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Surface Dynamics Using Infrared Synchrotron Radiation

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The original program at the Light Source utilizing infrared synchrotron radiation was the study of dynamics at surfaces. The field has yielded a surprising amount of unexpected information concerning, among other things, the effects of adsorbates on the modification of the conduction electron density in the near surface region of the substrate. In this article we review what has been learned so far in this field, and discuss what is being planned in the immediate future. We are indebted to our early collaborators in these experiments, Yves Chabal from AT&T Bell Labs/Lucent Technologies and Fritz Hoffmann formerly from Exxon for bringing their expertise to bear on the difficult issues which we faced during the development phase of the beamline.

The original aim of the experiments was to study the bonding vibrational modes of adsorbates on the surfaces of metals and semiconductors which occur in the 5 - 1000 mm wavelength region, (2000-10cm^{-1} or 1.2 -240 meV). The spectrum shown in **Figure 1** summarizes the most important information relevant to the vibrational dynamics. The reflectance change induced by a 1/3 monolayer of CO/Cu(111) at 90K , in the frequency region from 220 - 420 cm^{-1} exhibits, on the one hand an overall decrease, and on the other hand, two sharp resonant absorption and antiabsorption bands. Both the background spectrum for clean Cu, and the adsorbate spectrum were taken in 80 seconds with a reproducibility, as judged by the noise, of 0.01%. Originally, we were expecting to observe only a single dip (at 339 cm^{-1}) on a flat background, due to the adsorbate-substrate mode, which, in itself, and for the short recording time, fully justifies the use of the synchrotron source to study these modes in the far-IR. The peaks were identified using isotopes of CO and the modes are shown in **Figure 1**. This experiment really utilizes the brightness of IR synchrotron radiation since the samples are small single crystals, which, in the case of metals, have to be illuminated at grazing incidence angles (approximately 88°) as shown schematically in the figure, since any field parallel to the surface suffers a phase change upon reflection, resulting in a cancellation of the electric field in the surface plane. Thus the sample throughput is small but with the NSLS we are easily able to measure these small changes.

There is another advantage of IRSR for surface science in addition to the brightness. This is the fact that the source intensity is strictly proportional to the beam current (in the absence of electron orbit shifts), meaning that *absolute* reflectance changes can be measured to high accuracy. We have used this property to quantify the broadband changes in the reflectance of several systems. Even in the well explored region of the intra-molecular modes, the C-O stretch for example, at 2000 cm^{-1}, there are background changes, that were not noticed with conventional sources despite hundreds of measurements. Absolute reflectance changes of ±0.2% can be obtained at beamline U4IR across a broad spectral region from 300 cm^{-1} to 3000 cm^{-1} using a Michelson interferometer with a silicon beamsplitter and a liquid helium cooled Cu doped Ge photoconductive detector, provided the data are all taken within 10 minutes or so. Over longer periods of time, as the current decays in the storage ring, there are slight orbit shifts due to changing thermal loads on the synchrotron, which translate into broadband reflectance lineshape drifts. With continual injection into the storage ring, the so-called top-off mode, in which the stored beam current is maintained at, say 500 mA by topping off at 490 mA, the reproducibility of the signal can be maintained for much longer periods. We have tested this for periods of over an hour.

One of the main advantages of synchrotron radiation over conventional sources is the fact that it puts the signal well above the thermal background. This can be seen in **Figure 2**, in which we have plotted the brightness of a 1200K thermal source, the 300K background and the NSLS. Often the noise is limited by the fact that the detector is seeing "signal" from this thermal background. It is possible sometimes to cool the sample and the optics, but in our experiments we often measure samples at or even above room temperature. In this case synchrotron radiation, being 100 to 1000 times brighter, puts the signal well above this background.

The initial experiments and observations at U4IR led to a large theoretical and experimental program to try to understand these phenomena, and particularly the broadband background reflectivity change. Bo Persson has been leading the theoretical effort. It turns out that in many cases there are unusual dynamics in which the Born-Oppenheimer rules break down, meaning that the electrons do not follow the core vibrations. Under these circumstances adsorbate atoms or molecules, with their own vibrational frequency parallel to the surface, play the role of impurities sitting at the surface, breaking the translation symmetry, and introducing an impurity potential, from which the conduction electrons can scatter without conserving the parallel component of their momentum. The reflectivity change across a broad spectral range then arises from the inelastic scattering of the substrate electrons originating in the skin depth region - the near surface region where the conduction electrons feel the incoming oscillating field. The strength of the reflectance change depends on the ratio of the skin depth and the electron mean free path of the metal. This ratio is ~1/10 for Cu at low temperatures, providing the clearly observable reflectance change seen in **Figure 1**. When the frequency of the light coincides with the frequency of the parallel motion of the adsorbate, there is no inelastic scattering, and the reflectivity returns to the original value for the bare substrate. Persson's theory describes the dynamics theoretically in terms of a friction force between the adsorbate and the substrate as illustrated in **Figure 3**. This explains both the broadband change as a loss of IR signal (case w ¹ w_0) by a friction force between an adsorbate and the conduction electron sea moving at different velocities, and the antiabsorption peak as the absence of the friction force when $w = w_0$.

One of the controversial claims of the theory is the connection between the friction term and the lifetime of the parallel vibrational mode of the adsorbate, due to excitation of electron hole pairs. However, the scattering theory can be more rigorously tested and, for example, it predicts a linear relationship between the DC resistivity change and the broadband IR change. This has been verified recently, by simultaneously recording the resistance change and the IR spectra upon adsorption on the same Cu(111) thin film. These phenomena are important since if the DC conductivity of a thin film is determined by surface adsorbates, then as interconnects in semiconductor circuits become thinner, this could play a critical technological role.

The experiments are shown schematically in **Figure 4**. The Cu(111) films were grown in a separate chamber, in Duesseldorf, and after being mounted in the U4IR surface science chamber, they were cleaned by ion sputtering. The original thickness could be reached again by *in situ* epitaxial copper deposition. Data for a 50 nanometer thick film are shown in **Figure 5** and clearly demonstrate the predicted linearity between resistance and reflectivity changes as the films were exposed to CO gas in the vacuum chamber. Several films with different thicknesses, and different adsorbates (oxygen, and copper) were measured. The linear relationship was found to hold in all cases.

Although this article has concentrated on one system, Cu(111), experiments have been carried out on several Cu, Au, Ni, Pt and Ag surfaces with many adsorbates including H, C_{60}, NO and formate. In one series of experiments vibrational modes for isotopic mixtures were measured to try to understand intermolecular dipole-dipole interactions. The theoretical model has stood up well but many more experiments on these and other systems as a function of temperature are required to understand these phenomena more thoroughly and finesse the theory.

We are extremely grateful for the skilled technical assistance of Dennis Carlson, Gary Nintzel and Tony Lenhard and for the many discussions with our colleagues, Larry Carr, Dieter Moeller, Bo Persson, Roger Tobin, Yves Chabal and Fritz Hoffmann.

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Introduction by the Chairman

Michael Hart

NSLS

The report of the BESAC Synchrotron Light Source Working group, chaired by Dr. R. Birgeneau, was endorsed by BESAC in October. Historic statements about the achievements of the last decade or two and the prognosis for the future included:

"The most straightforward and most important conclusion of this study is that over the past 20 years in the United States synchrotron radiation research has evolved from an esoteric endeavor practiced by a small number of scientists primarily from the fields of solid state physics and surface science to a mainstream activity which provides essential information in the materials and chemical sciences, the life sciences, molecular environmental science, the geosciences, nascent technology and defense-related research among other fields. The user community at U.S. synchrotron facilities continues to grow exponentially, having reached more than 4000 on-site users annually in FY97. The research carried out at the four D.O.E. synchrotron sources is both very broad and often exceptionally deep. It is self-evident that research which requires very high brightness will be carried out overwhelmingly at the third generation sources. . . . Nevertheless, most current synchrotron research requires high flux as opposed to high brightness and therefore can be carried out equally well at second and third generation sources.

The panel was very impressed by the outstanding performance of the second generation facilities (SSRL and NSLS), by the number of users they serve well, by their ability to renew and improve themselves, by their ability to continue cutting-edge research even though the storage rings themselves are not the most advanced, by their commitment to education, and by their abilities to engage new users and address new problems. Given the outstanding track record and clear vision demonstrated by these facilities, the panel expects these facilities to continue to thrive scientifically in a cost-effective manner. These centers are national resources and they should be adequately funded, upgraded and modernized in a timely fashion to serve better the national needs."

The path to at least another decade of outstanding research at NSLS was clearly laid out with appropriate funding recommendations. In particular, our "Phase 3" proposals were endorsed with strong encouragement to seek partnerships between funding agencies. In anticipation of these conclusions I mentioned in the July 1997 NSLS Newsletter that I would be setting up a Task Force to map our strategy and then call for investment and potential PRT members to build and operate narrow gap undulator (IVUN) beamlines. That Task force has been set up, with the following charge:

There are two straight sections in which In Vacuum UNdulators (IVUN) or other short insertion devices could be installed in the X-Ray storage ring: the X9 and X29 straights. The characteristics of these insertion devices have been established through the NSLS R&D program on X13. Although the time is ripe to exploit these new sources in x-ray experiments, within present DOE budget predictions it would be, at best, a very long time before these two new beamlines might be fully equipped. The Task Force is asked to:

(1) determine at least two scientific subject areas, in priority order, which would benefit most from these opportunities;

(2) outline the required insertion device and beamline specifications;

(3) recommend a plan that would lead to the formation of PRTs to secure the funding required and eventually to build and manage the beamlines;

(4) report by the end of 1997.

The present R&D straight and its associated beamline on X13 may be made available for exploratory research on a collaborative basis.

Dr. J.B. Hastings will chair the Task Force and the following members have agreed to serve on it: Dr. Stephen Burley (Rockefeller University); Dr. G. Slade Cargill (Columbia University); Dr. Mark Chance (Albert Einstein College of Medicine); Dr. Steven Dierker (University of Michigan); Dr. Kenneth Evans-Lutterodt(AT&T Bell Labs. / Lucent Technologies); Dr. Wayne Hendrickson (Howard Hughes Medical Institute); Dr. Chi-Chang Kao (NSLS); Dr. Ho-Kwang Mao (Carnegie Institute of Washington); Dr. Peter Stefan (NSLS).

Comments, suggestions and recommendations would be most welcomed.

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A Message from the U.S. DOE

Patricia M. Dehmer

Associate Director of Energy Research

for the Office of Basic Energy Sciences

As a user of the National Synchrotron Light Source (NSLS) you have been routinely asked to provide a variety of demographic and research information before you conduct your experiments and also provide subsequent feedback regarding your satisfaction with your NSLS experiences. This information is very important. It is needed by NSLS staff to better serve your needs, but much of it is also used by the Office of Basic Energy Sciences (BES) to better manage the NSLS along with our other 16 scientific user facilities and to help justify their annual operating budgets to the Department of Energy, the Office of Management and Budget, the Office of Science and Technology Policy, Congress, and other stakeholders.

Efforts have intensified in recent years to define value as it relates to scientific research, to determine the measures by which value can be quantified, and to assess the results of scientific research by using various tools and metrics. The incentive derives in part from a series of laws and executive orders, including the Government Results and Performance Act (GPRA) of 1993, that focus on performance management. However, in these times of tight Federal budgets for science, it is especially crucial to maintain timely and accurate data on as much of our research activities possible to help make informed funding decisions and to better communicate the worth of our programs to key decision makers and other interested groups.

As part of our efforts to improve BES' information base, a more comprehensive data call was developed for FY 1998 in collaboration with the directors and user coordinators of our 17 user facilities. As a result, we will be asking you to provide some additional demographic information and to complete a user satisfaction survey that includes questions on availability, reliability, dependability, and service provided by the NSLS; the outputs of research from the facility (e.g., papers, patents, students trained, new collaborations formed, etc.); and other impressions such as safety-related issues.

We truly appreciate receiving this information to help us better understand who uses our facilities and their views. It helps us tremendously to illustrate and quantify to others the excitement of a facility such as the NSLS that operates 24 hours a day with many dozens of experiments going on simultaneously, where scientists, technicians, administrators, graduate students, and postdocs from universities, industries and government labs are there all the time, talking to each other, exchanging ideas, and producing fantastic results.

The US Department of Energy Home Page can be accessed via the NSLS Home Page, or directly at http://www.doe.gov/.

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A User's Perspective

Joel D. Brock

Cornell University

UEC Chair

A lot has happened since the last news letter. Brookhaven National Laboratory (BNL) just seems to keep landing in the news. The ongoing search for a new contractor to manage the laboratory, the ongoing environmental remediation effort to combat the chemical waste plumes originating in long closed land-fills and the small amounts of tritium leaking from the spent fuel rod storage pool associated with the High Flux Beam Reactor (HFBR) have all received significant news coverage. On the positive side, in just the past few days, the particle physics program at BNL has received significant news coverage about their new discoveries. There is also a lot of good news about things going on at the NSLS; however, since I have yet to read a story in my home newspaper about the NSLS, I suspect most of you don't hear much of the good news either. Therefore, I thought I'd use this issue's column to fill you in on some of the good news I've heard this summer at various meetings about research being done at the NSLS.

On June 25th and 26th, a panel appointed by the Basic Energy Sciences Advisory Committee (BESAC) and chaired by Professor Robert J. Birgeneau of MIT reviewed the scientific and technical programs of the NSLS. This "BESAC Panel on Synchrotron Radiation Sources and Science" is charged with reviewing all four of the DOE synchrotron facilities (the ALS at Berkeley, the SSRL at Stanford, the APS at Argonne, and the NSLS at BNL) and making recommendations for future funding priorities. The panel was explicitly requested to consider the ramifications of closing one or more of these facilities. Several of the staff and users of the NSLS were called on to discuss the impact synchrotron radiation has had on their particular field of science. It was a very impressive presentation to witness. In the short time available, leading experts from over a dozen distinct scientific fields presented the impact science done at the NSLS has had on their specific field. Although I have spent a considerable amount of time working on the NSLS X-Ray Ring floor, I got an education about the breadth of the work being done at the NSLS. These talks covered the usual areas associated with synchrotrons, materials science, surface/interface physics, magnetism, lithography, tomography, powder diffraction, macromolecular crystallography, infrared sources, imaging, and so on. But several other talks were given, such as the one by Dr. Barbara Illman (USDA, Madison and new UEC member) on the applications of synchrotron radiation to forest science which illustrated the enormous potential of synchrotron based measurements to impact non-traditional synchrotron disciplines. My informal discussions with the BESAC panel members suggested that they were as impressed as I was.

On July 8, the NSLS Proposal Study Panels met to review and rate the newly received General User proposals. A new rating scheme had just been instituted with the goals of expanding the range of scores received and also making the ratings consistent among the different panels. Again, it was very impressive to sit on one of these panels and read the large number of high quality research proposals. One quickly comes to the conclusion that, with very few exceptions, the work being proposed is of extremely high quality and quite diverse.

On August 14, the Users' Executive Committee held its summer Town Meeting. In addition to several technical reports, the BESAC panel desired more direct input from NSLS users and sent Dr. Linda Horton from Oak Ridge National Laboratory as its representative to solicit input from NSLS users. She spoke with users during the Town Meeting and then joined a few of us for dinner. The next morning Dr. Horton spent another hour with the UEC and then toured the experimental floor, stopping to speak with experimenters whenever possible.

On September 8 and 9, the NSLS Science Advisory Committee met to perform tenure reviews of a number of PRTs. Again, the quality of the science presented was uniformly high. This impressive committee consists of senior scientists from all over the world and they were extremely complimentary of the scientific and technical programs of the NSLS.

All of this took place, of course, in the midst of the turmoil associated with Secretary of Energy Federico Peña's decision earlier this spring to terminate the management contract with Associated Universities, Inc. which has managed Brookhaven National Laboratory for the past fifty years. In late June, Secretary Peña responded to a tragedy at the Lab in which a contractor working on a sewer line at a remote location on site was killed by a front end loader; the Secretary called for an immediate stand down of the Laboratory's activities which began on a Friday afternoon lasted until the following Tuesday. Recently, local Congressman Michael Forbes and Senator Alfonse D'Amato have both introduced legislation to shut down immediately and permanently the High Flux Beam Reactor, without waiting for the results of the review process underway. And, the DOE extended the deadline for proposals from potential new contractors a second time.

When these events happened late this summer, a weakness in the UEC's ability to communicate with the user community became apparent. Until now, the UEC has been using BNL computer facilities for its Web page and e-mail services. However, DOE rules prevent government equipment from being used to lobby the government. Consequently, the UEC cannot use this equipment to communicate with the user community when it needs to take a position that supports the NSLS in its dealings with the government. We are in the process of remedying this situation by purchasing a commercial WWW/e-mail account. We hope to be able to provide you with much more information in a more timely fashion very shortly.

In closing, things are going to continue to evolve rapidly this fall. By the time you receive this Newsletter, much more will have happened. The Birgeneau panel is scheduled to present its report to the BESAC in early October. The DOE plans to choose a new contractor later this fall and the new contractor should begin phasing in early next year. All of these things will directly impact the NSLS and its users. The UEC will do its best to keep you informed about all of these events.

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Macromolecular X-Ray Crystallography Detectors Update

Lonny Berman

NSLS Beam Line Support/R&D Section

The March 1995 NSLS Newsletter issue contained a summary of the capabilities of the NSLS macromolecular x-ray crystallography beamlines, which was re-printed in the July 1995 issue of Synchrotron Radiation News. Several major changes have taken place in the macromolecular crystallography environment at NSLS since then:

> (1) New macromolecular crystallography PRTs have formed and others have reconstituted, with the overall addition of two beamlines since 1995.
>
> (2) The beam time access procedures for these beamlines have been consolidated within the NSLS General User Program, which now offers two access pathways for these lines: one-year single project and two-year program proposals.
>
> (3) Documentation for more of these beamlines has been added to the World Wide Web, reachable via the NSLS Web site www.nsls.bnl.gov (information about the NSLS General User Program is also available via this Web site).
>
> (4) Of greatest importance, the area detectors for practically all of these beamlines have been upgraded within the past several months, or are about to be upgraded.

This note will focus upon the detector upgrades. A future article will describe the current capabilities of all the macromolecular crystallography beamlines and, as such, will supersede the March 1995 Newsletter article.

X4A: Operated by the Howard Hughes Medical Institute (HHMI), this beamline has just had installed a Rigaku R-Axis IV automatic imaging plate detector, replacing the manual Fuji imaging plates which required off-line readout. The new detector contains two plates, allowing one plate to be read out (in about 3 minutes) while the other is exposing. This allows more efficient use to be made of the available beam time and simultaneously reduces the necessary labor to run the experiment, because manual exchange, readout, and erasure of imaging plates (with the concomitant book-keeping) are no longer necessary.

X4C: This new beamline, also operated by HHMI, is now undergoing commissioning. Its optics are based on a tunable, sideways-diffracting single crystal monochromator. When operational, this beamline will use an Area Detectors Systems Corp. (ADSC) Quantum-1 single-module, 1Kx1K CCD detector, which has a readout time of less than 10 seconds.

X8C: The macromolecular crystallography program on this beamline was formerly operated by the Argonne Structural Biology Center (SBC). After the departure of the SBC at the end of the 1996 fiscal year, the program was reconstituted under the auspices of Los Alamos National Laboratory (which already operated the non-crystallography program on X8C), the BNL Biology Department, UCLA, the National Research Council of Canada, and Hoffman-La Roche Corp. The detector provided for the reconstituted PRT is the X-Ray Research (MAR) 300 mm diameter automatic imaging plate system, which had been used on the X12C (BNL Biology Department) beamline for several years. This single-plate detector has a full-area readout time of about 3 minutes.

X9B: A macromolecular crystallography program was begun on this beamline during the 1996 fiscal year, under the auspices of the Albert Einstein College of Medicine, using manual Fuji imaging plates. Recently, the National Institutes of Health joined this PRT and provided a MAR 345 mm diameter automatic imaging plate system. This new single-plate detector has a full-area readout time of under 1.5 minutes.

X12B: Earlier this year, the MAR 300 mm diameter imaging plate detector that had been in use on this beamline (operated by the BNL Biology Department) was removed from its base and remounted on the long two-theta arm in the experimental hutch, that had historically been employed for small angle x-ray scattering experiments. This upgrade allows for having a very long detector distance (so that large unit cell crystals could be studied) and, by tilting the two-theta arm, attaining high resolution. An ADSC Quantum-4 four-module CCD detector (1Kx1K per module), funded by the 1996 DOE Basic Energy Sciences Facilities Initiative Program along with some contributions by a few pharmaceutical companies, is on order for X12B and shall be installed in time for the winter 1998 scheduling cycle.

X12C: Last fall, a single-module, 1Kx1K CCD detector, built by a collaboration of Brandeis University and the BNL Biology Department, was installed on this beamline (operated by the BNL Biology Department) as an alternative to the MAR 300 mm diameter imaging plate detector which had been in use on X12C for several years. The bulk of the macromolecular crystallography experiments on X12C have been serviced by the CCD detector since. This fall, a four-module CCD detector will be delivered by the Brandeis / BNL Biology collaboration to X12C for commissioning, and it will be made available for use on X25 as soon as it has proven itself.

X25: A new MAR 345 mm diameter imaging plate system, provided by the NSLS via Facilities Initiative Program funds, has just been installed on this wiggler beam line (operated by the NSLS) to service macromolecular crystallography experiments. This replaces use of two different MAR 300 mm diameter imaging plate systems, loaned during the past few years to X25 by the BNL Biology Department and Cold Spring Harbor Laboratory. The readout time of the new detector (per unit plate area) is 3 times faster than of the old MAR detectors, and it has a finer spatial resolution. In addition, a MAR single-module, 2Kx2K CCD detector, with even finer spatial resolution and a readout time of less than 10 seconds, was also funded by the NSLS via the Facilities Initiative Program, and its delivery is expected shortly. It will be able to substitute on X25 for the MAR imaging plate detector, on the same mounting base, when desired. The use of this CCD detector will not be restricted to macromolecular crystallography. Finally, the Brandeis / BNL Biology four-module CCD detector mentioned above which will be commissioned shortly on X12C, could be available for use on X25 during early 1998.

X26C: Primarily operated in the past with focussed and unfocussed white x-ray beam capabilities, for Laue macromolecular crystallography and other white beam experimental programs, by the University of Chicago Consortium for Advanced Radiation Sources (CARS) and the BNL Applied Science and Biology Departments, this beamline has undergone substantial changes in the past year. A tunable channel-cut crystal monochromator was provided by the NSLS for this beamline, in a new upstream enclosure, giving it a standard monochromatic beam capability; previously, attainment of such a capability on X26C required the user to set up a crystal monochromator within the white beam hutch. Cold Spring Harbor Laboratory and the State University of New York at Stony Brook joined this PRT in the 1997 fiscal year, and began a monochromatic macromolecular crystallography experimental program, based upon a MAR 300 mm diameter imaging plate system provided by Cold Spring Harbor. This system replaces the manual Fuji imaging plates that had been provided by CARS for several years.

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A Record Structure from the NSLS

Sylvie Doublié and Tom Ellenberger

(Harvard Medical School)

Robert M. Sweet (BNL-Biology)

One year ago this fall, multi-wavelength anomalous diffraction (MAD) data were collected from a crystal of selenomethionyl-T7 DNA polymerase at the BNL Biology Department beamline X12C. These data rapidly produced the largest new structure (108,000 Da) to be determined by this novel method, raising the prospect that MAD phasing can succeed in most macromolecular structure determinations, large or small.

The MAD experiment was performed after months of unsuccessful attempts to obtain phases by the conventional method of multiple isomorphous replacement (MIR). The success of an MIR structure determination depends on the selective binding of heavy metals such as mercury or platinum to a limited number of sites in the crystal. Heavy atoms scatter x-rays more strongly than the light atoms they replace, resulting in a perturbation of the diffraction pattern. Metal-induced changes in the intensities of diffracted x-rays can reveal the locations of the metals in the crystal, and this information yields the amplitudes and phases of the x-rays scattered by the heavy atoms.

In a further extension of the isomorphous replacement method, x-rays with energies near the energy levels of electrons in the heavy metals experience large phase and amplitude shifts as they resonate with these electrons, and this "anomalous" scattering provides additional phase information. Unfortunately, in many cases metal binding also shifts the positions of the macromolecules within the crystal lattice, causing large and unpredictable changes in x-ray diffraction. (The crystals are no longer of the "same form", that is "isomorphous" with the original crystal.) In extreme cases the metals may render the crystals severely disordered and useless for diffraction experiments.

Heavy atom soaks were unsuccessful for crystals of the T7 DNA polymerase, so we used a trick developed by Wayne Hendrickson and co-workers at the Columbia University and the Howard Hughes Medical Institute beamline X4 at NSLS. They biosynthetically incorporated selenomethionine into proteins in place of methionine. This was accomplished by overexpressing the protein in bacteria grown in defined media containing selenomethionine. The selenium atom replaces a sulfur in the methionine side chain, and this electron difference is readily detected by x-ray diffraction from crystals of the selenomethionyl-protein. Unlike the larger metals typically used for MIR, these selenium atoms are tolerated well by most proteins. In fact, the selenomethionine imposter is convincing enough to fool the cell's own protein biosynthetic machinery. Selenomethionyl-proteins generally behave like their natural counterparts during protein purification, and crystals of these modified proteins typically grow under the same conditions as crystals of the native protein.

In pioneering studies, Hendrickson and co-workers showed that diffraction data from single crystals of proteins containing selenium or another anomalous scatterer could produce accurate phases for a crystal structure. Their method of multi-wavelength anomalous diffraction (MAD) requires x-ray diffraction measurements at two to four x-ray energies near an atomic absorption edge of the heavy atom, chosen to maximize the real and imaginary components of anomalous scattering. MAD phasing is rapidly becoming the method of choice for determining new crystal structures of small to medium-sized proteins, and MAD has succeeded for a variety of anomalous scatterers including Se, Fe, Cu, Br, Tb, Pt, and Hg. Several of the NSLS beamlines devoted to macromolecular crystallography are equipped with x-ray optics that produce low bandwidth, high intensity x-rays at energies near the K absorption edge of selenium (l = 0.98 Å). This situation is ideal for the accurate measurement of anomalous scattering from weakly-diffracting crystals of selenomethionyl-proteins.

So why was a MAD experiment using the selenomethionyl-T7 DNA polymerase not our first choice for determining the crystal structure? We crystallized a complex of the polymerase bound to its processivity factor *E. coli* thioredoxin, a DNA primer-template, and a nucleoside triphosphate, totaling 108,000 Daltons in the asymmetric unit. This complex is larger than any of the protein crystal structures that had been successfully phased solely on the basis of anomalous scattering from selenium. Moreover, it would be necessary to locate most or all 15 selenium atoms in the crystal asymmetric unit. This is typically accomplished with a Patterson function calculated from the anomalous intensity differences of Friedel mates in the diffraction pattern. Looking at all possible vectors connecting 15 selenium atoms, one would have to sift through 210 interatomic vectors in the difference-Patterson map — a daunting task! Nevertheless, the quality of phase information obtained from other MAD experiments at NSLS beamlines X12C, X4, and X25 suggested to us that a multiwavelength experiment with the selenomethionyl-polymerase complex was appropriate.

We performed a 3-wavelength MAD experiment at beamline X12C, guided by the automatic MAD data collection method developed by J. Skinner and R. Sweet (BNL-Biology). Diffraction data were collected with the hybrid MAR Research imaging-plate/Nonius diffractometer to a resolution of 2.2 Å from a single crystal, cryo-cooled at 100 K. The x-ray data sets were collected at the inflection point of the Se K edge (l_1 = 0.9822 Å), the absorption peak (l_2 = 0.9788 Å), and a high-energy wavelength remote from the edge (l_3 = 0.95A; **Figure 1**). We collected an additional data set from the same crystal using a laboratory x-ray source (l_4 = 1.54 Å) upon returning from NSLS. Phase information was derived from the anomalous differences at the absorption peak, and from dispersive differences involving all pair-wise combinations of data sets. Care was taken in aligning the plate-like crystal in the plane of the cryoloop so that Bijvoet pairs appeared on the same image or adjacent images, maximizing the accuracy of intensity difference measurements. Crystal diffraction quality was assessed in our laboratory at Harvard Medical School prior to transporting the frozen crystals to the NSLS.

The MAD experiment requires the energy-resolution and brilliance of a synchrotron beamline in order to measure small intensity differences arising from anomalous scattering, coupled with an ease and reproducibility of repeated wavelength changes. Beamline X12C clearly met these requirements, producing high quality data of the polymerase complex. However, we were still faced with the problem of finding the 15 selenium atoms in the crystal asymmetric unit. Direct phasing methods implemented in George Sheldrick's program, SHELXS-86, readily solved the problem. All 15 selenium atoms were located from dispersive differences calculated from data collected at the inflection point and at the high energy remote wavelength. The resulting MAD-phased electron density far exceeded our expectations! The experimental electron density map, completely free of model bias, is in many regions indistinguishable from the $2F_o$-F_c map calculated with phases from the final, refined model (**Figure 2**). The MAD experiment not only ended our laborious search for heavy atom derivatives, but it also produced electron density of exceptional quality, expediting model building and refinement of this large complex.

The structure of the T7 DNA polymerase complex provides the first glimpse of a replicative DNA polymerase poised for the incorporation of an incoming nucleotide into a growing DNA strand (**Figure 3**). The structure yields many insights concerning the high fidelity of template-directed DNA synthesis (selecting the correct nucleotide for incorporation), a means for detecting misincorporated bases, and the mechanism of metal-assisted nucleotidyl transfer by this and a large group of related polymerases. An article describing the crystal structure is in press at *Nature* (S. Doublié, S. Tabor, A.M. Long, C.C. Richardson, and T. Ellenberger. "Crystal structure of bacteriophage T7 DNA polymerase complexed to a primer-template, a nucleoside triphosphate, and its processivity factor thioredoxin").

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THEY'RE YOUR HANDS, EYES AND LUNGS!!!!!

W. Thomlinson, Associate Chairman for ES&H

N. Gmür, ES&H Coordinator

What do a bursting dewar, a falling sheet of metal, an exploding acid bottle and a runaway reaction in a hood have in common? There is a simple answer.

Over the past year or so, such incidents have resulted in real and potential harm to employees and users of the NSLS. The one key element in all of them was carelessness on the part of the individuals involved in the experiment or work effort.

The NSLS, from its inception, has put in place many programs designed to make the working environment here passively safe. Some of these are extensive shielding designs, interlocks which are tested routinely, safety approval forms for experimental reviews, training sessions for users and staff, work control procedures, Tier I and II safety assessments and detailed follow-through, and skilled Operations Coordinators ready at all times to help ensure a safe work place. Our record is one that we are very proud of and we are continuing to work to improve our efforts. That is not in response to any external reviews or findings, it is just our way of life.

However, the NSLS cannot control the behavior of each individual 24 hours a day, here or elsewhere. Each member of the community, users and staff, is foremost in control of his/her own safety. It is the responsibility of the individual to be properly trained in the procedures which will be used and to apply good common sense in carrying out all tasks being performed. Following mandated procedures, carefully analyzing hazards, not taking any chances with potentially harmful tasks, and getting help when in doubt about procedures or safety issues is just good practice which we expect, and indeed which any synchrotron facility depends upon to ensure a safe work place.

Clarification of the reportable occurrences over the past year will serve to highlight the essentials of good practice which were not followed. The first example is the recent episode in which an NSLS employee received a serious injury to his thumb while placing additional lead shielding in a hutch. That is a job which we all do and is certainly within our level of skills. However, he made an error in judgment while propping a sheet of metal in an elevated position instead of properly securing it by clamps or other support to ensure that it did not fall. While installing a lead brick, the metal fell and acted like a guillotine, almost severing his thumb. Fortunately, he had followed good safety practice and was wearing gloves during the work, and that certainly saved the thumb from additional injury. This is a case of almost doing it right, but not completely thinking through the potential danger of the unsecured metal sheet. Proper analysis of the risk was not done by the employee and the proper work procedure was therefore not followed. Advice on the use and machining of lead may be obtained from Andrew Ackerman (x5431) or Gerry Van Derlaske (x4926).

A second incident involved the over-pressurization and rupture of a small dewar used in the transport of some biological samples. The samples were put in a metal dewar (actually a commercial coffee thermos) with a screw-on top which was "kept loose" to allow the venting of the CO_2 gas released by the dry ice used as a cryogen. This has apparently been a standard form of transport, with this particular thermos having been transported from Europe in the cabin of a commercial airliner. It was understood by the personnel involved that high pressures could develop if the cap was tightened. However, at some point after arrival at the NSLS, the cap did become sufficiently tightened so that the gas was prevented from escaping and pressure built up inside. As the experimenter and one of the beamline personnel were attempting to open the thermos, the cap ruptured causing minor injuries to them. The potential for serious injury to the eyes of the workers was very real but fortunately did not occur. This is an example of the experimenters using a container which was not designed and approved for use with cryogenic materials. Lack of training, poor judgment, and no careful review of the handling procedures certainly led to this potentially serious event. The NSLS will assure that experimenters follow proper cryogenic safety procedures at the NSLS. Beamline personnel are also being informed of their responsibilities with regard to including cryogenic safety in the BLOSA training. There is no excuse for anyone not being trained in the safe use of cryogens and cryogenic vessels if they in fact are using them. Anyone can take the Cryogenic Safety Course (#OSH-025) given each month. Contact x4151 to register.

Another example of a near miss situation was the incident in which a bottle of acid ruptured in the hazardous waste storage shed. The bottle was not labeled by the users as to its contents, history or ownership. When found by our safety staff, it was transported to the hazardous waste storage area. A few days later, the bottle ruptured inside the shed due to overpressurization, fortunately when no one was there. Acid and glass were spread throughout the storage shed. Had this event occurred when the bottle was being handled by the safety personnel or when someone was accessing the shed, the resulting injuries would have been very serious. Here is a case where good safety practice was in place by the NSLS staff but an event occurred. The Safety Engineer was in fact wearing protective equipment during the handling of the bottle and it was stored in a proper containment building. However, some user (not identified) had violated the mandated procedures for labeling, storing and disposing of hazardous materials, in this case acids (probably a mixture of acids based on the analysis of the event). It is a simple matter to label bottles and there are very clear procedures for disposing of materials at the NSLS. Everyone who works at the NSLS is trained in the proper procedures, and help is always available. A short computer-based Hazardous Waste Generator training course is given by Chris Weilandics (x2593) upon request. This event was a case of a user ignoring good safety practices for which they had been trained.

The final example is an incident in which experimenters were carrying out an etching procedure in a hood. They were untrained in the procedures to be used and the reaction became exothermic. The result was that acid fumes from the uncontrolled reaction filled the hood and were subsequently drawn back into the NSLS from the outside of the building and distributed by the air handling system onto parts of the experimental floor. Although the faulty design of the venting and air handling systems was part of the problem (the system is being redesigned and upgraded), the basic cause was untrained users. The reaction should have been carried out in a water-cooled, temperature controlled apparatus which would have prevented the reaction from running away. Once again, the proper training prior to coming to the NSLS would have prevented this incident.

The message is clear. Safety is everyone's responsibility, not just that of the NSLS staff. Proper training prior to coming to the NSLS is essential for any work that involves potentially hazardous materials, equipment, or processes. Failure to follow the procedures for which individuals have been trained, either at home institutions or at the NSLS, cannot be allowed to happen. The results can be tragic. Remember that they are your fingers, your eyes and your lungs — as well as those of co-workers at the NSLS — which are in danger.

We urge all users and staff to take the time to assure themselves that:

1. They know what they are doing, are trained in the procedures, and plan all work carefully.

2. They carry out all tasks keeping safety as the number one priority.

3. Procedures are diligently followed.

4. Expertise be sought when procedures or safety issues are unclear.

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FOCUS ON.................Accelerator R&D Group

Samuel Krinsky

Accelerator R&D Head

NSLS Deputy Chairman

Synchrotron radiation sources are playing an increasingly important role in basic scientific, technological and environmental research. Innovative accelerator physics has been of key importance to the development of synchrotron radiation facilities. Of particular note is the Chasman-Green lattice pioneered at the NSLS, which has been adopted at many of the third generation facilities. Continued improvements in source brightness, stability and reliability have enabled experimenters to increase their productivity and to carry out ever more challenging research programs. Always looking to the future, the Accelerator R&D Group at the NSLS has spearheaded many advances in the state-of- the-art of storage ring sources, and in the design of insertion devices. In addition, we have worked aggressively to develop the theory of short wavelength free electron lasers, and to carry out innovative proof-of-principle FEL experiments at the BNL Accelerator Test Facility (ATF). While these experiments at the ATF are in the infrared, development of ultraviolet FELs is underway at the Source Development Laboratory (SDL), now under construction.

The first global orbit feedback systems were developed and implemented at the NSLS, by a group led by Li Hua Yu. These feedback systems have provided the NSLS with a level of orbit stability unsurpassed even at the newest facilities. Through improvements in orbit monitoring and feedback algorithms, the stability can be even further increased. Continued development and optimization of the orbit feedback systems has been pursued by Om Singh of the Electrical Engineering Section, who is now leading the development of a digital orbit feedback system.

Machine physics studies on the X-Ray Ring, carried out by James Safranek (now with the B-Factory group at SLAC), resulted in the reduction of the vertical emittance from 2 nm-rad down to the present operational value of 0.1 nm-rad. James has also implemented, in Studies, a new configuration of the X-Ray Ring lattice which reduces the horizontal emittance from its present value of 90 nm-rad down to 45 nm-rad. With continued work, this lower emittance lattice may well become the preferred operational mode of the X-Ray Ring. Another important contribution of James Safranek has been the development of a computer program to determine with high precision the electron optical characteristics of a storage ring lattice, utilizing the orbit response data generated by measuring the orbit displacement at each beam position monitor produced by excitation of each trim magnet. This program has been used at many of the synchrotron radiation facilities around the world, to improve their understanding of their storage ring lattices.

The NSLS has been a leader in the development of advanced insertion devices. R&D in the X13 straight section has led to the successful development of a time-varying elliptically polarized wiggler (with APS and BINP Novosibirsk), and of two small gap undulators. The prototype small gap undulator (PSGU) demonstrated for the first time that long lifetime could be maintained in a storage ring with a vertical electron beam aperture as small as 3 mm. The in-vacuum undulator (IVUN) recently built in collaboration with SPring-8, operated successfully with a magnet gap of 3.2 mm, with only a slight reduction in beam lifetime. George Rakowsky and Lorraine Solomon have made important contributions to the magnetic design and measurement of these devices. Peter Stefan, of the Long Range R&D Group, was project leader for the PSGU and IVUN, and for each of these devices he designed and built difficult vacuum vessels which have performed impressively. The development of small gap, short period undulators will have useful applications at the NSLS and at the third generation facilities, extending the wavelength range achievable by undulators.

We have been actively pursuing FEL sources, and aim to provide our users with access to these powerful new tools. Important theoretical results are the first universal scaling law for FEL gain, and an analytic calculation of the effect of wiggler errors in the exponential gain regime. Key experimental initiatives are our participation in the ATF and the establishment of the SDL. The ATF, headed by Ilan Ben-Zvi, is operated jointly by the NSLS and the BNL Center for Accelerator Physics as a user's facility for accelerator physicists. The ATF program in RF photocathode guns is recognized internationally as cutting-edge R&D, as exemplified by its recent measurement of slice emittance in a 10 ps electron bunch. The SDL, headed by Erik Johnson, was established to pursue the science outlined in the NSLS Deep Ultraviolet Free Electron Laser (DUV-FEL) conceptual design report. The cost of the SDL facility has been minimized by using an existing 210 MeV linac and the 10 m long NISUS wiggler, originally built by STI Optonics for Boeing Aerospace.

One important constituent of the SDL accelerator system is a magnetic bunch compressor, which has been designed by Bill Graves. This will make possible very short, high peak current bunches. Important applications of the bunch compressor are: increase of FEL gain; generation of coherent transition radiation in the infrared; and generation of short pulses of hard x-rays by Compton backscattering. Another possible experiment at the SDL is the introduction of a 2856 MHz superconducting RF system into the small 150 MeV storage ring originally built as part of an x-ray lithography project. The use of such a storage ring system for the coherent generation of far infrared radiation has been studied by Jim Murphy.

Key to our plans is the development of sub-harmonically seeded FELs in which harmonic generation converts a laser seed to much shorter wavelength radiation. Much of the theory of these devices has been developed by Li Hua Yu at the NSLS. A proof-of-principle High-Gain Harmonic-Generation (HGHG) FEL experiment is planned to be carried out at the ATF in the infrared, using an undulator borrowed from Cornell and upgraded at the APS. Self-amplified spontaneous emission (SASE) has already been observed at the ATF at 1 micron and 0.62 micron wavelengths, and further work is planned for this winter. An important advantage of HGHG over SASE is that HGHG will produce a beam with much higher longitudinal coherence. Both the SASE and HGHG work will be extended into the VUV at the SDL . The experiments planned for the ATF and the SDL are milestones, not just for the BNL program, but also for other projects like the proposed Linear Coherent Light Source at SLAC and the Tesla Test Facility FEL at DESY, Hamburg.

See also Chapter 5 in the NSLS Activity Reports.

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The 1997 Annual Users' Meeting

Joel Brock

Cornell University

UEC Chair

The users of the National Synchrotron Light Source (NSLS) held their annual Users' Meeting on May 20, 1997, at Brookhaven National Laboratory (BNL). This meeting serves several functions: celebrating the scientific and technical accomplishments of the previous year, obtaining the latest news on the U.S. Department of Energy's support for scientific facilities in general and the NSLS in particular, and providing an opportunity to visit with old friends and colleagues. As in past years, six workshops on scientific and technical topics (descriptions follow this article) were held the day before and the day after the main meeting.

The meeting began with a lively and humorous key-note address titled "Future Schlock" by Robert Park, Professor of Physics at the University of Maryland and author of the WWW news/opinion page "What's New". The main point of Park's address was that one cannot make accurate predictions about the future. He began by pointing out that one year ago, no one was predicting that today we would have budgets before Congress which both balance the budget by the year 2002 and give science annual funding increases. He then continued with several examples of predictions made by various futurists and concluded with the point that no one predicted that scientists would become politically active or how powerful their voice would be.

The Interim Director of BNL and President of AUI, Lyle Schwartz, was the next to address the meeting. He began his remarks by addressing recent statements in the press by DOE Assistant Secretary Tara O'Toole (see, for example, *Science News*, Vol. 151, p. 284, May 10, 1997) that the highly publicized deficiencies in Environment, Safety and Health at BNL were the fault of users. Dr. Schwartz strongly defended the users. He then solicited their comments and observations during this time of change at BNL.

The U.S. Department of Energy was represented by the Associate Director of Energy Research for the Office of Basic Energy Sciences, Patricia M. Dehmer. In light of the two previous speakers' remarks, she began her remarks by assuring the audience that during all the upheaval associated with BNL's problems there has been one constant: "the high regard" for the NSLS, its users and the quality of their science. She then went on to outline the organizational structure of the DOE, pointing out that the Basic Energy Sciences (BES) budget is roughly equal to that of the National Science Foundation, but that BES funds three (3) times the amount of physical science research and most of the major user facilities in the country. She urged the audience to communicate with BES (BES@oer.doe.gov) answering the question, "How has your discipline been affected by synchrotron radiation and how would it be affected by the lack of it?"

Next, Larry Dubois, director of DARPA/DSO, gave an overview of materials research from a DARPA perspective. Emphasizing the potential applications, he cited in situ studies of fuel cells, studies of "relaxor" piezo-electric materials, and x-ray patterning of materials as examples of areas where synchrotron x-ray techniques might be of interest to DARPA.

The last speaker of the morning was Michael Hart, Chairman of the NSLS. His remarks were focused primarily on the BESAC review panel on synchrotron radiation facilities which would be visiting NSLS on June 25 and 26. Part of the review will be presentations by NSL users. He requested the continued assistance of the user community during this review process.

In dramatic contrast to the morning session, the afternoon was devoted to scientific talks spanning the wavelength spectrum from the far infra-red through the ultra violet into the hard x-ray region. Albert J. Sievers led off, discussing the coherent generation of FIR and describing experiments he has performed using the LINAC at Cornell. In these experiments, he used FIR as a diagnostic probe to measure the profile of the electron bunch. As part of his presentation, he walked the audience through a very clear explanation of how one uses Kramers-Kronig relations to solve the phase problem in time/frequency Fourier transforms.

Robert Bartynski spoke next on his coincidence spectroscopy measurements of TiO_2. The basic idea of the technique is to trigger off of a core level photo-electron and then require the coincidence of a particular Auger electron associated with the death of the core hole. Requiring the coincidence gives the technique both elemental and valence sensitivity and the low background enables one to determine defect densities on the order of 2%. Data illustrating the effects of different surface preparations on the densities of point defects with particular Ti valence states were presented.

Thomas Gog discussed his recent work on Multi-Energy X-ray Holography, presenting reconstructed images of several different systems. Although the technique is still at an early stage of development, the potential to produce atomic resolution real-space images was tantalizing. He discussed several current technical challenges yet to be overcome, including developing a better understanding of the effects of wavelength filtering on the numerical Fourier transform.

The last speaker of the afternoon, Donald Weidner described his work using x-ray diffraction at high pressures and temperatures to study the phase diagram of materials found in the Earth's crust. He related that work to a self-consistent mathematical model which explains an anomaly in the frequency distribution of deep earthquakes.

Two other important features of the Users' Meeting were the scientific poster session, with a reception at the Brookhaven Center, and the equipment exhibit in the Lobby of Berkner Hall. Both were popular with the users, providing an opportunity to get a preview of some of the latest work performed at the NSLS and to see some of the new equipment available from suppliers.

The election of the NSLS UEC occurred during lunch. Paul Stevens, Barbara Illman and John Parise were elected as general members. The SPIG representatives will be elected by e-mail ballot after the meeting. On Wednesday, in executive session, the UEC chose John Parise to be its Vice-Chair (Chair-Elect).

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New Experiment Safety Approval Forms

Andrew Ackerman

NSLS Industrial Hygienist

The procedures for reviewing experiments for environment, safety, and health (ES&H) concerns are themselves under review and undergoing some change. We want to re-organize and better document the review process and are beginning with the revision of the NSLS Experiment Safety Approval Form. Some of the changes are prompted by new reporting requirements from the Department of Energy, but most are a result of our desire to assure adequate work planning and to provide a mechanism for our users to better report their activities at the beamlines. The changes being made will enhance the dialogue between users and ES&H Staff and allow us to better document noteworthy aspects of the review process. The old Safety Approval Form has served well; the new version is better.

What's NOT changing: We are not revising our overall approach to experimental review and controlling risks on the floor. We will continue to depend on an open dialogue between our users and the ES&H Staff to identify, evaluate, and control potential hazards.

What IS changing: We are expanding written analysis of experiments, collecting new information about funding sources and the subject of your research, and recording the beam time devoted to your work. The biggest change is inclusion of a section entitled, "Task and Hazard Analysis". We want our users to provide an outline of tasks they plan at the beamline, to think about potential hazards associated with those tasks, and describe what measures are needed to control any risks presented. We want to show that our experiments are well planned. Most experiments will require only a single paragraph to outline their plan, perhaps less than 200 words. Those that present significant ES&H concerns will require more detail. That is good, as it will mean more thought will be directed to potential hazards and those hazards will be better controlled.

The deadline for implementing the new Safety Approval Form and procedures has been set at October 1, 1997. Any beam time occurring on or after October 1 will require the new Form; more details as well as Word and PostScript versions of the form can be found on the NSLS Home Page www.nsls.bnl.gov, under the "News" section. The plan is to move towards electronic Web submission of this information and we expect to have that operational by January 1998. Until that time, we need to proceed with the new paper form. We are counting on the Operations Coordinators for support to the users and I am expecting people to call for help or clarification as we begin these changes. People to contact for assistance are listed on the last page of the new form.

Experience has shown that the people working at the Light Source do so with care and attention to detail. Our experiments are well planned and ES&H review is successful. We expect that to continue and we hope that you will help make the transition to the new documentation as easy as possible.

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X-Ray Ring Update

Roger Klaffky

X-Ray Ring Manager

Beamline personnel continued to use dedicated studies shifts to survey the effectiveness of additional lead shielding for 2.8 GeV operation. To expedite this process, beamlines having sufficiently low radiation levels were put on a list to operate in a "self-monitoring" mode. In this mode users are able to immediately check newly-installed shielding and make required modifications. During the 2.8 GeV operations/survey week of September 23-29, the X-Ray Ring operated reliably with 230 mA fills. By the end of the week there were 34 beamlines operating in either the self-monitoring mode or in an "approved" mode after a final survey indicated that all beamline components had levels less than 500 cpm on a Ludlum pancake Geiger counter. Self-monitoring lines are expected to continue their shielding efforts to satisfy the 500 cpm requirement.

Substantial improvements were made on the X17 cryogenics. The two helium supply and return transfer lines were totally rebuilt, thereby eliminating an erratic heat leak. Another major problem was solved when a metal chip was removed from the autofill valve seat to the magnet cryostat. After these repairs the system has been running smoothly.

Following the installation of the IVUN (In-Vacuum UNdulator) in the X13 straight section during the May 1997 shutdown there was a commissioning period. Initial conditioning was carried out at a gap of 10 mm. After 10 amp-hr of conditioning the pressure at 330 mA was 7.1 nTorr which further decreased to 1 nTorr after 230 amp-hr. During studies periods, IVUN operated with magnet gaps between 10 mm and 3.2 mm. At a gap of 3.2 mm the ring lifetime decreased to about 12.7 hr, corresponding to a partial lifetime contribution of over 100 hours. The observed photon spectrum from the IVUN was measured in the X13 hutch using a single crystal spectrometer. The agreement between the observed and theoretical spectrum was good. The brightness at the peak of the 4.6 keV fundamental was $3x10^{17}$ photons/sec/0.25amp/mm^2/mrad^2 /0.1% bandwidth.

The programmable boards for the analog global feedback system were received. Initial studies have been conducted on the low emittance lattice with the low emittance lattice response matrices successfully downloaded onto these boards. Studies of the new lattice will continue inorder to check orbit stability and to increase the fill current to 350 mA.

The December 1997 shutdown will be shorter than previous winter shutdowns because a decision was made by NSLS management to install both the new superconducting X17 wiggler and the new rf cavity during the April 1998 shutdown. There are several major tasks for the December 1997 shutdown. The remaining thirteen upgraded beamline beryllium windows required for high current operations will be installed. Also, new safety shutters rated for operation up to 500 mA at 2.584 GeV will be installed in the X21 and X25 frontends. There will be a number of improvements to the NSLS water systems. A primary aluminum water system heat exchanger which is now leaking will be removed and replaced with a new unit. A plugged high-pressure copper heat exchanger will be disassembled and cleaned. An aluminum water system control valve will be rebuilt and the three deionizer systems will be relocated to make them more accessible. The X-Ray Ring RIA and RIB interlock chains will be physically separated in new conduits running around the ring. The X14A beamline interlock system will be upgraded to a Phase II system and a new X14B beamline system will be installed. Work on upgrading the interlock system on critical ring component water cooling will continue with the installation of Klixon temperature sensors at the output of existing Proteus flow meters. The Klixons will dump x-ray beam if the cooling water temperatures from critical ring components (aluminum chamber, crotch, front end components, etc) exceed specified setpoints. Presently there are interlocks that will dump the ring if the global aluminum supply temperature exceeds 105 degrees F. Additional 1 inch diameter experimental water spigots will be added at certain beamlines to enhance cooling capacities. As part of a continuing ES&H effort, 208 V power panels will be relocated inside the X-Ray Ring tunnel to make them more accessible. Some of the required building modifications for the new X17 wiggler system will be completed during this shutdown and some preparatory work for the installation of the new rf cavity will also be carried out.

During the April 1998 shutdown, a new all-copper cavity will replace the existing copper-clad steel cavity and a new copper coupling loop brazed to a beryllia ceramic window will allow the transmission of up to 150 kW into the cavity. This additional power will enable reliable 438 mA operation at 2.584 GeV and operation at more than 250 mA at 2.8 GeV. Two of these cavities have been ordered. The first was delivered to BNL on September 12. The specification had called for completed rf cavities with proper resonant frequency, conditioned interior surfaces, and proven vacuum seals. All requirements, with the exception of resonant frequency, have been fulfilled. The error in the resonant frequency has been attributed to the bakeout during vacuum conditioning. This value can be easily corrected by replacing the large Helicoflex vacuum seal with a larger cross section seal. The cavity has been transported to the NSLS rf test room area for system testing prior to installation. A series of tests will be performed, including a high power test of the main input coupler, temperature control, dynamic tuning, higher order mode dampers and vacuum system. At the present time the main ion pump, tuner, and input coupler have been installed and the cavity is being checked for vacuum integrity. The other major task for the spring shutdown will be the installation of the new X17 superconducting wiggler. The new wiggler will be able to operate in three different modes: as a 7 pole 4.7 Tesla wiggler (the same as the existing X17 wiggler), as a 13 pole 3.0 Tesla wiggler, or as a 3 pole 5.5 Tesla wave shifter. The wiggler is undergoing tests in the Oxford Instruments test cryostat. Full system tests will be carried out in November with delivery expected by January 1, 1998.

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VUV Ring Status

Stephen L. Kramer

VUV Ring Manager

The VUV Ring has been operating extremely reliably since the turn-on from the winter shutdown. The May shutdown, although it was felt that it would have minimum impact on operations, in fact caused significant reduction in lifetime for several months following the shutdown. The major work performed on the ring in May was a bleed-up of half the ring to a liquid nitrogen boil-off for the replacement of a leaking front end vacuum valve on the U3 beamline. This was the same period of the ring that was opened during the winter 1996-1997 shutdown, but since no new metal was added to the ring vacuum chamber this time, only three days were allocated for conditioning of the ring. Injection and stored beam operation were easily achieved in this short time period, but the lifetime showed a slow recovery similar to the one coming back from the winter shutdown. Since the winter shutdown had more than a week of scheduled conditioning of the vacuum chamber with beam, the lifetime had recovered closer to pre-shutdown values by the start of operations. The spring shutdown had less amphere hours accumulated prior to the start of operations, resulting in a lower beam lifetime. The lifetime continued improving during the first month of operations.

Further complicating the recovery from the May shutdown was the conditioning of the new U12IR mirror. Unlike most beamline mirrors, the U12IR first mirror is inserted into the ring vacuum chamber and the gas from it diffuses throughout the ring. Despite the excellent cleaning of this mirror prior to insertion, its outgasing was significant and caused about a 20% reduction in lifetime at the highest currents. The recovery of the lifetime had a similar dependence on integrated current as any of the previous bleed-ups of the ring chamber. As a result of this experience, the operators will periodically use the 0:00 to 08:00 hour VUV Ring study shifts to sweep the beam vertically, in order to desorb gas that has diffused from other parts of the vacuum chamber normally illuminated by the operational beam or coming from beamlines and mirrors. This should help maintain a cleaner vacuum chamber and possibly improve the lifetime and radiation levels.

Most of the improvement work on the VUV Ring since the shutdown has been in the area of diagnostics. We welcome Nathan Towne to the staff. Nathan will be working on understanding the very complicated RF systems on the VUV Ring and on improving their impact on the beam stability. Significant improvements have already been made on reducing the phase noise of the stretched beam. Under the direction of Ron Nawrocky, other diagnostic work has been progressing on: (1) fast beam position monitors (BPM) to measure the non-linearity of the beam, (2) a calibration system for the slower BPM's that will help maintain the absolute beam orbit position in the ring, (3) new profile monitor mirror to reduce the apparent beam motion with current (an artifact of the heating) and (4) a faster beam profile monitor to measure fast beam size variations and separate them from the fast beam centroid motion. Al Borrelli has been carefully going through the difficult task of upgrading the slow BPM electronics and cabling, which will improve their dynamic range and reduce their noise pickup.

Looking toward the future, the 1997-1998 winter shutdown will see the installation of the long awaited radiation shielding improvement to the VUV Ring. Once this reduces the radiation during injection, plans can then be made to implement the proposed Top-Off Method for Injection (TOMI). New injection bump magnets are being planned which should allow this method of injection to take place with minimum orbit motion. Once studies have shown that TOMI is acceptable to the users, the issue of injecting with the shutters open will be addressed and then the question of how frequent the injections should be (how much current variation), will have to be decided.

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NATIONAL SYNCHROTRON LIGHT SOURCE 1998 ANNUAL USERS' MEETING AND WORKSHOPS

The 1998 NSLS Annual Users' Meeting and associated Workshops will provide a scientific forum in the areas of physics, chemistry, biology, materials science, geology, and medicine. New research opportunities and significant accomplishments wil be presented in both invited talks and contributed posters.

Workshops

The program of one-day workshops will focus on specific scientific topics and techniques of interest to the synchrotron community. The following one-day workshops are currently planned:

- "Using Powder Diffraction Data to Solve Crystal Structures", Larry Finger, Carnegie Institute of Washington
- "XANES: Theory, Practice and Data Analysis", Boyan Boyanov, No. Carolina State University
- *"In situ* Surface Manipulation", Ian Robinson, University of Illinois @ Chicago
- "Crystallography at High Pressure, Techniques for Everyone", Jiuhua Chen, SUNY @ Stony Brook
- "Application of Infrared Synchrotron Radiation", Larry Carr, NSLS
- "Biological and Environmental Applications", Barbara Illman, U. of Wisconsin, USDA/FS

Social Functions

A reception will be held for all meeting/workshop registered attendees on Monday evening in Berkner Hall. Both the equipment exhibit and poster session will be on display for viewing. Details are currently underway to make Monday's reception a memorable evening for both long-time and new Users of the NSLS. A varied selection of hot and cold hor d'oeuvres will be served along with complimentary refreshments. The conference Banquet will be held on Tuesday evening on May 19 overlooking the Long Island Sound at the Port Jefferson Country Club at Harbor Hills. You won't want to miss the incredible sunset!

Equipment Exhibit

An instrumentation and equipment exhibit will be held beginning on Monday evening at 5:30 pm in Berkner Hall with a reception for all registered meeting/workshop attendees. The exhibit will close on Tuesday afternoon at the end of Tuesday's Annual Meeting session.

Contributed Posters

Poster Sessions will be held concurrently with each workshop and with this year's Equipment Exhibit. Please request the Registration Booklet in order to obtain for further details on this year's unique presentation of your new and important research results and beamline instrumentation advances. The deadline to submit an application for poster session submission is **April 24, 1998.**

Registration

Complete registration, housing, and transportation information will be sent to those returning the attached response card before February 17, 1998. Individuals registering on or before May 1, 1998 with full payment will receive a discount on the Users' Meeting registration fee. Mail your response card early! Don't miss out on this year's meeting!!

Planning Committee Members

John Parise, SUNY @ Stony Brook Chairman

Harald Ade, NCSU Workshop

Elaine DiMasi, BNL-Physics Poster Session

Linda Feierabend, NSLS Meeting Coordinator

John Hill, BNL-Physics Meeting Program

Eva Rothman, NSLS User Administrator

Nancye Wright, NSLS Equipment Exhibit

To receive additional registration information: Contact Linda Feierabend @

Brookhaven National Laboratory, User Administration Office, NSLS Bldg. 725B, P.O. Box 5000, Upton, NY 11973-5000. You may also respond via Phone: 516-344-5763, FAX: 516-344-7206, or e-mail LSUSRMTG@BNL.GOV.

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TRAFFIC REGULATIONS AT BNL: INCREASED ENFORCEMENT

BNL has traffic rules that must be followed by everyone on site - employees, contractors, and yes, all guests such as NSLS users. BNL Safeguards and Security has begun enforcing traffic rules much more vigorously, and in the past two months over 86 tickets have been issued. Avoid fines, inconvenience, and endangering your colleagues at BNL: follow the traffic rules!

THE FINES: All traffic fines are $50, except moving violations which are $100. Fines will increase by $25 for repeat offences within a year. From the time a ticket is written the offender has 30 days to appeal. Appeals are heard by the Traffic Safety Committee which meets regularly and schedules appears as the first agenda item.

THE RULES: The general traffic rules for New York State have been adopted for all vehicles operated on the Laboratory site. In addition, on-site rules limit speed to 30 miles per hour unless otherwise posted, and allow parking only in designated areas.

REMEMBER, paying attention to safety does not end when you walk outside the NSLS. Moving violations are safety violations. And BNL Safeguards and Security is taking traffic rules and violators very seriously.

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Identification and Tagging of Equipment

The Department of Energy requires that all capital equipment at BNL have bar codes or tags to indicate ownership. If your organization does not have tags (logo's, etc.) we will supply blank tags (see sample below).

These tags are available at the NSLS stockroom free of charge. Please obtain tags, fill in your organization in the space provided, and apply to all unidentified equipment belonging to your organization. The serial numbers on the blank tags are for your optional use in recordkeeping.

BNL's Supply & Materiel Division will be conducting periodic inspections to ensure proper identification of all equipment. If, during the inspection, untagged equipment is found, a tag will be applied. If you have any questions or need assistance please call Donna Buckley at extension 3599.

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DISCLAIMERS : Revised Date : October 13, 1998.

IMPORTANT SAFETY NOTE:

Proper Containers For Cryogens

New rules are in effect now as a result of a recent incident at the NSLS. A coffee thermos with a screw top was used to store and transport biological samples cooled by shaved dry ice; the screw top, normally kept loose, was somehow tightened enough so that the CO_2 gas could not escape. The thermos ruptured, spraying two users with its contents and creating projectiles which landed as far as 20 feet away. Luckily, only minor injuries were sustained.

RULES NOW IN EFFECT:

- 1. All containers used to hold cryogens must either have engineered over pressure protection or must remain unsealed.
- 2. Screw cap containers may be used, but the screw caps must be replaced with some other loose fitting cap. A styrofoam plug works well.

Beamline Local Contacts and Spokespersons must enforce these rules. Operations Coordinators will monitor this enforcement.

ES&H Highlight #10, available from Nick Gmur @ x2490

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CALL FOR GENERAL USER PROPOSALS

Deadline for proposals and requests for beam time on the NSLS X-Ray and VUV Rings is

Monday, February 2, 1998 for scheduling May - August 1998

NEW PROPOSAL FORMS! THE NSLS General User Program is now accepting both 2-year and single-project protein crystallography proposals; please follow directions on proposal form. Forms are available from the Web or via anonymous ftp to ftp.nsls.bnl.gov, /nsls/pub/lsadmin

Prior to Submitting a Proposal

You must contact the beamline personnel responsible for the beamline(s) selected in order to verify technical feasibility on the beamline(s) and discuss any special arrangements for equipment. Your chance of getting beam time is improved by being able to use more than one beamline.

Preparing Your Proposal

The same form is used for new proposals and for beam time requests against existing proposals. Follow the instructions on the proposal information sheet. All information must be typed or printed legibly. Be sure all of the required sections are completed and submitted at the same time. MAIL OR FAX **ONE COPY** of the proposal form, and any attachments to the NSLS User Administration Office. Only **one copy** is required - do not mail a hard copy or fax a second if you have already faxed one. **NEW: Do not send a Safety Approval Form.**

Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Monday, February 2 in order to be considered for the May - August cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on February 2. We encourage submitting new proposals by mail prior to the deadline. Beam time requests for active proposals will be accepted after the deadline, but will be allocated beam time only after requests received on time have been allocated. Late requests are not eligible for a rating upgrade if beam time could not be allocated to them.

Each proposal will receive a prompt preliminary review to verify that it is complete and legible. If there is a problem with the proposal, you will be contacted immediately. Submitting your proposal well in advance of the deadline date assures that the User Administration Office has time to reach you and that you will have enough time to correct any deficiencies.

Additional Information and Forms

Blank proposal forms and instructions, a guide to the NSLS beamlines, and more information about the General User Program are available on the World Wide Web at **www.nsls.bnl.gov**, or by contacting E. Pinkston or L. Rogers at the NSLS User Administration Office. Office hours are Monday through Friday, 8:00 am to 5:00 pm Eastern Time. Contact information is on the back page of this Newsletter.

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Important Upcoming Dates

Nov. 13, 1997	**NSLS Town Meeting**
Nov. 14, 1997	**Users' Executive Committee Meeting**
Nov. 22, 1997	**15th Anniversary of NSLS Dedication**
Jan. 16, 1998	**Deadline for Submissions, March Newsletter**
Feb. 2, 1998	**Deadline for General User Proposals**

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