to replace the X1 soft x-ray undulator
- Design study of superconducting, short-period undulators to generate tunable hard x-rays in the 2-20 keV range in the present x-ray ring, as well as for the proposed NSLS Upgrade
- Participation with the Stanford Linear Accelerator Center in Menlo Park, California, the Advanced Light Source in Berkeley, California, and the Ad vanced Photon Source in Argonne, Illinois, in a collaborative proposal to the Department of Energy (DOE) for research and development on a superconducting undulator
- Magnetic design for a proposed permanent-mag net, in-vacuum, hard x-ray undulator to replace the X25 wiggler

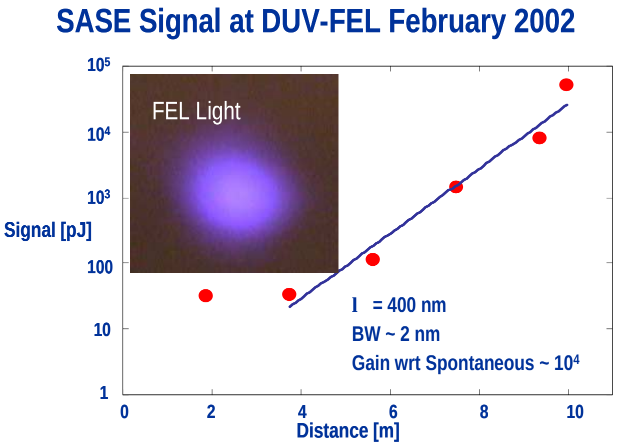
A primary emphasis of the AD in 2002 was to explore options for future light sources for the NSLS. The existing NSLS storage rings were designed more than a quarter of century ago. Although they continue to operate with exceptional reliability and high flux, they have been eclipsed by third-generation light sources. To maintain the NSLS at the cutting edge will require development of new light sources at the NSLS; high brightness storage rings, energy recovery linacs, and free electron lasers were all considered as part of this investigation. The NSLS is collaborating with its user community to refine the requirements of any new source and is working with DOE to ensure that the NSLS is in line with DOE's long-term plans for their facilities.

The scientists, engineers, and technicians working on the DUV-FEL experiment are developing advanced free electron laser sources and their scientific applications for the NSLS user community. The DUV-FEL laboratory consists of a radiofrequency photoinjector driven by a solid-state titanium sapphire laser system which provides an electron beam that is sent inside a linear accelerator, where the electrons' energy is increased to 200 MeV. To generate the high peak current required for the FEL, a four-magnet chicane bunch compressor is installed at the midpoint of the linear accelerator. To generate the FEL radiation, the electron beam traverses a 10-meter long NISUS undulator (Near Infrared Scalable Undulator System).

In 2002, tremendous progress has been made at the DUV-FEL. In February, the FEL generated spontaneous self-amplified emission (SASE) at 400 nanometers with a gain of 10^5. SASE at 266 nanometers with a gain length of 65 centimeters was subsequently measured in August, followed, in September, by the first achievement of saturation of an FEL seeded with 266-nanometer light from the titanium sapphire laser.

In the third quarter of 2002, the staff working at the DUV-FEL installed an evacuated laser transport line and matching optics for a High Gain Harmonic Generation (HGHG) FEL. After completion of the HGHG hardware installation in October 2002, HGHG saturation at 266 nanometers was obtained using 800-nanometer laser seeding, and measurements were performed to both characterize the HGHG output radiation and benchmark the performance against theory. An important benefit of achieving saturation in the HGHG FEL is that radiation at the third harmonic (89 nanometers) was also generated with an intensity of about one percent of the intensity at 266 nanometers.

Users of the FEL light performed the first chemistry experiment in December 2002 by using the 89-nanometer light to perform ion pair imaging spectroscopy. Two other BNL chemists have submitted proposals to explore the potential of the DUV-FEL radiation. The high intensity VUV light provided by the DUV FEL is complementary to the VUV ring sources and opens new horizons for the NSLS user community.



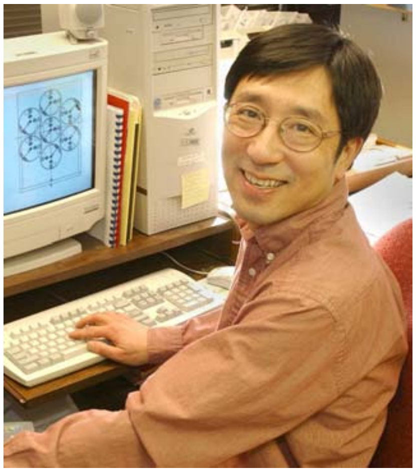
Gain length **L_G=0.9 m**

User Science Report

Chi-Chang Kao

Associate Chair for User Science Division

Organization and Mission



The User Science Division was established in late 2001 through reorganization of the NSLS. The mission of the division is to coordinate major facility activities related to users in order to:

- Be more effective in communicating with the user community
- Strengthen existing scientific programs
- Foster the growth of new scientific programs
- Raise the visibility of the exciting science produced by our users.

The division consists of five sections: User Administration (Mary Anne Corwin), Information and Outreach (Lisa Miller), Beamline Development and Support (Steve Hulbert), Scientific Program Support (Ron Pindak), and Detectors and Controls (Peter Siddons).

2002 Activities

During 2002, with the help of many users, the User Science Division organized more than a dozen scientific workshops to promote the exchange and stimulation of new ideas. The topics of these workshops included research frontiers ranging from biology to materials science, and the development of new synchrotron experimental techniques. These workshops have been instrumental in helping us identify new scientific opportunities and user communities, guide the research/development effort and the allocation of resources within the division and the department, and formulate new funding proposals in collaboration with user groups. This effort has resulted in several new projects at the NSLS funded by the National Science Foundation (NSF), the Department of Energy (DOE), and the Department of Defense (DOD). These include a facility for advanced materials processing, a high-resolution infrared spectrometer, a high-resolution x-ray spectrometer, and a major nanoscience research program on soft condensed matter.

In addition, two multi-year NSF research resources, the Consortium for Materials Properties Research in Earth Science (COMPRES) and the Center for Environmental Molecular Science (CEMS), were established at Stony Brook University last year. The wide range of experimental capabilities at the NSLS and the close coordination of effort between the NSLS and user groups are crucial to the success of both resources. We expect that these two research resources, similar to the very successful National Institutes of Health (NIH) research resources at the NSLS, will form the focal point for the earth science and environmental science user communities in the development of new experimental techniques as well as in outreach and education activities. In order to meet the increasing demand for beamtime by these two user communities, the NSLS has initiated a major upgrade of the X17 superconducting wiggler beamline and the construction of a new x-ray microprobe endstation.

A program to upgrade the beamline controls infrastructure at the NSLS has begun. Prototype systems were built and tested in 2001, and a recommended set of hardware and software generated. The system is based on the Experimental Physics and Industrial Control Systems (EPICS) software used at several other synchrotron facilities, and leverages the large effort that has been coordinated by those institutions. During 2002, beamlines X1B, X12A and U5U were upgraded to this new EPICS-based control system, and components were ordered to upgrade beamlines X13 and X21. Beamlines X12B, X12C, X8C, X25, X26A and X26C were also upgraded using funds from other sources (mainly NIH). During 2003, beamlines X21 and X13 will be upgraded using components procured in 2002. Components will be procured for an X17 upgrade. Other beamlines will be upgraded as resources allow.

There is a real need for advanced detector development for synchrotron applications that is not currently met by commercial devices. We have started three small efforts in this direction. One of them is a silicon detector designed to optimize EXAFS and XANES spectroscopy of dilute systems. To this end, it will have nearly 400 independent detector elements and associated counting chains, increasing by more than an order of magnitude the counting rate capability of such systems. The readout system is realized as a custom integrated circuit, with each chip handling 32 channels. The system is completely computer-controlled, and the user software is designed to minimize operator intervention in its calibration and setup. A prototype having 96 channels is operational and undergoing characterization at a beamline in order to develop the user interface and understand the limitations of the device. We have also targeted powder diffraction as an area where advanced detectors could bring benefits. We have built a high-speed curved gas detector that covers a 45-degree segment of the diffraction pattern, and is capable of acquiring events at more than 3 MHz (**Figure 1**). This device is also operational and waiting for beamline tests. Since silicon-based detectors and gas detectors all become inefficient at higher photon energies, we are performing R&D into higher-Z materials suitable for use as x-ray detectors. In particular, Cadmium Zinc Telluride is available with good quality and we have begun fabricating devices with this material. We hope to have useful detectors before the end of this year.

The macromolecular crystallography community at the NSLS has been extremely productive, as shown in the latest survey conducted for the BioSync report released in October 2002. This report emphasizes the importance of the NSLS as a national resource for structural biology and strongly recommends a major upgrade of the NSLS. However, in the short term, in order to provide more insertion device beamtime for users who are working on brightness-hungry experiments, the NSLS has been working on the development of in-vacuum small gap undulators for the past several years. Last year, a proposal to construct the X29 beamline, led by the Albert Einstein College of Medicine, the BNL Biology Department, and the NSLS, was jointly funded by NIH and DOE. This new, dedicated, undulator-based macromolecular crystallography beamline is expected to begin operation in 2004.

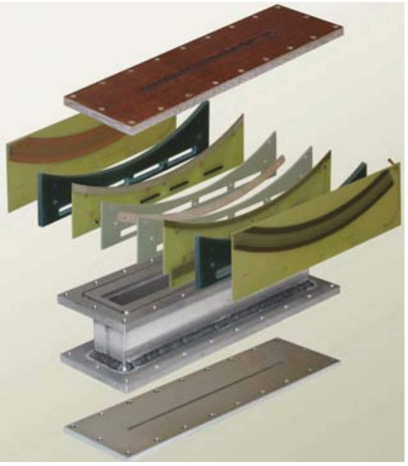


***Figure 1**. The NSLS curved blade detector, exploded view. The thin steel blade in the center is the anode. The other plates are spacers and cathode strip arrays. The cathode strips are read out through individual processing channels and a Digital Signal Processing (DSP) system is used to identify each event location.*

As synchrotron radiation matures and the use of synchrotron radiation spreads to the wider scientific community, we expect that new users of the NSLS increasingly will not be experts in synchrotron radiation techniques, and will need better training and support from the facility. This trend is going to be accelerated by the continued growth in life sciences, and the anticipated growth in the nanoscience user community. In response to this changing need, we have initiated the development of a new user access policy at the NSLS to ensure fair and open access to beamtime and provide better user support. This new policy, which is being developed in close consultation with the NSLS user community and other DOE synchrotron facilities, is expected to be in place by the summer of 2003. In parallel with this new user access policy, a long-term beamline operation and staffing plan is being developed. As a first installment, the department has added or reassigned a total of ten staff to the User Science Division in the last year.

Finally, we have taken the following more immediate steps to enhance user science at NSLS. First, a short course on EXAFS data collection and analysis was held with the help of many experienced users. The format of the workshop mimicked the very successful short course on “Rapid Data Collection and Structure Solving at the NSLS,” which is organized by the NSLS and BNL Biology Departments for macromolecular crystallography. Feedback on the EXAFS short course has been so extremely positive that it will become an annual event, and more short courses will be organized. Second, a faculty-student research support program was re-instituted. This program provides funding for first time users, and/or new faculty members, to perform initial experiments. Third, the NSLS homepage now features exciting, new science highlight articles weekly that focus on recent publications by NSLS users and staff. Fourth, the NSLS website has been redesigned to incorporate a new section aimed at guiding new users through the process of choosing the proper technique, applying for beamtime, and accessing the NSLS for their experiment.

User Administration Report

Mary Anne Corwin

User Administration Head

User Statistics

In the twenty years since the commissioning of the NSLS, its user community continues to be strong and the largest in the BES complex. During the past year, 2413 badged users performed experiments onsite, 30% of them (719 users) for the first time.

For users who conducted experiments at the NSLS facility during the year, 34% indicated their primary field of research as materials sciences, 39% as biological/life sciences, 8% chemical sciences, 8% geosciences and ecology, 5% applied science and engineering, and 6% optical/nuclear/general physics. While changes in the number of users in a particular field of science are generally insignificant from year to year, there was a moderate increase in the number of geosciences and ecology users from last year. A moderate decrease was noticed in the fields of high energy physics and nuclear physics, and the number of biology/life sciences users decreased slightly.

While the number of users in particular fields of science is indicative of *who* is using the facility, it does not show *how* the facility is used. Since 1999, the NSLS has reported statistical data based on actual usage of the facility. This information is extracted from beam time used by each of the 1103 experiments performed in FY02. This year, more than 44% of the beam time used was utilized by experiments conducted in the field of materials sciences. The remaining beam time was utilized by optical/nuclear/general physics (19%), biological and life sciences (15%), chemical sciences (9%), geosciences/ecology (8%), applied science and engineering (3%) and other fields of research (2%). The primary difference in the number of users versus the amount of beam time utilized for a given field of research is explained by the fact that materials sciences experiments utilize more beam time than other experiments. In addition, there are generally a lower number of experimenters per experiment, especially in comparison to biological/life sciences experiments.

We can also determine the primary source of user funding based on beam time usage at the facility. During this year, experiments funded by the Department of Energy's Office of Science's Basic Energy Sciences (DOE/BES) utilized 34% of the facility's beam time. Other programs in the DOE complex utilized an additional 6% of the beam time. Facility usage by other *primary sources* of user support included NSF (14%), NIH (10%), and Industry (12%).

Over 65% of our users are affiliated with U.S. and foreign academic institutions. Other affiliations include BNL employees who are facility users (10%), other DOE contractor employees (3%), other federal agencies (5%), industry (7%), and other (10%).

The 2413 users who visited our facility in FY02 are affiliated with 416 unique U.S. and foreign institutions including 255 academic institutions, 69 industrial institutions, 32 federal government agencies, 26 non-governmental laboratories, and 34 other institutions.

Faculty members at universities or colleges, professional staff and scientists at private, national or industrial laboratories account for the greatest population of users (38%), followed by graduate students (33%), postdoctoral research associates (21%), undergraduate students (4%), and retired, self-employed or other (4%).

Organization

As User Administrator, I have several new projects this year including design of an online system to administer proposals, safety approval forms and a beamline-scheduling program. Brian Bindert joined our staff in late 2002 to work on development of this project and several other database application projects to follow. Another new task initiated this year involves user administration and database support for the Center for Functional Nanomaterials (CFN). Nearly all

User Administration staff members will be working steadily over the next few months to ensure that users who wish to perform nanoscience research are registered, badged and trained as quickly and efficiently as their counterparts at the NSLS facility. Liz Flynn's responsibilities will be shared equally among NSLS and CFN tasks. Her replacement on the registration desk is anticipated in late winter. Lydia Rogers, the Deputy User Administrator, has been very busy coordinating the Users' Meeting scheduled for May, and Gretchen Cisco is assisting with proposal coordination for the CFN, development of the online proposal system, and is very busy keeping up with policy and procedural modifications related to the proposal program.

Security Compliance

As of the end of FY02, the NSLS was instructed to strictly adhere to Department of Energy Notice 142.1 "Unclassified Foreign Visits and Assignments." As a result, our users were notified of new site access policies and requirements. Though the NSLS had hoped to see little disruption in our programs and use of the facility, we have found that several users encountered problems accessing the site. Complications resulted primarily from inappropriate visa and INS documentation and the user's failure to secure visit approval prior to arrival at BNL. For more information regarding site access policies and procedures, please visit the NSLS website.

DIAL 344-USER FOR NSLS USER ADMINISTRATION

In our goal to facilitate user access to the National Synchrotron Light Source at Brookhaven National Laboratory, you may now dial (631) 344-USER to reach the NSLS User Administration Office, or simply Ext. **USER** while onsite. The old number (631) 344-7976 will remain in service as a rollover number. If you have questions regarding facility access, registration, user training, or proposals, please phone our office at (631) 344-USER and we'll be happy to assist you.

Safety Report

Bob Casey

Associate Chair for ESH/Q

Organization and Mission

The Environment, Safety, Health, and Quality (ESH/Q) Division is headed by William (Bob) Casey. The ESH/Q Division consists of 6 full time personnel who are assigned to develop and manage the NSLS ESH/Q program. The department also receives important support from members of the BNL Radiological Control and Environmental Services Divisions.

This division strives to work with all department staff and users to ensure safe and productive work within our facility. Working safely is one of the highest priorities within the Laboratory, and the ESH/Q mission at the NSLS is to help make that happen. ESH/Q staff are heavily involved with the daily work of the department through activities such as: experimental safety review, work planning, training, interlock tests, radiation and industrial hygiene monitoring, design review, chemical/waste handling and storage, inspections, audits and assessments, emergency planning, and accident and injury investigation.

2002 Activities

It is disappointing to report that the NSLS had inconsistent safety performance during FY 2002. While a number of important performance indicators remained excellent (e.g. the total radiation exposure was low, the number of injuries was low, the amount of hazardous waste generated continued to decrease, and no noteworthy spills occurred), we had a significant increase in the number of incidents on the experimental floor that required investigation and follow-up. No one was hurt in these incidents, however each event involved a failure to adhere to one or more important requirements that users and staff are expected to follow while working on the experimental floor. A considerable amount of time was devoted to each incident, as well as examination of the implications to the overall safety program at the NSLS. It is worthwhile to briefly review each incident.

Incident # 1 – A first time user arrived at the NSLS with a number of small samples of biological material. One of these materials had been designated by the Center for Disease Control as a biological "Select Agent", which invokes a special set of requirements, potentially involving a lengthy review and approval process external to BNL. Failure to comply with these requirements also carries the potential for very significant fines by the federal government. While this incident was resolved within a few hours to everyone's satisfaction, handling of the issue rose to the level of the Laboratory's Deputy Director for Operations and the Lab's Chief Counsel. Failures by the user to submit his Safety Approval Form in a timely manner, and to confirm approval to bring the materials to the NSLS, were the principal underlying factors.

Incident # 2 – A new beamline was being commissioned by an experienced beamline group. Difficulties with a monochromator required the beamline to be opened and a number of adjustments to be made. In reconfiguring the beamline prior to resumption of commissioning, a lead glass cover, required to control radiation levels through a view port, was not replaced. Although no significant radiation exposure occurred before the omission was corrected, the failure to maintain a required shield on the view port during operation was a significant violation of NSLS requirements.

Incident # 3 – A postdoc serving as a local contact for one of the beamlines received an electrical shock when he touched an exposed surface energized by a 1000 volt, 100 mA dc power supply. He had made modifications to his equipment in an effort to address a problem created by a failed thermocouple. Inadequate planning and analysis of the changes resulted in him unknowingly working with surfaces exposed at 1000 V.

Incident # 4 – Two general users arrived at a beamline on a Sunday morning and found the hutch posted as a "Radioactive Material Area – Radiation Work Permit Required." The users disregarded the sign since it was posted for a previous experiment and did not apply to their work. Although no conditions existed within the hutch warranting a permit, their failure to comply with an existing radiological posting until removed by authorized personnel was cited by the BNL Price Anderson Act Coordinator as a non-compliance with BNL and DOE radiological requirements.

Incident # 5 - Four general users arrived at a beamline on a Sunday morning without a radiation dosimetry badge. A fifth user from the same group reported to the beamline wearing a radiation badge assigned to another worker. The failure of the group to wear proper dosimetry resulted in cancellation of the experiment and the revocation of their research privileges at the NSLS for 90 days. This incident was also cited by the BNL Price Anderson Act Coordinator as a non-compliance with BNL and DOE radiological requirements.

In each case, these incidents involved a lack of compliance with established safety requirements and created the obvious concern that our safety program and culture needs attention. An extended critique conducted over the past several months with users and staff has identified the following areas for improvement in 2003:

- Greater emphasis on compliance and increased overall rigor in safety expectations
- Increased support and oversight of the general users at the beamlines
- Improved definition of roles and safety responsi bilities of users and beamline personnel
- Increased safety training for all beamline staff
- Improved work planning for routine duties at the beamlines
- Improved NSLS facility specific training

Everyone will hear more about these issues during FY 2003. Improvement in our performance is needed, and the efforts and involvement of our users will be vital to this effort.

Key ESH Requirements Applicable to working at the NSLS

- Make sure that your Safety Approval Forms (SAFs) are submitted in a timely and complete manner (at least 1 week prior to planned arrival - some types of experiments will require much greater lead time) and confirm that the SAF has been approved before traveling to the NSLS.

- Ensure that any conditions required in the safety review are understood by all members of the experimental team and are adhered to throughout the experiment.

- Ensure that all required training is completed - keep in mind that there is specific additional training required if you generate hazardous wastes, if you operate a laser, or if you need to operate material handling equipment such as a hoist.

- Minimize the amount of chemicals that are used in your experiment and ensure that they are safely stored at the NSLS and properly transported to and from the NSLS.

- Abide by the following requirements at all times while working on the experimental floor:

    - Do not work on powered electrical systems with exposed parts above 50 V without a formal permit issued by the NSLS Electrical Safety Officer.
    - Always comply with posted requirements for work in radiation or radioactive material areas.
    - Do not alter interlock systems.
    - Do not alter beamline shielding configurations without a permit.
    - Do not discharge hazardous materials (chemicals or oil) to a sink or drain.
    - All hutches must be cleared of personnel and secured prior to introduction of beam.
    - Wear radiation badges at all times while on the floor and adhere to other requirements specified on signs or designated in training or work planning.
    - Store all hazardous and industrial wastes in designated areas and abide by all storage rules for these materials.
    - Call x2222 or 911 (and the NSLS control room, x2550) if you discover any kind of emergency (medical, spills, fire, etc). If no phone is close by, pull a fire alarm box.
    - Leave the building immediately through the nearest safe exit when fire alarms sound and proceed to the front of the building so that we can account for personnel.

Building Administration Report

Gerry Van Derlaske

NSLS Building Manager

On September 28th, 1978, a shovel hit the soft pay dirt of what once was a favorite place to ice skate for many employees and guests at BNL. Each day draws us closer to the 25th anniversary of Donald Stevens' (DOE) and Ari van Steenbergen's (NSLS Project Head) groundbreaking event. While piecing together the activities that took place at the NSLS during 2002, one puts into perspective the vision of the people behind the push to bring the NSLS to fruition. In review, it is evident that NSLS staff and users alike continue to work together, still providing "groundbreaking" events on a regular basis.

Security Concerns:

We are still trying to cope with the trickle down effects relating to the events of September 11, 2001. Tighter security is noticed at the Lab's Main Entrance, as well as increased requirements to access the NSLS Experimental areas. New rules and regulations affect the placement of such items as refuse containers and dumpsters. Packages must not be left unattended, and certain rooms within Bldg. 725 with restricted and limited access have been deemed as PPA's (Property Protection Areas). Many members of the Laboratory staff are working behind the scenes to resolve these issues in the least intrusive way as possible. Your patience and cooperation during these changing times has been appreciated.

Office space:

All users with offices located in Bldg. 510E were transferred to new quarters in Bldg. 535A and 535M. Additional space will become available in 535A upon the final movement of equipment assigned to the Laboratory Personnel Monitoring Group. The second floor of the NSLS has taken on a new appearance with the addition of the Information and Outreach office, headed up by Lisa Miller in room 2-102. NSLS staff residing in this office includes Nancye Wright, Patrice Pages and Steve Giordano. Other familiar faces in new locations at the NSLS include: Al Boerner, 2-112; Nathan Towne, 2-118; and Scott Buda, 2-108; Laura Miller, 2-127A; Erik Johnson, 2-129; and X.J. Wang, 2-131; Ari van Steenbergen, 2-144; Sam Krinsky, (on Sabbatical), 2-143; Paul Montanez, 2-155; Diane Hatton, 2-189. Old friends that are still near to lend a helping hand after many years of dedication to the NSLS, and to the Lab as a whole, include: Manny Thomas, room 2-119, Marty Woodle, Tom Dickinson and Joe Sheehan, who all reside in Bldg. 728. New Facility Support Representatives from the Radiation Control Division include Steve Musolino and Earle Edwards, found in offices 1-177 and 1-174, respectively. Temporary ITD staff assigned to the NSLS is located in office 2-111.

Construction Projects:

The NSLS 2nd floor office addition has seen projected start up dates come and pass, due to a number of complicated issues. Liquid nitrogen lines near the tank farm, on the northwest side of the NSLS, have been relocated in advance of the construction activities.

Major work has been ongoing for the X17 B2/B3 projects, as the two new lead-lined hutches were installed by Calder-Fabcast in the last quarter of 2002. Interlock and utility work continue as the anticipated commissioning dates of the new hutches approach.

On a smaller scale, the Beamline Development and Support Group's Crystal Cutting room, found in the basement of Bldg. 535, has been expanded and now facilitates the use of a new crystal grinder, able to hold better dimensional tolerances and polish larger crystals than equipment previously utilized.

Facility Upgrades:

New flat panel technology has been installed in the main lobby of the NSLS, and both the employee directory and the four informational Beam Status Monitors were fired up this past spring. Revamping the liquid nitrogen fill station near the west roll-up doors helped make the process easier and safer for filling both the large and small dewars. Plant Engineering provided us with over 200 man hours for maintenance painting to some of our most commonly used areas on the experimental floor. They also installed 2 backflow prevention devices on the main domestic water feeds to Bldg. 725, replacing older units that no longer met the stricter DEC and SCDOH standards set by the Department of Environmental Conservation (DEC) and Suffolk County Department of Health. New vending machines, with a slightly varied selection of food products, were installed within the experimental areas.

Winter Shutdown:

Without a doubt, the most highly visible project on the X-ray ring during the 2002 Winter Shutdown was the re-vamping of the X29 front end and sawtooth conversion projects. The redesigned sawtooth and plug door assembly was installed to allow room for the future construction of the new Mini Gap Undulator (MGU) beamline, championed by the Albert Einstein College of Medicine, BNL's Biology Department, and the NSLS. Existing water piping, water manifolds, and electrical wiring were rerouted to accommodate the X29 beamline expansion project, coupled with the installation of the MGU base mounting feet inside the X-ray tunnel. Two new sets of Pick-Up Electrodes (PUE's) for the active interlock system were installed, along with a set of VAT isolation valves, that will facilitate the insertion of the MGU during the Spring, 2003 shutdown.

Simultaneously, a major effort in the x-ray ring during the 2002 winter shutdown was the installation of the a third, all copper, radio frequency (RF) cavity, replacing the original System #4 cavity. The new RF cavity provides superior cooling for more efficient heat transfer, has eliminated water to vacuum joints, and incorporates independent real-time RGA scans. In addition, the improved interior vacuum surfaces provide for higher power capabilities and a better vacuum for enhanced X-ray beam performance. A redesigned input drive loop and vacuum window allow for a more reliable, higher power operation. Due to the cavity's weight, a complete stress analysis was performed and approved by the BNL Critical Lift Safety Review Committee, while necessary fixtures were installed without impact to the RF cavity installation schedule. This cavity is one of a straight section pair of new cavities, each capable of being isolated with the abovementioned VAT isolation valves, allowing for the future insertion of the X29 MGU.

Beam Position Monitor (BPM) wiring was pulled inside the X-ray ring and run to equipment racks located at strategic points around the perimeter of the X-ray experimental floor. Electric solenoids controlling air cylinders within the booster to the VUV transport tunnel were removed and placed outside of the tunnel for ease of service in case of mechanical or electrical failure.

In conclusion, it can be seen that numerous activities and programs continue to thrive, as the pulse of the NSLS is alive and well. We continue to do our best to provide staff and users with the services one would expect from a world-class facility.

Facility Facts and Figures

Facility Facts & Figures

The National Synchrotron Light Source (NSLS) is a national user research facility funded by the U.S. Department of Energy's Office of Basic Energy Science. The NSLS operates two electron storage rings: an X-Ray ring (2.8 GeV, 300 mA) and a Vacuum UltraViolet (VUV) ring (800 meV, 1.0 A) which provide intense light spanning the electromagnetic spectrum from the infrared through x-rays. The properties of this light, and the specially designed experimental stations, called beamlines, allow scientists in many fields of research to perform experiments not otherwise possible at their own laboratories.

Over 2500 scientists representing more than 400 institutions, over 50 of them corporations, come to Brookhaven National Laboratory annually to conduct research at the NSLS. The facility operates 7 days a week, 24 hours a day, throughout the year, except during periods of maintenance and studies.

As a national user facility, the NSLS does not charge for its beamtime, providing that the research results are published in the open literature. Proprietary research is conducted on a full cost recovery basis.

There are two ways to obtain beamtime at the NSLS: either as a General User or as a member of a Participating Research Team (PRT). General Users are independent investigators interested in using the NSLS for their research. Access is gained through a peer-reviewed proposal system. All operational beamlines at the NSLS reserve at least 25% of their available beamtime for General Users. PRTs are groups of researchers with related interests from one or more institutions. Membership in a PRT is open to all members of the scientific community who can contribute significantly to the program of the PRT (i.e., funding, contribution of equipment, scientific program, design and engineering, operations manpower, etc.).

The NSLS currently has 56 X-Ray and 23 VUV operational beamlines for performing a wide range of experiments. The following pages list the operational beamlines at the NSLS and their unique characteristics.

Beamline Guide

Technique	Description
ARPES	UV photoelectron spectroscopy, angle-resolved
DAFS	X-ray diffraction anomalous fine structure
DEI	Diffraction-enhanced imaging
EXAFS	X-ray absorption spectroscopy, extended fine structure
HARMST	High aspect ratio microsystems technology
IRMS	Infrared microspectroscopy
MAD	Multi-wavelength anomalous dispersion
NEXAFS	X-ray absorption spectroscopy, near edge x-ray absorption
SAXS	Small angle x-ray scattering
SPARPES	UV photoelectron spectroscopy, spin- and angle resolved

Technique	Description
STXM	Scanning transmission x-ray microscopy
UPS	UV photoelectron spectroscopy
UV-CD	Ultraviolet circular dichroism
WAXD	Wide-angle x-ray diffraction
WAXS	Wide-angle x-ray scattering
XAS	X-ray absorption spectroscopy
XMCD	X-ray magnetic circular dichroism
XPS	X-ray photoelectron spectroscopy
XRD	X-ray diffraction
XSW	X-ray standing waves

Beamline	Source	Type of Research	Energy Range	Organization
U1A	Bend	NEXAFS XAS	20-1500 eV	ExxonMobil Research and Engineering Co.
U2A	Bend	IR spectroscopy IRMS High pressure research	50-4000 cm-1	Carnegie Institute of Washington
U2B	Bend	IR spectroscopy IRMS	50-4000 cm-1	Albert Einstein College of Medicine
U3C	Bend	XPS	50-1000 eV	Bechtel Nevada Lawrence Livermore National Laboratory Los Alamos National Laboratory Sandia National Laboratory
U4A	Bend	UPS	10-300 eV	Boston University Brookhaven National Laboratory North Carolina State University Rutgers University
U4B	Bend	XPS UPS XMCD X-ray fluorescence X-ray scattering, resonant X-ray scattering, magnetic	20-1200 eV	Brookhaven National Laboratory Montana State University Naval Research Laboratory Pohang Institute of Science & Technology
U4IR	Bend	Infrared spectroscopy IRMS	20-4000 cm-1	Brookhaven National Laboratory
U5UA	Insertion Device	UPS ARPES SPARPES Magnetospectroscopy	15-150 eV	Argonne National Laboratory Brookhaven National Laboratory University of Texas
U7A	Bend	XPS NEXAFS	180-1200 eV	Brookhaven National Laboratory Dow Chemical Company National Institute of Standards & Technology Rutgers University Texas A&M University University of Michigan
U7B	Bend	NEXAFS UPS XPS	15-300 eV	Brookhaven National Laboratory
U8A	Bend	ARPES NEXAFS	7-1300 eV	University of California, Riverside
U8B	Bend	NEXAFS ARPES	100-1000 eV	IBM Research Division University of California, Riverside University of Michigan
U9A	Bend	Photon-stim. desorption	White Beam	Brookhaven National Laboratory
U9B	Bend	UV-CD UV fluorescence	2.1-8.9 eV	Brookhaven National Laboratory
U10A	Bend	IR spectroscopy	30-10000 cm-1	Brookhaven National Laboratory
U10B	Bend	IRMS IR spectroscopy	50-4000 cm-1	Brookhaven National Laboratory University of Saskatchewan
U11	Bend	UV photoabsorption UPS UV photoionization	3-30 eV	Brookhaven National Laboratory

Beamline	Source	Type of Research	Energy Range	Organization
U12A	Bend	XAS	100-800 eV	Brookhaven National Laboratory Oak Ridge National Laboratory
U12IR	Bend	IR spectroscopy Far-IR spectroscopy Time-resolved spectroscopy	3-400 cm-1	Brookhaven National Laboratory SUNY Stony Brook University of Florida
U13UA	Insertion Device	White Beam	White Beam	Brookhaven National Laboratory
U13UB	Insertion Device	UPS ARPES	5-30 eV	Boston University Brookhaven National Laboratory
U14A	Bend	XPS UPS	15-300 eV	Brookhaven National Laboratory
U16B	Bend	XPS	50-1000 eV	Brookhaven National Laboratory Rutgers University University of Texas
X1A1	Insertion Device	STXM	250-500 eV	Brookhaven National Laboratory ExxonMobil Research & Engineering Co. SUNY Plattsburgh SUNY Stony Brook University of Texas
X1A2	Insertion Device	STXM	250-1000 eV	SUNY Stony Brook
X1B	Insertion Device	XAS X-ray fluorescence XPS X-ray scattering, coherent	100-1600 eV	Boston University Brookhaven National Laboratory Fritz-Haber-Institut University of Groningen
X2B	Bend	X-ray microtomography	8-35 keV	ExxonMobil Research & Engineering Co.
X3A1	Bend	XRD, single-crystal	19,31 keV	Alfred University Amoco Corporation SUNY Buffalo SUNY Stony Brook
X3A2	Bend	XRD, single-crystal Molecular crystallography XAS SAXS	6-30 keV	Alfred University Amoco Corporation SUNY Buffalo SUNY Stony Brook
X3B1	Bend	XRD, powder EXAFS XAS	6-30 keV	Alfred University Amoco Corporation SUNY Buffalo SUNY Stony Brook
X3B2	Bend	XRD, surface	6-26 keV	Alfred University Amoco Corporation SUNY Buffalo SUNY Stony Brook
X4A	Bend	MAD	3.5-20 keV	Howard Hughes Medical Institute
X4C	Bend	MAD	7-20 keV	Howard Hughes Medical Institute
X5A	Bend	Laser backscattering	140-470 MeV	Brookhaven National Laboratory Forschungszentrum Juelich (KFA) Norfolk State University Ohio University Syracuse University University of Paris

Beamline	Source	Type of Research	Energy Range	Organization
X5A (cont'd)	Bend	Laser backscattering	140-470 MeV	University of Rome II University of South Carolina University of Virginia Virginia Polytechnic Inst. & State University
X6A	Bend	MAD	5-20 keV	Brookhaven National Laboratory
X7A	Bend	XRD, powder	5-45 keV	Brookhaven National Laboratory Chevron Research & Technology Company Ohio State University SUNY Stony Brook University of Birmingham University of California, Santa Barbara University of Pennsylvania
X7B	Bend	WAXS or WAXD XRD, time-resolved XRD, single-crystal	5-21 keV	Brookhaven National Laboratory
X8A	Bend	Metrology	0.26-5.9 keV	Bechtel Nevada Lawrence Livermore National Laboratory Los Alamos National Laboratory Sandia National Laboratory
X8C	Bend	MAD	5-19 keV	Brookhaven National Laboratory Hoffmann-La Roche Los Alamos National Laboratory National Research Council of Canada University of California, Los Angeles
X9A	Bend	MAD	5-15 keV	Albert Einstein College of Medicine Rockefeller University Sloan-Kettering Institute for Cancer Research
X9B	Bend	MAD XAS EXAFS NEXAFS	5-15 keV	Albert Einstein College of Medicine National Institutes of Health
X10A	Bend	SAXS XRD, time-resolved XRD, powder WAXS or WAXD X-ray reflectivity	7-15 keV	ExxonMobil Research & Engineering Co.
X10B	Bend	XRD, powder XRD, surface WAXS or WAXD X-ray reflectivity	14.2 keV	ExxonMobil Research & Engineering Co.
X10C	Bend	XAS EXAFS NEXAFS	4-24 keV	ExxonMobil Research & Engineering Co.
X11A	Bend	XAS EXAFS NEXAFS DAFS	4.5-35 keV	Brookhaven National Laboratory Hunter College Naval Research Laboratory Naval Surface Warfare Center New Jersey Institute of Technology North Carolina State University Pacific Northwest National Laboratory Paul Scherrer Institute U.S. Environmental Protection Agency University of Connecticut

Beamline	Source	Type of Research	Energy Range	Organization
X11B	Bend	EXAFS	3.5-25 keV	Brookhaven National Laboratory Naval Research Laboratory Naval Surface Warfare Center New Jersey Institute of Technology North Carolina State University Paul Scherrer Institute U.S. Environmental Protection Agency University of Connecticut
X12A	Bend	Optics R&D	5.6-40 keV	Brookhaven National Laboratory
X12B	Bend	MAD	5-20 keV	Brookhaven National Laboratory
X12C	Bend	MAD	7.5-13.5 keV	Brookhaven National Laboratory
X13A	Insertion Device	X-ray scattering, resonant Magnetospectroscopy XMCD	250-1800 eV	Brookhaven National Laboratory
X13B	Insertion Device	X-ray microdiffraction	5-15 keV	Brookhaven National Laboratory
X14A	Bend	XRD, single-crystal	5-26 keV	Oak Rodge National Laboratory University of Tennessee
X14B	Bend	HARMST X-ray lithography	White Beam	Brookhaven National Laboratory
X15A	Bend	DEI XSW	8-45 keV	Argonne National Laboratory Brookhaven National Laboratory European Synchrotron Radiation Facility Illinois Institute of Technology North Carolina State University Northwestern University University of North Carolina
X15B	Bend	XAS EXAFS NEXAFS	1.5-15 keV	Lucent Technologies, Inc.
X16A	Bend	XRD, surface	4-12 keV	Lucent Technologies, Inc. University of Illinois, Chicago
X16B	Bend	XRD, powder XRD, surface	7.85 keV	Lucent Technologies, Inc.
X16C	Bend	XAS XRD, powder	4.5-25 keV	Lucent Technologies, Inc. University of Illinois, Chicago
X17B1	Insertion Device	XRD, powder	20-100 keV	Brookhaven National Laboratory Carnegie Institute of Washington SUNY Stony Brook
X17C	Insertion Device	XRD, powder XRD, single-crystal High pressure research	5-80 keV	Carnegie Institute of Washington Lawrence Livermore National Laboratory Naval Research Laboratory University of Chicago
X18A	Bend	WAXS or WAXD XRD, single-crystal XRD, surface XRD, powder X-ray reflectivity SAXS	4-19 keV	Brookhaven National Laboratory Pennsylvania State University Purdue University University of Maryland University of Missouri
X18B	Bend	XAS	5.6-40 keV	AlliedSignal, Inc.

Beamline	Source	Type of Research	Energy Range	Organization
X18B (cont'd)	Bend	EXAFS NEXAFS	5.6-40 keV	Brookhaven National Laboratory Chevron Research & Technology Company Dow Chemical Company General Electric Institute of Paper Science and Technology North Carolina State University PPG Industries, Inc. Rutgers University University of California, Davis University of Kentucky University of Tennessee UOP
X19A	Bend	XAS EXAFS NEXAFS	2-7.9	Brookhaven National Laboratory Chevron Research & Technology Company Dow Chemical Company General Electric Institute of Paper Science and Technology North Carolina State University Rutgers University University of California, Davis University of Kentucky University of Tennessee UOP
X19C	Bend	X-ray scattering, liquid XRD, surface X-ray reflectivity X-ray topography	6-17 keV	Army Research Laboratory Carnegie Mellon University Dartmouth College Johns Hopkins University National Aeronautical and Space Agency SUNY Stony Brook University of Chicago University of Illinois, Chicago University of Wisconsin
X20A	Bend	XRD, surface X-ray microdiffraction	6.3-12 keV	IBM Research Division University of Toronto
X20B	Bend	XRD, surface	17.4 keV	IBM Research Division University of Toronto
X20C	Bend	XRD, surface XRD, time-resolved	5-11 keV	IBM Research Division University of Toronto
X21	Insertion Device	X-ray inelastic scattering X-ray fluorescence SAXS	5.6-10	Brookhaven National Laboratory
X22A	Bend	XRD, single-crystal XRD, surface XRD, time-resolved X-ray reflectivity WAXS or WAXD X-ray scattering, surface	10,32 keV	Brookhaven National Laboratory Rutgers University University of Maryland
X22B	Bend	X-ray scattering, liquid XRD, surface	6-10 keV	Brookhaven National Laboratory Harvard University Rutgers University
X22C	Bend	XRD, single-crystal XRD, surface X-ray scattering, magnetic X-ray reflectivity	3-11 keV	Brookhaven National Laboratory Rutgers University University of Maryland

Beamline	Source	Type of Research	Energy Range	Organization
X23A2	Bend	XAS EXAFS NEXAFS DAFS XRD, powder	4.7-30 keV	Brookhaven National Laboratory
X23B	Bend	XAS EXAFS NEXAFS XRD, powder	3-10.5 keV	Naval Research Laboratory
X24A	Bend	XSW XPS X-ray fluorescence EXAFS Auger spectroscopy	1.8-5 keV	Brookhaven National Laboratory National Institute of Standards & Technology
X24C	Bend	XPS	6-1800 eV	Naval Research Laboratory
X25	Insertion Device	MAD	3-30 keV	Brookhaven National Laboratory
X26A	Bend	X-ray microprobe X-ray microdiffraction	3-30 keV	Brookhaven National Laboratory University of Chicago University of Georgia
X26C	Bend	MAD	5-20 keV	Brookhaven National Laboratory Cold Spring Harbor Laboratory SUNY Stony Brook
X27A	Bend	X-ray microtomography	8-40 keV	Brookhaven National Laboratory
X27B	Bend	HARMST	White Beam	Brookhaven National Laboratory
X27C	Bend	SAXS WAXS or WAXD XRD, time-resolved	10 keV	Basell USA, Inc. (formerly Montell) Honeywell International National Institute of Standards & Technology National Institutes of Health Naval Surface Warfare Center New Jersey Institute of Technology SUNY Stony Brook U.S. Air Force
X28C	Bend	X-ray footprinting	White Beam	Albert Einstein College of Medicine

NSLS Linac Parameters as of December 2002

Injection Energy	100 KeV
Final Energy	120 MeV
Number of Sections	3
Number of Klystrons	3
Frequency	2856 MHz

NSLS Booster Parameters*

Injection Energy	120 MeV
Extraction Energy	750 MeV
Circumference	28.35 m
Number of Superperiods	4
Dipole Bending Radius	1.91 m
Nominal Horizontal Tune	2.42
Nominal Vertical Tune	1.37
Maximum Horizontal Beta	8.63 m
Minimum Horizontal Beta	1.01 m
Maximum Vertical Beta	5.26 m
Minimum Vertical Beta	1.73 m
Maximum Dispersion	1.21 m
Minimum Dispersion	0.41 m
Momentum Compaction	0.106
Peak RF Voltage	25 KV
RF Frequency	52.88 MHz
Horizontal Acceptance	1.66E-04 m-rad
Vertical Acceptance	6.11E-05 m-rad
Momentum Acceptance	±0.0025

Booster Magnetic Elements (Fields at 750 MeV)*

Name	Type	Quantity	B (kG)	B′ (kG/m)	B″ (kG/m)	Effective Length (m)
BB	Dipole	8	13.099	-7.97	-125	1.5
Q1	Quadrupole	4		68.82		0.3
Q2	Quadrupole	4		93.60		0.3
SF	Sextupole	4			1223.7	0.2

*Data from J. Galayda, et al., I.E.E.E. Trans. Nucl. Sci. NS-26 (1979), 3839.

VUV Storage Ring Parameters as of December 2002

Normal Operating Energy	0.808 GeV
Peak Operating Current (multibunch ops.)	1.0 amp (1.06 x 10^{12}e-)
Circumference	51.0 meters
Number of Beam Ports on Dipoles	18
Number of Insertion Devices	2
Maximum Length of Insertion Devices	~ 2.25 meters
$\lambda_c(E_c)$	19.9 Å (622 eV)
$B(\rho)$	1.41 Tesla (1.91 meters)
Electron Orbital Period	170.2 nanoseconds
Damping Times	$\tau_x=\tau_y$= 13 msec; τ_ε = 7 msec
Lifetime @ 200 mA with 52 MHz	360 min
(with 211 MHz Bunch Lengthening)	(590 min)
Lattice Structure (Chasman-Green)	Separated Function, Quad, Doublets
Number of Superperiods	4
Magnet Complement	8 Bending (1.5 meters each) 24 Quadrupole (0.3 meters each) 12 Sextupole (0.2 meters each)
Nominal Tunes (ν_x,ν_y)	3.14, 1.26
Momentum Compaction	0.0235
RF Frequency	52.887 MHz
Radiated Power	20.4 kW/amp of Beam
RF Peak Voltage with 52 MHz (with 211 MHz)	80 kV (20kV)
Design RF Power with 52 MHz (with 211 MHz)	50 kW (10 kW)
Synchrotron Tune (ν_s)	0.0018
Natural Energy Spread (σ_e/E)	5.0 x 10^{-4} , I_b< 20 mA
Bunch Length (2σ)	9.7 cm (I_b < 20 mA)
($2L_{rms}$ with 211 MHz Bunch Lengthening)	(36 cm)
Number of RF Buckets	9
Typical Bunch Mode	7
Horizontal Damped Emittance (ε_x)	160 nm-rad
Vertical Damped Emittance (ε_y)	≥ 0.35 nm-rad (4nm-rad in normal ops.)*
Power per Horizontal Milliradian (@ 1A)	3.2 Watts

Arc Source Parameters

Betatron Function (β_x, β_y)	1.18 to 2.25 m, 10.26 to 14.21 m
Dispersion Function (η_x, η'_x)	0.500 to 0.062 m, 0.743 to 0.093 m
$\alpha_{x,y} = -\beta'_{x,y}/2$	-0.046 to 1.087, 3.18 to -0.96
$\gamma_{x,y} = (1+\alpha^2_{x,y})/\beta_{x,y}$	0.738 to 0.970 m^{-1}, 1.083 to 0.135 m^{-1}
Source Size (σ_x, σ_y)	536 to 568 μm, >60 to >70 μm (170-200 μm in normal ops.)*
Source Divergence (σ'_x, σ'_y)	686 to 373 μrad, 19.5 to 6.9 μrad (55-20 μrad in normal ops.)*

Insertion Device Parameters

Betatron Functionμm (β_x, β_y)	11.1 m, 5.84 m
Source Size (σ_x, σ_y)	1240 μm, >45 μm (220 μm in normal ops.)*
Source Divergence (σ'_x, σ'_y)	112 μrad, >7.7 μrad (22 μrad in normal ops.)*

* ε_y is adjustable

X-Ray Storage Ring Parameters as of December 2002

Normal Operating Energies	2.800 GeV
Maximum Operating Current	280 mA
Lifetime	~20 hours
Circumference	170.1 meters
Number of Beam Ports on Dipoles	30
Number of Insertion Devices	6
Maximum Length of Insertion Devices	< 4.50 meters
$\lambda_c(E_c)$ at 1.36 T	1.75 Å (7.1 keV)
$\lambda_c(E_c)$ at 5.0 T (W)	0.48 Å (26.1 keV)
$B(\rho)$	1.36 Tesla (6.875 meters)
Electron Orbital Period	567.2 nanoseconds
Damping Times	$\tau_x = \tau_y = 4$ msec; $\tau_\varepsilon = 2$ msec
Lattice Structure (Chasman-Green)	Separated Function, Quad Triplets
Number of Superperiods	8
Magnet Complement	16 Bending (2.7 meters each)
	40 Quadrupole (0.45 meters each)
	16 Quadrupole (0.80 meters each)
32 Sextupole	(0.20 meters each)
Nominal Tunes (ν_x,ν_y)	9.8, 5.7
Momentum Compaction	4.10^{-3}
RF Frequency	52.88 MHz
Radiated Power for Bending Magnets	198 kW (0.25A)
RF Peak Voltage	1120 kV
Design RF Power	450 kW
Synchrotron Tune (ν_s)	0.0023
Natural Energy Spread (σ_e/E)	9.2×10^{-4}
Natural Bunch Length (2σ)	8.7 cm
Number of RF Buckets	30
Typical Bunch Mode	25
Horizontal Damped Emittance (ε_x)	7.5×10^{-8} meter-rad
Vertical Damped Emittance (ε_y)	1.5×10^{-10} meter-rad
Power per Horizontal Milliradian (0.25A)	32W

Arc Source Parameters

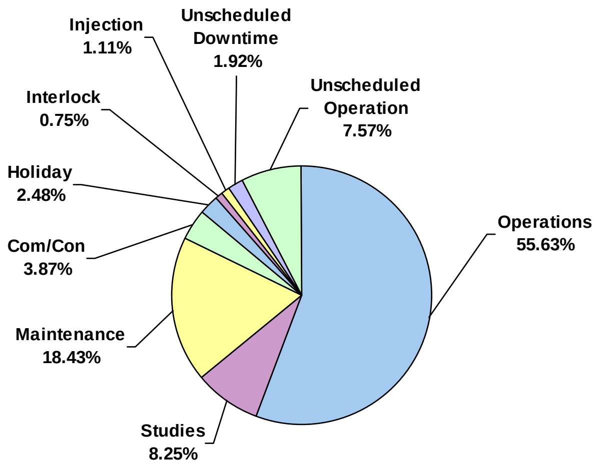
Betatron Function (β_x, β_y)	1.0 to 3.8 m, 7.9 to 26.5 m
Dispersion Function (η_x,η'_x)	0.47 to -0.11, -0.39 to 0.22
$\alpha_{x,y} = -\beta'_{x,y}/2$	-0.49 to 1.62, -3.4 to 4.5
$\gamma_{x,y} = (1 + \alpha^2_{x,y})/\beta_{x,y}$	0.952 to 0.962 m^{-1}, 0.81 to 0.52 m^{-1}
Source Size (σ_x,σ_y)	371 to 612 µm, 27 to 53 µm
Source Divergence (σ_x',σ_y')	476 to 324 µrad, 9 to 7 µrad

Insertion Device Parameters

Betatron Function (β_x, β_y)	1.60 m, 0.35 m
Source Size (σ_x, σ_y)	300 µm, 6 µm
Source Divergence (σ_x', σ_y')	260 µrad, 35 µrad

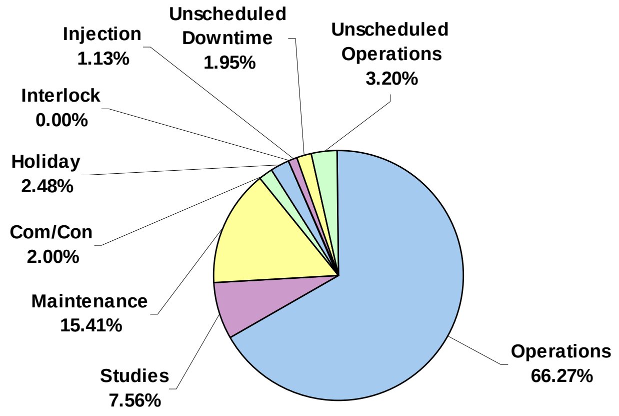
2002 Ring Performance and Usage

X-Ray



The breakdown of the X-Ray Ring usage based on total time (not scheduled time).

VUV



The breakdown of the VUV Ring usage based on total time (not scheduled time).

Month	VUV Actual FY 02			X-Ray Actual FY 02		
	Planned Hours	Reliability[1]	Availability[2]	Planned Hours	Reliability	Availability
October	579	99.9%	105.8%	532	97.6%	109.1%
November	423	93.0%	93.0%	363	98.0%	98.0%
December	0	-	-	0	-	-
January	505	99.2%	99.9%	331	97.4%	118.6%
February	555	99.3%	101.5%	494	94.6%	102.2%
March	624	97.0%	100.1%	583	96.8%	109.8%
April	595	92.7%	97.3%	509	99.2%	121.7%
May	450	99.5%	104.1%	181	82.5%	149.3%
June	591	99.2%	103.2%	535	95.4%	100.8%
July	513	96.6%	102.7%	454	94.0%	103.2%
August	579	99.2%	108.7%	568	99.3%	110.2%
September	594	87.4%	96.3%	540	93.1%	102.6%
	6008			5090		
Fiscal 2002 Delivered	5804	96.6%	101.3%	4887	96.0%	109.1%

[1] Operations during scheduled time
[2] Operations compared to total scheduled time

Publications

The following pages list all publication citations reported to the NSLS in FY 2002 (October 1, 2001 through September 30, 2002). Citations are listed in order of beamline number and then alphabetically by the last name of the first author. This list contains unique citations for journal, published conference proceedings, books, chapters in books, formal reports, informal reports, technical reports, theses, dissertations, and patents when reported. For citation submissions where research was performed on more than one beamline, the citation is listed under each beamline. However, each citation was only counted once. Citations bearing publication dates prior to the Year 2002 are listed only if they had not been previously reported to the NSLS and did not appear in a prior fiscal year's activity report. Diligent effort has been made to ensure that each reference is unique and complete. For journal articles, online searches were performed to locate missing reference information (e.g., year of publication, volume, issue or page numbers). With regard to conference papers, considerable effort was put into ensuring that the citations appeared in published proceedings. Many citations for conferences papers have been omitted from this list if verification could not be made. We apologize to our users and authors for any citations incorrectly omitted.

Several types of journal articles are reported in this list, including premiere journals, peer-reviewed journals and a few that are not peer-reviewed. Premiere journals include: Physical Review Letters, Science, Nature, Cell, EMBO Journal, Nature Structural Biology, Proceedings of the National Academy of Sciences of the United States of America, Structure, and Applied Physics Letters.

In FY 2002, the following types and numbers of publication citations were reported to the NSLS where research was performed in part or in whole at the NSLS.

Journals, peer-reviewed, premiere	134
Journals, other peer-reviewed	437
Journals, non peer-reviewed	22
Total Journals and Magazines	593
Books/Chapters in Books	14
Published Conference Proceedings	79
Reports: Technical, Formal, Informal	3
Theses/Dissertations	19
Patents	0
Total Misc. Publications	115
Total Citations Listed	**708**

NSLS VUV User Publications	69
NSLS X-Ray User Publications	584
NSLS Staff Publications	55
	708

NSLS Users

Beamline U1A

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