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Real-Time Protein-Structure Solving

Researchers at the NSLS solve a protein crystal structure within hours of taking the data

R.M. Sweet

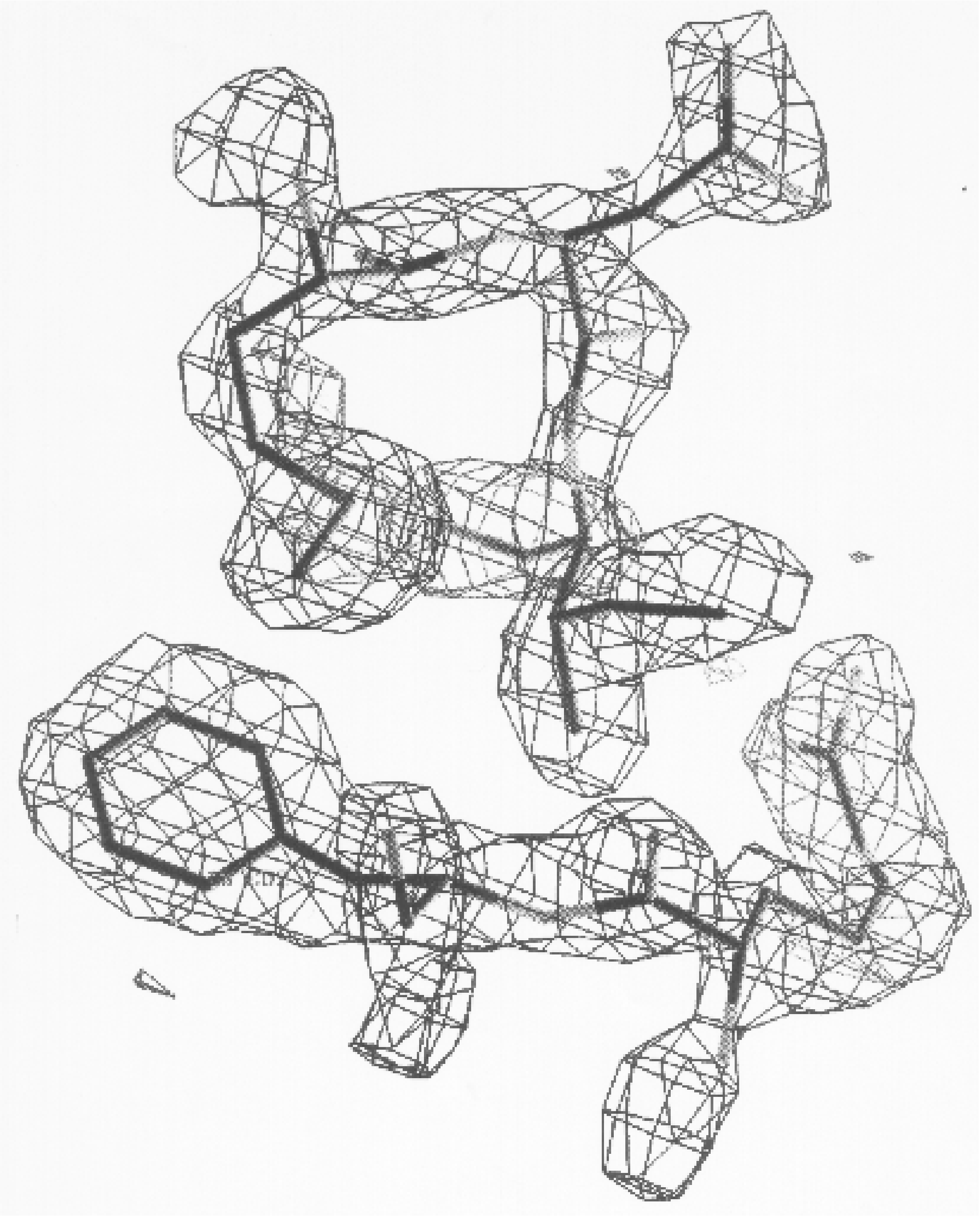
Biology Department, BNL

During the single-bunch running of the NSLS X-Ray Ring at the end of November 1996, and working at the BNL Biology Department's beamline X12C, Chris Hill's group from the University of Utah solved a protein crystal structure within hours of taking the data and before they left the NSLS. The molecule was an 80-residue domain of the HIV-1 capsid protein.

The solving of the structure depended on a trick pioneered at NSLS beamline X4A - the use of selenium as an anomalous scatterer in the protein. In each protein chain the four natural sulfur-containing methionine residues were replaced, during the bacterial-cell culture that was used to produce the protein for crystallization, by the synthetic equivalent, seleno-methionine. Since the chemical properties of Se-methonine are so similar to S-methionine, the protein molecule folds in the same way and the crystals grow the same. Diffraction data were taken at three wavelengths at beamline X12C using the "automatic" MAD (multi-wavelength anomalous diffraction) data-collection method, developed by J. Skinner and R. Sweet. In this technique the beamline's software takes care of tuning the monochromator to the precise wavelengths of the features of the Se-atom absorption in the crystal. The dispersive and anomalous differences among these data will provide strong information about the phases of each diffraction amplitude. The data were taken on a CCD-based area-sensitive detector provided to the BNL Biology Department by the group of Walter Phillips and Marty Stanton (Brandeis University). The diffraction data were taken to a resolution of 2.2 Å from the crystal, which forms in spacegroup $\mathbf{P4_32_12}$.

Hill's group from the University of Utah included Theresa Gamble, Sanghee Yoo, Felix Vajdos, and Cynthia Phillips as the visitors to the NSLS, and Wes Sundquist, a collaborator at Utah. While working at the beamline to collect data from their next project, they located and refined all four Se sites, refined the phases by solvent flattening and histogram shifting, and calculated an electron-density map to reveal the structure. This was despite no prior information on selenium locations or any other phase information. The first electron-density map, produced roughly 54 hours after they *began to take the data*, was very high quality (**Figure 1**). Peptide bonds showed clear carbonyl oxygen atoms and most side chains could be assigned with ease, e.g. the one-atom difference between phenylalanine and tyrosine is quite distinct. The course of the polypeptide chain could be traced immediately (by Hill in Utah) from the original map that resulted from the MAD phases. The crystal structure reveals protein-protein interactions that appear to contribute to the HIV (AIDS-virus) structure. A manuscript currently is in preparation.

Figure 1. View of the electron-density map calculated from MAD phases calculated by Chris Hill's group 54 hours after beginning data collection at beamline X12C. The final structure is shown as a stick model.



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Chairman's Introduction

Michael Hart

As this newsletter goes to press the NSLS starts, once again, to deliver user beam time. An enormous amount was achieved during the 1996 winter shutdown, and much of the work is described in a series of articles in this Newsletter. Congratulations go to all of the staff involved in this most successful venture.

In parallel with the current round of PRT tenure reviews the NSLS has been updating the Policies and Procedures Document which is the guide book for all experimenters at the NSLS. The structure and content of the previous edition, first issued in September 1988, was strongly determined by the experiences of the previous few years of construction, commissioning and operations with both PRT and General User experimental programs.

In the current edition we have made substantial changes in the details of procedures based on the experience of the last decade. We have seen a need to alter the compositions of several important committees, but overall, the structures which have evolved and been in place for some time are robust and do not demand revolutionary change.

This task has taken longer than I had originally planned, and we now have a backlog of completed PRT Memoranda of Understanding (MoUs) from about half of the PRTs awaiting our completion of the Policies and Procedures Document. I am sure that I do not have to remind the remaining Spokespersons that their MoUs are now overdue!!

Let me give just three examples of the change in emphasis, but not in policy, which have arisen and which are planned or implemented already for the current Fiscal Year:

The normal cycle of storage ring operations includes periods set aside for the specific purpose of improving the performance. For historical reasons the protocols for experimentation with the accelerator facility - "studies" - have not coincided with the protocols under which PRTs and General Users have worked when conducting "experiments" on the VUV and X-Ray Rings. In general terms, although the two activities are conducted in compliance with DOE and BNL ES&H requirements, they have been unconnected because most machine physics experiments have been contained within the ring shielding.

The "studies" experiment on Labor Day 1996 was different from the usual machine experiment, but not unique or unprecedented, in that the experiment was not totally contained within the shield wall of the X-Ray storage ring. The electron source characteristics in storage rings can be altered to achieve different optimizations of brightness, source shape and beam lifetime. But the electron source characteristics also influence the production of gas bremsstrahlung in the insertion device straight sections of the storage ring. The machine experimenters had set up diagnostics on several beamlines to monitor straight section bremsstrahlung in various locations when the electron source size and emittance were varied. Dose rates from scattered gas bremsstrahlung off the first optical component in the beamline were observed; the integrated dose over about 10 minutes at the front end of X21 was about 100 mRem. These levels were measured at the locations where doses were maximum, and where no personnel would be present under normal circumstances. In this case, these areas were monitored by Chipmunk radiation monitors and no personnel were near the source of the radiation. Appropriate radiation monitoring equipment and shielding were in place, thus ensuring that no individual was at risk of receiving a substantial radiation dose. Consequently the outcome of this experiment was not classified as reportable under any of the DOE criteria.

Nevertheless, the NSLS management decided that there were lessons to be learned and set up a Task Force, under the Chairmanship of Dr. L. Berman, to collect the experiences of all present and to report with recommendations to the Deputy Chairman, Dr.S. Krinsky. In conjunction with the Associate Chairman for Environment, Safety and Health, Dr.W. Thomlinson, the Coordinator for ES&H, Mr.N.Gmür and Dr.L.Berman an internal memorandum has been lodged in the record and the policies and procedures for the conduct of machine physics studies have been updated to coincide, in so far as that is reasonable and practicable (ALARA), with Policies and Procedures for user experiments at the NSLS. Copies of that memorandum are, of course, available to anyone interested in the technical details from the User Administration Office.

Evolution and improvement are the objectives of changes which I raised for discussion with the Users Executive Committee, some of the Proposal Study Panels and some of the General User Oversight Committee members during the last cycle. Although the work of those three groups is crucial to the operation and success of the NSLS as a National Resource for synchrotron radiation science, it is nevertheless a fact that it has become increasingly difficult for scientists external to BNL to contribute fully to those activities. I therefore intend to remove the present task of allocation and scheduling from the General User Oversight Committee and to concentrate the contribution of peer review in the Proposal Studies Panels.

The Users' Executive Committee (UEC) is an independent representative group, elected by you, the users, and crucial to our joint efforts and ability to develop and exploit to the full the facilities which the NSLS is able to offer.

The Proposal Study Panels (PSPs) have the important role of assessing the quality and beam time needs of new programs. Past and present members tell me that in the larger subject areas it is crucially important that panels meet - face to face - for a variety of reasons. In more specialized areas, meeting by conference telephone or even by e-mail is appropriate. I have no doubts about the efficacy of both of these methods of operation, according to the circumstances. Some users have suggested to me that anonymous multiple peer review, as practiced by some journals, is appropriate. I intend that the Proposal Study Panels should continue to be named individuals who are seen to be practicing peers in the fields of scientific endeavor present at synchrotron radiation facilities. I intend to increase the number of members of the PSPs in Biology in the short term, in all areas of science which exploit our unique infra-red beamlines in about one year's time, and overall to roughly double the present number of PSP members.

Something has to give!

Over the years the formulaic approach to proposal grading and progression has been firmly established at NSLS. The PSPs give a grade 1-5 (with 1 high) and specify the total beam time for the term of the application (two years for most). This grade progresses as beam time is used with penalties and bonuses as set out in NSLS Policies and Procedures. Scarce external scientific peer power has been used in the **General User Oversight Committee** to schedule the PSP awards onto individual beamlines. I intend that that task shall be done by on-site personnel; PRT schedulers (as at present), NSLS Beamlines staff, BNL staff from other departments and resident PRT personnel, for example Spokespersons. That is not to say that the GUOC will vanish! A very important aspect of the GUOC's terms of reference relates to the arbitration of disputes. For this role the reconstituted General User Oversight Committee will be composed of about four members external to Brookhaven. As at present, the GUOC will be the appeal committee for disputes between PRTs, General Users, and NSLS. Questions relating to beamline damage etc. are reported to the User Administration Office and referred to the Associate Chairman for ES&H, Dr.W.Thomlinson with the possibility of appeal to the GUOC. Questions relating to scheduling will be referred to the Head of the Beamline R&D Group, Dr.D.P.Siddons with the possibility of appeal to the GUOC. Based on past experience these tasks, though very important, will only rarely require the intervention of the GUOC.

Finally, let me comment on the future of Insertion Device Executive Committees (IDECs). Many readers will have had no experience of IDECs, so let me remind you of what they are:

For the last decade the NSLS insertion devices have been unique resources, not just within the United States but within the world scene of synchrotron radiation. With unique facilities, outside the experience of most users and to some extent outside any-ones experience), the IDEC was charged not only with the task of scheduling those precious beamlines but also with the task of developing new experimental opportunities and giving preferential encouragement to novelty. As the NSLS continues to develop user programs based on new insertion devices, such as the elliptically polarized wiggler and the in-vacuum narrow gap undulators, new IDECs will be formed. By their very nature IDECs have a natural lifetime of 1-3 years, depending on the technological complexity of the beamline design and state of development of the relevant experimental methods. After that period the IDEC should be dissolved and the normal PRT arrangements will pertain.

I have already received comments about these changes from present and past members of the Users Executive Committee, the Proposal Study Panels and the General Users Oversight Committee. Your comments too are most welcome.

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Upgrade of the Soft X-Ray Microscopy Beamline X1A

Janos Kirz

Physics Department, SUNY @ Stony Brook

The X1A beamline was designed over a decade ago, as the first insertion device beamline on the X-Ray Ring. It has been used since 1989 to support our program in soft x-ray microscopy [1]. (*NSLS Newsletter, November 1993*). Since the experiments require coherent illumination, the beamline is designed not to collect a large solid angle but rather to deliver the coherent portion of the beam to the experimental stations.

The original design [2] was based on the idea that one grating monochromator can feed two experiments simultaneously, as long as one of these uses the undulator fundamental, the other the second harmonic. The two wavelengths were focused on two separate exit slits, beyond which steering mirrors directed the beams to the experiments. This arrangement worked well until it became clear that the combination of microscopy with spectroscopy is a remarkably powerful technique. For absorption spectroscopy one branch needed to scan the grating, and the other necessarily found its photon energy scanned, so that simultaneous operation was lost. Space limitations also affected the optical performance and the separation between the two experimental stations.

The new design [3] uses a scraping mirror to split the beam into two independent branches. This mirror, as well as a similar one on the inboard branch that intercepts the undeflected part of the beam, is a toroid designed to focus the beam horizontally onto the monochromator entrance slit and vertically onto the exit slit. They are both made of single crystal silicon blanks with gold coating on the reflecting surface, and water cooling from the back. Past the mirrors the the optics of the two branches is identical. The water cooled entrance slits are followed by sperical gratings on silicon blanks, and by exit slits. The location of the slits along the beamline is fixed. We can get away with this scheme and obtain good resolution (resolving power over 4000) throughout the 250 eV - 540 eV range because we are only interested in the coherent part of the beam. This corresponds to very small acceptance, so off-axis aberrations do not have a chance to affect the resolution in a serious way.

The beamline takes full advantage of the brightness of the undulator source, and of the relatively large angular divergence of the electron beam in the straight section. The former determines directly the coherent flux at the experiment, and the latter broadens the peak in the undulator spectrum so that we get good coherent flux over a broad energy range without changing the gap.

The performance of the monochromators (**Figure 1**) agrees with our expectations. We have much improved resolution compared to the old beamline. The flux, as measured with a calibrated photodiode [4], is also improved compared to what we had before. The beamline promises improved stability, and has provided much needed space around the experimental stations.

The outboard branch is the home of the scanning transmission x-ray microscope, STXM [5]. It shares time on this branch with the brand new cryo-STXM [6] which keeps the specimen at or near liquid nitrogen temperature to reduce radiation damage. This is particularly important for wet, unfixed biological specimens, and when multiple images are taken of the same specimen area for chemical mapping or tomography.

The inboard branch supports the scanning photoemission microscope (SPEM) [7], as well as programs in holography [8] and soft x-ray diffraction [9]. While the two branches (as well as the spectroscopy beamline X1B) share the undulator source, they can now tune their monochromators individually through the relatively broad undulator peaks, improving the flexibility and overall throughput of the beamline by a large factor.

We are fortunate to have benefited from the NSLS building expansion, funding from DOE OHER and DMR, from APS and NSLS hardware that had been used on the X13A beamline, and from support from Stony Brook and NCSU. Naturally, we are re-using as many components of the "old" beamline as practicable. The basic ideas for the upgrade came from Harald Ade (NCSU), Chris Buckley (King's College, London), and Malcolm Howells (ALS). Important contributions to the design were made by Steve Hulbert (NSLS), Ian McNulty (APS), and by Chris Jacobsen, Barry Winn, Jianwei Miao and Angelika Osanna (SUNY @ Stony Brook). Tom Ovesluizen (Creative Instrumentation) had the critical role of engineering consultant. We shut down for the upgrade at the start of the July 4th holiday. Construction and commissioning was done by the Stony Brook microscopy group (especially Sue Wirick and Barry Winn), as well as Tom Oversluizen and Huihuang Zhang (NCSU). We received much welcome support from the NSLS staff.

Completion of the upgrade involves final adjustments to the toroids, and this will be done when beam returns. The microscopy program should be back in full swing before the end of January.

Figure 2: One of the new gratings.

Figure 3: Layout details of the new X1A beamlines.

[1]J. Kirz, C. Jacobsen and M. Howells, *Quarterly Reviews of Biophysics* **28**, 33-130 (1995).

[2]H. Rarback *et al.*, *J. X-ray Science & Techn.* **2**, 274 (1990).

[3]B. Winn *et al.*, *Rev. Sci. Instrum.* **67**, A31 (1996).

[4]AXUV - 100 from International Radiation Detectors, 2545 W. 237 St., Torrance, CA 90505

[5]C. Jacobsen *et al.*, *Optics Comm.* **86**, 351 (1991).

[6]J. Maser *et al.*, SPIE Proc. **2516**, 78 (1995).

[7]C.-H. Ko *et al.*, *Rev. Sci. Instrum.* **66**, 1416 (1995).

[8]S. Lindaas, M. Howells, C. Jacobsen, and A. Kalinovsky, *J. Opt. Soc. Am.* **A13**, 1788 (1996).

[9]D. Sayre and H. N. Chapman, *Acta Cryst.* **A51**, 237 (1995).

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The Winter Shut-Down Low-Down

Marty Woodle

Mechanical Section Head

The amount of work to be accomplished made the winter shutdown an extremely busy one for both the Mechanical and Electrical sections. This situation was exacerbated by the loss of several key technicians due to illness. We were able compensate for this by borrowing some personnel from RHIC and AGS. Additionally, the NSLS Operations section lent us two of their coordinators to help with the mechanical work.

The major accelerator related tasks were to install a new chamber in the VUV Ring at the U1/U2 location featuring a U2 port with a 50 × 100 mm infrared photon aperture. The RF Group in the meantime, installed a new transmitter and coupled together two existing ones to increase the available power to an X-Ray Ring RF cavity. They also replaced one of our original Klystrons. Another Electrical Group task was the initial installation of a number of beam chamber measuring stations in the X-Ray Ring which when completed will very accurately monitor PUE movement during operation.

A major undertaking in the X-Ray Ring beamline area was the installation of 20 new Be windows. The Mechanical Group is very grateful for the help of the participating users, some of whom carried out the installation themselves.

In the area of the NSLS utilities systems, three major tasks were accomplished: The replacement of our aged main cooling tower, the tie-in of Central Chilled Water Facility cooling to the Experimental Water System as well as the installation of additional utility systems instrumentation.

I have asked the project engineers on these tasks to provide greater detail on their portions of the shutdown:

- U2 Dipole Chamber Replacement
- Additional RF Power for the X-Ray Ring
- Linear Accelerator Klystron System Upgrade
- Measurements of the Movement of the Vacuum Chamber on the NSLS X-Ray Ring
- Beryllium Windows
- Capacity / Control for NSLS Cooling Systems

U2 Dipole Chamber Replacement

Don Lynch

Infrared beamlines for infrared microspectroscopy at the NSLS are one of the faster growing segments of the NSLS user community. In order to extract the IR radiation from the VUV storage ring, larger chamber openings than the standard VUV beam port are required. Over the past several years new dipole vacuum chambers with larger beam ports subtending 90 × 90 milliradians have been installed at U12 and 50 (vertical) ×100 milliradians at U10. In the December 1996 shutdown, a new dipole vacuum chamber with a 50 (vertical) × 100 milliradian beam port has been installed at U2. For comparison, standard NSLS VUV ports are 10 (vertical) × 90 horizontal milliradians.

Concurrent with the installation of the modified chamber, a new water-cooled mask was installed immediately downstream of the new U2 port. The new mask was required to accommodate the larger beam emerging from the new port. The December 1996 shutdown efforts for U2 were concluded by the reinstallation of the existing U2 UHV isolation valve. The remaining components for the completion of the new U2 beamline are currently being fabricated and/or procured and will be installed during the next few months.

The modification of this chamber will bring the number of IR-capable ports operating and/or under construction in the NSLS VUV Ring to 4 (at U2, U4IR, U10 and U12IR). This commitment by the NSLS to provide infrared beamlines reflects our desire to satisfy the demand for facilities in this regime by a diverse group of experimenters.

Additional RF Power for the X-Ray Ring

Manny Thomas

During the recent NSLS winter shutdown, an additional x-ray 52.8 MHz, RF power amplifier string was added to the system. RF and mechanical technicians, with programming help from the Computer Group (see page 9), worked diligently many extra hours during the past six weeks to complete this six month project.

Before the shutdown, the x-ray beam energy was supplied by four 125 kW amplifiers, each driving an individual resonator within the x-ray beamline. Running at 80% maximum per system for reliability, thirty-six kilowatts per cavity is supplied by each system to develop the synchronous gap voltage needed for acceleration including a large overvoltage factor. The remaining 64 kW is available for the beam current.

At 350 mA beam current, running at 2.584 GeV with all insertion devices active, the total power per system approaches maximum.

To increase reliability and to be able to consider higher beam currents and higher energies, an additional transmitter was added which could provide another 100 kW of RF power to the beam. This was done by paralleling an existing amplifier with another using a hybrid combiner. An added feature using this technique is that the combiner provides some isolation between the beam and the transmitter making the transmitter tuning process less critical. Performance will be studied and reported on in the near future.

By redistributing the power in the four systems, each amplifier could be operated at lower power levels reducing the possibility of failure. The limiting factor at this time is the cavity drive loop in the dual system, and its ability to transmit double its present power. A newer design with better cooling has been constructed but is untested. A new cavity will be available soon to facilitate this test.

When the overall system is completed, running 500 mA at the present energy with high reliability will be feasible.

Linear Accelerator Klystron System Upgrade

Joe Sheehan

The linear accelerator is powered by three 20 Megawatt Klystrons to provide the electron beam that is injected into the booster ring. The Klystron which powered the pre-buncher and first accelerator section has been in operation since the initial construction of the linac system in 1978. The ancillary pulse transformer tank assembly that houses the Klystron was originally used for other laboratory operations. This transformer assembly was a one-of-a-kind item requiring special control equipment and not having compatible spare parts. A new transformer/Klystron assembly was installed that is identical to the new systems on accelerator sections 2 and 3, and will provide a more reliable and stable injection operation.

Measurements of the Movement of the Vacuum Chamber on the NSLS X-Ray Ring

Om Singh

This project work also involved L. Solomon, D. Lynch, J. Safranek, M. Lehecka and C. Neilson.

At the NSLS, work is in progress to develop an accurate, sensitive and reliable system to monitor vacuum chamber motion. As the vacuum chamber heats and expands from synchrotron radiation, the electron beam position monitors (PUEs) move. This motion introduces errors into orbit measurement and orbit feedback systems. Future improvements in orbit stability may be attained by inclusion of this data into the orbit feedback system.

The chamber motion from all 48 PUE's locations will be measured and logged into the computer for various operational conditions. High sensitivity LVDT probes, mounted on their support structure (stand), are used to monitor horizontal as well as vertical chamber motion. To minimize the effect of temperature fluctuations in the x-ray tunnel on the LVDT support structure, temperature compensating stands have been designed and fabricated. The work is in progress to measure quality of thermal compensation of the stands under various temperature conditions. It is expected that commissioning of these stands in the X-Ray Ring will begin in the Spring this year.

Beryllium Windows

Don Lynch

Beryllium windows are used by most NSLS X-Ray beamlines to separate machine vacuum from beamline vacuum (or atmosphere). The properties of beryllium make it an excellent choice for this service. However, some of the radiation produced by the X-Ray Ring is absorbed by the beryllium, consequently heating the windows. This heating produces thermal stresses in the beryllium foil, which, if large enough can result in failure of the window. To limit these stresses, the windows are designed with active water cooling.

As part of the ongoing program to increase the NSLS X-Ray Ring operating current to 500 mA at a beam energy of 2.5 GeV (or other combination of energy and current using the maximum available RF power), an exhaustive study of the thermal effects of x-ray absorption by beryllium windows as well as other components was performed. The window analysis included a parametric study of the relationships between window height, width and thickness, absorbed energy, distance from the source, and differential pressure to maximum stresses in the window.

The goal of these analyses was to develop guidelines for beryllium window usage on NSLS X-Ray Ring beamlines which assure indefinitely long window lifetime at any operating condition up to 500 mA and 2.5 GeV (or equivalent), no decrease in x-ray throughput from current windows, a minimum number of window design permutations, mechanical dimensions compatible with existing installed windows, and minimum cost. This was achieved as follows:

Finite element thermal stress analyses were performed to correlate window height, thickness, width and x-ray intensity/distance from source with maximum allowable stress. It was found that for windows illuminated by bending magnet sources, the optimum thickness was 0.254 mm (0.010"), the width had no effect on maximum stress, and the height must be less than 1 mm for each meter of distance from the window to the source. In order to minimize the number of design variations, a "line" was draw at a distance of 10 meters: all windows closer than 10 meters would require a 5 mm high window; all windows 10 meters or more removed from the source would require a 10 mm high window. There are no exceptions to this rule.

Recent studies at other institutions have demonstrated improved beam quality for certain experiments from highly polished windows. All of the new windows are designed to be polished to a finish of 0.4 microns, average roughness, with polishing performed in the vertical direction.

To accommodate the variety of window widths and flange arrangements currently used on existing windows, 6 standard designs were adopted as follows:

1. 4-1/2 inch flanges, rotatable flange downstream, 5 mm high × 41 mm wide clear aperture, 6 inch length.

2. 4-1/2 inch flanges, rotatable flange downstream, 10 mm high × 41 mm wide clear aperture, 6 inch length.

3. 4-5/8 inch flanges, rotatable flange upstream, 5 mm high × 41 mm wide clear aperture, 6 inch length.

4. 8 inch flanges, rotatable flange downstream, 5 mm high × 123 mm wide clear aperture, 6 inch length.

5. 8 inch flanges, rotatable flange downstream, 10 mm high ×123 mm wide clear aperture, 6 inch length.

6. 8 inch flanges, rotatable flange upstream, 5 mm high × 123 mm wide clear aperture,

tapered approach, 4.625 inch length.

In order to increase X-Ray Ring current to 350 mA and 2.584 GeV as soon as possible, it was decided to replace beryllium windows in a 2-phase operation, prioritizing replacements on the basis of calculated thermal stresses. Phase I was accomplished in the December 1996 shutdown with the replacement of the 20 windows which our calculations indicate were inadequate for 350 mA and 2.584 GeV operation (or equivalent). These windows were at X3A, X4A, X6A, X12A, X12B, X12C, X13, X14A, X14B, X15A, X19C, X20A, X20C, X21, X22A, X23A, X23B, X25, X26A and X26C. Replacement of these windows allows these beamlines to operate at up to 500 mA / 2.5GeV (or equivalent).

Phase II of the beryllium window upgrade will replace 17 additional windows. Thermal stress calculations for these windows indicate that they are presently acceptable, however, before operation at 350 mA / 2.584 GeV, they must be replaced. These windows are at X3B, X4C, X6B, X7A, X10B, X11A, X16A, X16B, X16C, X18A, X18B, X20B, X22B, X22C, X27A, X27B and X27C. They will be replaced over the course of 1997, with completion during the December 1997 shutdown.

All other beryllium windows installed in the NSLS X-Ray Ring have been analyzed and found acceptable for operation at up to 500 mA / 2.584 GeV (or equivalent).

Capacity / Control for NSLS Cooling Systems

Ron Beauman

Just a few years ago it was common knowledge that the NSLS cooling systems were under significant strain. Considering the intimate relationship between the cooling systems and individual ring performance, this strain placed the NSLS and the user community at a disadvantage. To alleviate this, a water system improvement program was established and implementation began.

Our first and continuing task was to better understand our existing systems and a program of documentation and evaluation was started. This not only helped define the problem but also helped in our design, operations, and continuous upgrades.

As our evaluation progressed it became apparent that the connection to the Central Chilled Water Facility (CCWF) was necessary but not sufficient to solve our problems; that is, we designed, and this December completed, an independent, parallel, cooling capability for our four water systems. In order to reliably retain this capacity, our old cooling tower was replaced and we upgraded and expanded its controls. As we have seen on different occasions, this redundancy has allowed us to persevere operations when previously we would have had to shut down. This dual capacity allows us to preserve a flexible response to both our present and future capacity needs.

Simultaneously, we proceeded with a modernization and expansion of our temperature control capability. This work, which is presently completed on the 4 process systems, has resulted in significant improvement in temperature control. In addition, we expanded the experimental water system flow capacity by increasing the pump size and adding more taps for the users. This latter part of our program has supplied some new tap locations with more to be added as subsequent shutdowns permit.

As this phase of our program reaches completion, we begin a new phase. This consists of integrating all the systems into a single control system. Because, as our evaluations have shown, all the systems are coupled, either directly or indirectly, and as such our users' operations are dependent on the stable and integrated operation of all the systems. We have therefore, purchased and are installing an Advanced Process Automation and Control System (APACS). This system will allow us to control, monitor, and diagnose our system in a comprehensive and cost effective manner, therefore, during this shutdown, we began installing additional temperature and pressure sensors throughout the process and chilled water systems. Furthermore, to insure quicker response, we began the installation of automated valve actuators which when completed will result in a truly integrated system.

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FOCUS ON . . . . .NSLS COMPUTER GROUP

John Keane

Computer Systems Head

The NSLS Control Room creates a dramatic impression. This nerve center monitors and controls all the machines and hardware used to operate the accelerator, booster, and storage rings. The amount of hardware dispersed throughout the building is astounding and every piece of hardware has a link to the Control Room. This critical link is via the NSLS computer system. Indeed, without the computer system the staffing required to operate the machine would be prohibitive. This article will attempt to introduce you to the very professional and hard-working Computer Group.

There are four working sections in the Computer Group; they are the Micro, Workstation, System, and (hardware) Support sections.

The Micro section is responsible for the low-level code which monitors and controls the hardware throughout the complex. Susila Ramamoorthy, John Dabrowski, and Kate Feng-Berman receive the hardware descriptions and requirements from the engineers. They then write the code on the micros that communicates with and controls the hardware via commands sent from the workstations. Providing very reliable and fast communications to equipment spread over the building and sometimes into adjacent buildings is a requirement. A good understanding of both hardware and software is needed in order to work in this section.

The Workstation section establishes communication among the various micros and workstations. This is where all the high-level code is written. Application codes orchestrate a particular requirement such as turning the machine on, measuring an orbit, investigating specific types of machine performance, etc. Pauline Pearson and Yong Tang work in this area, interfacing with machine physicists and operators on one side and the Micro group on the other. The Workstation section also supports the staff and the System section by providing test programs and expertise to resolve miscellaneous problems.

The Systems section, composed of Herb Langenbach and Cheo Teng, is responsible for the compute servers used by all the NSLS staff, the Control Room workstations, and the maintenance of the network infrastructure. They must ensure that the data transmission from micros and workstations is both reliable and fast; that the whole NSLS staff has access to news, e-mail, the World Wide Web, and other services; that backups are done; and that software is updated periodically. An extremely important additional function is the security of the overall system.

Installation and troubleshooting of micros and hardware systems are the responsibilities of the Support section. This section is comprised of Wayne Rambo (supervisor), Henry Link (PC and networking support), Jack Tallent (diagnostics), Gary Frisbie (controls), and Charlie Nielson (insertion devices). This is the group which services and installs control system hardware, and they are the first level of support, day or night, for problems arising in the control systems. The nature of their job requires them to interface with all the computer system users. With the proliferation of computers this group has also become expert in the support of PC systems. Their skills are multifaceted but mainly hardware-oriented.

As the group leader, John Smith charts the technical direction in which the Computer Group moves. He keeps abreast of the newest hardware and software techniques in his mission to continually improve NSLS machine operations and satisfy user needs. Things that the future may bring include the network upgrade. The installation of fiber optics would not only increase transmission speed but also capacity and reliability. To the NSLS experimenters this means faster data acquisition with less dependence on the activities of other users. Uninterruptable power supplies will be added at strategic points in the computer control system with the goal of reducing the time of recovery after "power dips". Subsequent changes will be made to further secure the system. The planned micro upgrades will also enhance communications speed.

The vast technical breadth within the Computer Group has shown us how important communication is - both within the Group and with other groups at the NSLS. It helps us to understand the evolving needs of NSLS staff and users, so please keep us informed of your needs and we will try to keep the ship sailing in that direction.

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Surface Induced Layering in Liquid Metals

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Liquid metals play an important role in many technologies, from smelting and refining processes where nucleation of gas bubbles and slag/metal reactions are of immediate concern to problems in metallurgy, for example zone melting, casting and brazing. Each of these technologies is greatly influenced by the unusually high surface tension found in liquid metals which is, in turn, related to differences between the order at the liquid surface and in the bulk. The surface order/entropy has a profound effect on the melting and freezing of metals. For instance, the degree of supercooling in liquid metals is strongly believed to be related to the excess surface order. Understanding the phenomenology and Ångstrom-scale structure of surface induced order is very much needed, and although there have been major efforts to understand supercooling and pre-melting, structural data on the excess order at liquid metal surfaces remain almost non-existent.

An important theoretical prediction[1], central to the theoretical understanding of the structure of liquid metals, is that of atomic layering at the surface. According to this prediction, atoms near the surface will be ordered in layers parallel to the surface (**Figure 1**), and the amplitude of the density modulation should decay exponentially with depth below the surface with a characteristic length of several atomic diameters. Recent measurements on Hg and Ga provide the first unambiguous experimental verification of this effect[2-3]. Significant differences were found in the decay length and ordering properties of these two liquids. The decay length seems to be a universal property of the metal; it is temperature independent for gallium[4,5] and is virtually unaffected by chemisorbed organic monolayers on Hg[6]. These results provide strong confirmation for the prevailing theoretical understanding of the effective interactions in liquid metals, but also raise important additional questions resulting from several unexpected observations.

In an x-ray reflectivity measurement, surface layering gives rise to a broadened Bragg peak at a wave vector, $q_z \sim 2\pi / a$ where a is of order of the molecular spacing. For liquid metals, where $a \sim 3$ Å, this necessitates measurements out to greater than 2 $Å^{-1}$. The reflectivity at these large q-vectors is weak and can be measured only at a synchrotron source. Since the liquid sample has to be kept horizontal, specially designed liquid spectrometers are required which can continuously tilt the monochromatic incident beam downward by as much as 15 degrees. The reflectivity measurements reported below were obtained at the NSLS (X19C, X22B, and X25) with wavelengths ranging between 0.65 and 1.24 Å by scientists from the University of Chicago, Harvard University, Brookhaven National Laboratory, and Bar Ilan University.

Figure 2 shows the absolute reflectivity R of liquid mercury obtained under a hydrogen reducing environment at room temperature as circles[2]. The solid line is the theoretical reflectivity R_f for a perfectly flat surface, calculated from the Fresnel law of optics. The reflectivity falls from close to unity below the critical angle to 3×10^{-8} at $q_z = 2.3$ $Å^{-1}$, yet it remains within a factor of two of R_f. This clearly indicates a surface roughness smaller than the atomic diameter. The broad peak at $q_z = 2.15$ Å^{-1} is close in position to that of the bulk liquid structure factor and resembles similar features found in reflectivity measurements of surface induced layering in liquid crystals. This is a clear signature of atomic layering at the mercury interface.

The differences between the measured reflectivity, R, and the Fresnel reflectivity, R_f, can be related to the average density profile along the normal to the interface. In the weak scattering approximation the ratio R/R_f simplifies to the square of the absolute value of the Fourier transform of the density derivative. A physically appealing model of the liquid vapor interfaceis obtained from a truncated solid in which the root-mean-square (RMS) deviations of atoms in each layer increase with increasing distance from the interface[2,3]. Layering occurs when the RMS displacements are less than the interatomic spacing, e.g. near the surface. When the RMS displacements increase as the square-root of the distance, the model gives an exponential decay of the layering amplitude. By fitting the reflectivity to this model, the Hg surface density profile, shown in the inset of **Figure 2**, is obtained. It clearly indicates surface induced layering with an exponential decay length ~ 3.5 Å. The top-most layer RMS deviations (less than an Ångstrom) agree very well with those calculated using the thermally excited capillary wave model. In addition, this density profile gives a top most layer spacing which is expanded with respect to the underlying layers by several tenths of an Ångstrom. Similar lattice expansions have been observed at reconstructed solid metal surfaces. The Hg top-most lattice expansion, along with a slightly asymmetric top-most layer, give rise to the deviations from the Fresnel Law at about 0.6 Å^{-1}.

Might the surface normal layering at the interface be a manifestation of the same phenomena which give rise to the bulk liquid pair correlation function? In order to compare the two quantities, we show in **Figure 3** the bulk correlation function along with the density profile after deconvolution by the width induced by capillary waves[2]. Beyond about 4 Å there is excellent agreement between the bulk pair correlation function and the surface profile. This supports the notion that the short-range order in bulk liquid metals and the layering at the surface are closely related and, in turn, indicates that the latter is primarily a simple geometric consequence of the physical requirement to form a sharp interface. Deviations at small distances are in part due to the restricted interpenetration occurring around individual atoms which increases the amplitude of the first peak in the pair correlation function.

Gallium, the only other room temperature liquid metal, has several unique qualities. When compared to Hg, Ga has a smaller atomic diameter, much higher surface tension, an extremely small vapor pressure, an exceptionally large liquid range (~ 2000 K), a large supercooling range (~ 30 K), and short distance bond orientational correlations. It is not clear to what extent variations in bulk properties manifest themselves at the free surface. In **Figure 4** we show the normalized reflectivity from the sputtered clean liquid Ga surface in UHV[3] along with the corresponding normalized Hg reflectivity. Below 2.0 Å^{-1} the Ga reflectivity shows no appreciable deviation from the Fresnel theory, in contrast to the dip observed for Hg. The quasi-Bragg peak which at 2.4 Å^{-1} for Ga is much narrower than in Hg and indicates that the surface layering decay length is much larger in Ga. Fits to the density profile described above, confirm this expectation and give an exponential decay length of 6 Å. This length is approximately a factor of two larger than Hg surface layering length and the decay length of the Ga bulk pair correlation function.

Although there are no theoretical predictions for temperature dependence of the surface induced layering, we have recently carried out measurements of Ga at elevated temperatures[4]. The prominent layering peak at q_z=2.4 Å decreases dramatically upon heating from 22 to 160°C, but its width stays the same as shown in **Figure 5**. Thus, the corresponding density profiles (see inset) exhibits a temperature-independent layering decay length. However, the surface roughness integrated over the coherence area follows the expected square-root dependence of the capillary wave model. The amplitudes of the surface density modulation are found to be significantly underestimated by existing theory and molecular simulations. Recent reflectivity measurements from the liquid Hg surface[7], close to its freezing point (-38.5°C), indicate a dramatic increase in the peak intensity, consistent with the Ga observations

In addition to the experiments on clean liquid metals, reflectivity studies have also been carried out on binary alloys[8-10], surface oxides[10], and organic terminated surfaces[5]. In the case of binary liquid metal alloys, surface segregation of one of the components reduces the interfacial energy, e.g. the surface tension, and even for very dilute alloys, the surface may have a very different composition than that of the underlying bulk. Several different binary gallium alloys have been investigated at the NSLS using x-ray reflectivity and grazing incident angle diffraction techniques. In reflectivity measurements at the surface of a dilute bismuth-gallium alloy[8] (~0.02 % Bi), the sinusoidal like modulation in the reflectivity supports a density model in which the surface is composed of a nearly complete bismuth layer and where the underlying bulk metal (predominately gallium) is layered in a similar manner to the intrinsic gallium surface. Complementary grazing incident angle diffractions studies have provided a direct measure of bismuth segregation[9]. In addition, by modeling the diffuse scattering around the specular condition in terms of the capillary wave model, reasonable estimates of the surface tension have been obtained[8]. Analysis of reflectivity measurements from the gallium-indium alloy (16.5% In) indicates that the surface has a much higher electron density than the underlying bulk. Model fits assign this to a surface layer of almost pure indium, where subsequent layers are at the bulk composition. Finally, oxidation studies of the intrinsic gallium surface show that upon exposure to 180 L of oxygen a 5 Å thick oxide is formed. This layer is of uniform thickness, amorphous, and does not change upon further oxygen exposure, or heating to 300°C [10]. This finite thickness oxide passivation of the surface is analogous to that found for solid aluminum.

While organic films have been extensively studied on solid substrates and liquid water, nothing was known about the structures of these films on liquid metals. In a recent study, monolayer films of thiol molecules (a sulfur terminated alkane chain) were prepared on the liquid Hg surface. The normalized x-ray reflectivity spectrum, R/R_f, from a C18 thiol film on Hg is shown in **Figure 6** along with the spectrum from the pure Hg/vapor interface[6]. The interference between the reflected amplitudes from the vapor/thiol and thiol/Hg interfaces gives rise to the observed modulated reflectivity spectrum. The modulation period corresponds to a fully extended alkane chain and the amplitude supports the notion of a densely packed monolayer. Remarkably, the essential features of the reflectivity, including the quasi-Bragg peak and the dip at small q_z, are identical with and without the thiol monolayer. This clearly supports the notion that the surface layering is intrinsic. Complementary in-plane x-ray diffraction measurements indicate that long-range positional order is only observed when multilayers form and not for monolayers. The absence of an ordered monolayer is surprising, especially when compared to Langmuir films on water where Van der Waals interactions between the carbon chains promote lateral order. These results suggests that the strong chemical interaction with the liquid Hg subphase is responsible for the lateral disorder.

In summary, the present studies unambiguously demonstrate that Hg and Ga exhibit surface induced layering, in accordance with decades old theoretical predictions. Measurements on alloy, oxide, and organic covered surfaces have provided additional information on surface layering and in addition they have provided new insight into these complex interfaces. These studies should lead to an improved theoretical understanding of liquid metals and show a promising potential for the further development of the surface science of liquid interfaces.

This short review describes work carried out by several groups and many people. We gratefully acknowledge the contributions of Lonny Berman, Elaine DiMasi, Olaf Magnussen, Mike Regan, Holger Tostmann and discussions with Ning Lei and Stuart Rice.

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A User's Perspective

Peter Stephens
SUNY @ Stony Brook, UEC Chair

This is my last contribution as UEC chairman to this newsletter, and I intend to close as I opened, haranguing my constituents to tell the world about the good work they're doing at the NSLS.

All of us who have been working on the 1996 Activity Report have been struck by the inconsistent level of response among the various beamlines. For example, I know of one x-ray beamline that seems to be in use continuously, but didn't submit a single abstract. That is a shame, because I stumbled onto a nice Physical Review Letter from that line which should have been featured in the Scientific Highlights. Lonny Berman (NSLS) has compiled some statistics on this problem, and has found that the PRTs which are based nearby are much more likely to comply, and that the general users write far fewer abstracts than the PRTs (relative to beam time usage). The abstract submission procedures are not such a burden that anybody cannot knock out a few of them on the interesting work they've been doing here. There are still a few problems, but nothing fundamental. (For example, one of my abstracts was persistently attached to the wrong figure for a week after I submitted it. It was perfectly nice data, but it didn't have much connection with my text.) Last year, a number of people said that it was terribly inconvenient to access the submission forms via the World Wide Web, and so the NSLS User Administration Office set up a mechanism whereby abstracts could be accepted as word-processor files on diskettes. Ironically, they received only a few such submissions, but I'm sure that it was appreciated by the people who took advantage of this new pathway.

In the present environment of tight competition for research and facility support funds, we must make sure that the funding agencies, the government at large, and the public recognize the importance of the work we're doing here at the Light Source. Real decisions will be made based on our ability to explain the importance of this facility. For example, the Office of Basic Energy Sciences (which funds the operation of NSLS as well as the ALS, APS, and SSRL) is constituting a panel to make a major review, similar in scope to the 1984 Seitz-Eastman report, of the four DOE-supported synchrotron radiation facilities. One of the questions which this committee will address is, "What would be the consequences of the shutdown of one or more of the four DOE-supported synchrotron light sources." I trust that my readers will agree that the synchrotron community must pull together for the common good in thinking about such issues, and not start bickering about which machine should be shut off before their own favorite. Anybody who supports the continued operation of the NSLS must see the importance of preparing accurate information for this committee, but the NSLS can only respond with the data which they have been able to collect.

The PRTs have all agreed to keep the facility appraised of their scientific output (check your memorandum of understanding if you don't recall); likewise, this is a condition on the allocation of time to General Users. For the most part, we don't have to pay for the photons which come out of facilities like the NSLS. That doesn't mean that they are free, but the main obligation to the NSLS is the small administrative task of reporting what we've accomplished. As scientists, the basic reward for our work is the pleasure in telling other people about the results. Let's all do ourselves a favor, and brag a bit to the NSLS!

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New X-Ray Ring Fill Policy

During Normal Operations (with LEGS Running):
A refill will not take place at the pre-scheduled time (i.e. 7:00 a.m. or 7:00 p.m.) if there has already been a refill due to an unscheduled beam dump within 4 hours of that time. Previously the margin was 6 hours, so this should ensure higher average currents.

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Space Committee

Under the direction of Michael Hart, the NSLS has established a new "NSLS Space Committee" for the purpose of reviewing all space requirements for the Department and making recommendations to the Chairman. Space needs regarding NSLS personnel as well as Users will fall under the purview of this committee which will address issues pertaining to offices, floor space, labs, set-up and storage areas and so forth. Committee members include Steve Ehrlich, Mike Kelly, Roger Klaffky, Sam Krinsky and Frank Terrano. Individuals or groups with particular space needs, concerns or even good ideas may forward them to any of the committee members or to the Chairman.

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NSLS Library

Plans are in progress for changes to the NSLS library, room 1-111, located adjacent to the east roll-up door on the experimental floor. Previously this area housed all of the Department's library materials as well as tables and lounge chairs for Users, a computer hooked to the Internet, fax and copy machines, and analog phone lines for modem connections.

Library materials that are current, such as six months worth of active journals, conference proceedings, accelerator reports and selected handbooks and periodicals have been relocated to the second floor, room 2-142. A card reader will be installed on the door of the new area to allow access day or night.

Materials remaining in room 1-111 will be primarily archival. This change allowed us to remove the bookcases from the middle of the floor and several from around the walls to free up space which can be more utilized for User conveniences. An additional computer is planned to be installed as well as selected furniture pieces. Comments regarding room layout are welcome.

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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 Saturday, May 31, 1997 for scheduling September - December 1997

(proposals will be accepted until 5pm on Monday, June 2 because of the weekend deadline)

NEW PROPOSAL FORMS! THE NSLS General User Program is now accepting both 2-year and single-project protein crystallography proposals for beamlines X12B and X12C; please follow directions on proposal form.
Forms are available from the Web or via anonymous ftp to ftp.nsls.bnl.gov, /incoming/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, Safety Approval 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.

Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Monday June 2 in order to be considered for the September - December cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on June 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 here, or by contacting the General User Program Coordinators. Office hours are Monday through Friday, 8:00 am to 5:00 pm Eastern Time.

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MARCH 1997 NSLS NEWSLETTER

IMPORTANT UPCOMING DATES

May 19 - 21, 1997	NSLS Annual Users' Meeting and Workshops
May 16, 1997	Deadline for submissions, March Newsletter
May 31, 1997	Deadline for General User Proposals

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