JULY 1997 NSLS NEWSLETTER

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Important Upcoming Dates

Aug. 14, 1997	NSLS Town Meeting
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Sept. 19, 1997	Deadline for submissions, November Newsletter
Sept. 30, 1997	Deadline for General User Proposals
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Advanced Polymers PRT at Beamline X27C

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SUNY at Stony Brook

Introduction

In January 1997, the NSLS signed a Memorandum of Understanding (MOU) with the newly established Advanced Polymers PRT (AP-PRT) to approve its proposal for instrumenting and operating a beamline at the NSLS. The mission of this PRT is to support continuing development and application of a dedicated facility at the NSLS for real-time simultaneous small- and wide-angle x-ray scattering (SWAX) experiments with emphasis on structural and dynamic studies of polymer materials. PRT members from academic institutions (SUNY at Stony Brook), the U.S. government (BNL-Physics, NIST) and industry (AlliedSignal, General Electric, Hoechst Celanese, Montell USA) are the constituent base for this facility and define the objective of this PRT. The expected date for the completion of the commissioning phase at X27C is June 30, 1997. During the initial operational phase (July 1, 1997 - June 30, 1998), 15% of the beamtime will be allocated to the General Users; during the normal operational phase (after July 1, 1998), 25% of the beamtime will be made available to the General User Program. This beamline is the first dedicated SWAX facility in the U.S., which aims to serve the partial needs of the polymer community and as a model for the future development of facilities with similar objectives.

Motivation

It has been well demonstrated that x-ray scattering and diffraction methods are extremely useful to characterize polymer structures and morphologies with dimensions from Angstroms to microns. In the past decade, the availability of synchrotron x-rays has further revolutionized the research opportunities in these areas. For one, the cycle time for the determination of structure and morphology can be significantly shortened. Typical study requiring a one-month period for an x-ray experiment by means of a conventional x-ray source can be completed in an afternoon at a synchrotron x-ray source. Furthermore, many dynamic experiments for investigating transient properties that are not possible before can be carried out by synchrotron x-rays in real time and/or *in-situ*. However, doing synchrotron experiments for polymer studies in the U.S. has always been difficult partly because almost all the accessible beamlines were designed to accommodate multi-purposed measurements, resulting in enormous efforts involving experienced personnel manpower, specialized equipment and sample chambers. Furthermore, additional beamtime allocation is needed for optics realignment in order to carry out a simple polymer scattering experiment. These burdens discourage most users (academic, government and industrial) who are not in the synchrotron field but want to carry out SAXS/WAXD experiments using synchrotron radiation.

In contrast, there are many dedicated x-ray scattering beamlines available in Europe (Beamlines A1, B2, BW4 at HASYLAB; W.8.2, W.16.1 at SRS; ID-2 and ID-13 at ESRF) and in Japan (Beamlines 10C and 15A at the Photon Factory), which are suitable for polymer studies. These facilities are operated by experienced personnel and are relatively efficient in terms of the experimental change over, and so have served their national polymer communities well. Many new research opportunities and important findings in polymers are coming out from these facilities.

As a result, the AP-PRT was formed near the end of 1996 to serve the U.S. polymer community by scientists from several academic, government and industrial institutions: B. Chu and B. Hsiao (SUNY-Stony Brook), E. Amis (NIST), D. Nguyen (BNL), P. Siddons (NSLS), Y. Gao (General Electric), S. Murthy (AlliedSignal, Inc.), R. Phillips (Montell USA) and C. Saw (Hoechst Celanese). The primary focus of this PRT is to investigate polymer structure, morphology and dynamics from atomic (1-20 Å) to nano/micro/scopic scales (20 - 1000 Å) in real time and/or *in-situ* using simultaneous SAXS/WAXD techniques. Some examples of these studies include characterizations of complex fluids (biological systems, block copolymers, colloidal systems, dendrimers, gels, hyperbranched polymers, ionomers); crystallization, melting and phase transition of polymers and blends; polymer chemical reactions; polymer melts and solutions during shear; fiber and film formation and deformation; and polymers under high pressure and supercritical environment; etc.

X-Ray Optics and Collimation

The x-ray optics at the X27C beamline are very simple. After the radiation is emitted from the bending magnet, a double-multilayer (silicon/tungsten) monochromator is used to monochromatize the incident beam. This monochromator, provided by Peter Siddons (NSLS R&D group), has been demonstrated to increase the x-ray flux by more than about 10 times when compared with the conventional double-crystal monochromator. Higher order harmonics are eliminated by slightly detuning the two multilayers. Although the wavelength is adjustable from 0.7-2.0Å (energy 6-20 keV) with this monochromator, we have optimized the flux and the SAXS angular resolution by tentatively fixing the wavelength at 1.3 Å. The corresponding energy resolution (DE/E) at this wavelength is 1.1%, which is more than an order of magnitude broader than that from the crystal monochromator. However, this energy resolution is sufficient for SAXS and WAXD measurements of polymer materials. A typical level of x-ray flux at the testing condition is about 9x10^11 photon/s.

The most critical component of any SAXS facility is its collimation system, which directly determines the maximum spatial resolution. At X27C, we have constructed a three pin-hole collimation system for this purpose, shown as a schematic diagram in **Figure 1**). This system is unique in several ways:

(1) The construction cost of the pin-hole system is significantly less than its slit counterpart.

(2) The alignment of the pin-hole system was found to be considerably easier than the slit system (since only half of the variables are needed to control the pin-hole system).

(3) Several series of pin-holes with different apertures were prepared (their diameters were derived from theoretical calculation), and the changing of different size pin-holes is straightforward.

(4) All pin-holes were manufactured on tantalum substrates (2.5 mm thickness) using a special drilling technique to minimize the parasitic scattering from the pin-hole edge. Using the first pin-hole of 0.1 mm diameter, we have routinely obtained the maximum spatial resolution of 100 nm from the duck tendon standard. (**Figure 2**)

SWAX Setup and Sample Chambers

Figure 1 illustrates the SWAX setup for measurements of isotropic systems (without preferred orientation, such as crystallization, melting and phase separation). In this case, two linear gas-filled delay-line position-sensitive detectors (PSDs), connected in series, are used to detect a wide angular range. SAXS/WAXD signals can cover ~1 mrad < q < 0.5 rad, this means that about 4 orders of magnitude in q can be obtained. At X27C, two types of data acquisition systems are available for the data collection: a PC based system for direct histogram memory access (Mbraun PSDs), and a CAMAC based system with its own histogram memory module (detectors by European Molecular Biological Laboratory, EMBL) which is also controlled by a PC. The second system has several advantages over the first one because it can tolerate a much higher count rate, simultaneously measure auxiliary experimental variables (beam intensity, temperature, stress ...), and share many same electronic components with the two dimensional (2D) gas-filled multi-wire proportional chamber (MWPC) detector.

Currently, the 2D SWAX setup for measurements of anisotropic systems (with preferred orientation, such as fiber drawing and polymers under shear) requires the usage of two image plates (IPs). The WAXD IP contains a central opening which allows the passage of the SAXS signal. This system is only applicable to systems in still conditions or under the steady state. For dynamic time-resolved measurements, we have to measure SAXS or WAXD images separately using a MWPC detector (made by EMBL) or a CCD x-ray camera. Although it is possible to use two area detectors simultaneously, we opt not to do so for the WAXD image collected this way is usually in a partially distorted form.

For the SAXS measurement, we have made an important modification to the beamstop which allows us to obtain the absolute invariant from the scattering images. In this modification, the beamstop is substituted by a small pin-diode which directly monitors the main beam intensity and therefore the sample absorption. The absolute invariant is thus attainable using the calibration of a secondary standard.

Several unique sample chambers are available in this PRT: (1) a dual-cell temperature jump apparatus (max. 400°C), (2) single-cell heating stages (max. 400°C), and (3) a small tensile stretching apparatus with temperature capability (max. 250°C). In the future, we intend to develop other sample chambers including a rheological cell and a fiber spinning apparatus.

The commissioning of the AP-PRT beamline at X27C was supported by NSLS, SUNY at Stony Brook, AP-PRT memberships, and grants from DOD (DURIP program) and NSF (GOALI program).

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Introduction

Michael Hart

NSLS Chairman

During the May shutdown the In Vacuum UNdulator (IVUN) was installed in the X-Ray Ring. Pioneering experiments three years ago at NSLS showed that the conventional design rules which required high electron energy machines such as the ESRF, APS and SPring-8 to produce x-rays from undulators could be changed; small gap undulators could be used with correspondingly lower electron energies. (See the November 1994 NSLS Newsletter article on the PSGU) Now, in a collaboration with SPring-8, we have installed IVUN, an undulator which has a period of 11 mm, a minimum gap of 3.3 mm and a magnetic field of 0.678 T in the straight at X-13. These parameters together with the ring energy of 2.584 GeV lead to the fundamental at 4.64 keV, second harmonic at 9.28 keV and third harmonic at 13.92 keV. At 2.8 GeV electron energy these increase by about 20%.

have been delayed by the confiscation of funds this year to aid work on the tritium remediation project at Brookhaven. Later this year, in the December shutdown, we expect to install a new superconducting wiggler in X-17. This insertion device is funded by DARPA and has been manufactured by Oxford Instruments. It will provide a number of different field configurations and will introduce new spectral profiles and running modes. The modern cryogenic system will improve reliability.

The NSLS "White Paper" last year outlined the oppThe elliptically polarized wiggler has now been developed to operate at 23 Hz without perturbing the source position at other beamlines, well on the way to the design frequency of 100 Hz. Programs to utilize the EPW in experiments at X-13 ortunities for installing IVUNs in the straights at X-9 and X-29 presently occupied by the four radio frequency cavities - given adequate investment there are now clear upgrade paths for new or replacement insertion devices on several straight sections. A further update is available in the paper presented by P.M. Stefan, S. Krinsky, C.C. Kao, G. Rakovsky, O. Singh, and L. Solomon at the 1997 Particle Accelerator Conference, Vancouver, B.C., Canada.

For us to take advantage of the new developments at the desirable rate of implementation will require strong scientific cases to the funding agencies. As soon as we have experimental data confirming reliable operation of these new insertion devices I will set up a Task Force to map our strategy and call for investment and potential PRT members to take advantage of the opportunity. For those of you with long memories and an interest in the history, the last such invitation [by Michael Knotek] was 12 years ago in the 1984 NSLS Activity Report and in the July 1984 issue of *Physics Today*.

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High Pressure and Synchrotron Radiation:

The New Era of Megabar Research

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The study of materials at ultrahigh pressures is currently experiencing an unprecedented surge of breakthroughs that were deemed inconceivable only a few years ago. We are now able to study phase diagrams, melting, and equations of state at well-controlled and well-determined *P-T* conditions to thousands of degrees at megabar pressures and to measure single-crystal elasticity tensors, elastic wave velocities, shear strength, and preferred orientation of materials at pressures equivalent to the Earth's core. We are entering a new era in which megabar experimentation will be freed from previous technical limitations and will be able to address a wide range of new scientific problems in physics, chemistry, material sciences, and planetary sciences. The new era has resulted from integrated advances in diamond cell, synchrotron radiation, and laser techniques. Many of these advances were originally developed at the NSLS, which has the unique advantage of being able to provide state-of-the-art facilities for two crucial diamond-cell techniques: synchrotron infrared spectroscopy and synchrotron x-ray diffraction. Effort is underway to further enhance the NSLS advantage by establishing a high-pressure center which includes a group of beamlines and on-site laboratories. Highlights of recent achievements and future plans are presented below.

High-Pressure Infrared Spectroscopy

Synchrotron IR measurements may provide the most diagnostic signature of the long-sought dissociation transition of hydrogen to a monatomic metallic state at low temperature and high pressure. The broad band source extending to the far-infrared is also ideal for directly measuring intraband electronic (Drude) excitations associated with metallization. Recently, we extended the infrared measurements of absorption and reflectivity of dense hydrogen to lower photon energies with the synchrotron infrared technique, and put significant new bounds on metallization in the high-pressure phase [1] . Combined IR and Raman measurements further provide in-depth understanding of the rich phase diagram of hydrogen above one megabar (**Figure 1**) [2] . The results suggest that although both hydrogen phases II and III are orientationally ordered, the ordering objects are angular momenta (quantum ordering) in phase II and molecular bond direction (classical ordering) in phase III [3] . The concept provides a natural explanation for the striking IR vibron intensity and vibron softening in phase III (**Figure 2**) [4] .

The behavior of H_2O-ice under pressure is of broad fundamental interest and is important for planetary science. Ice forms a number of closely related structures at low temperatures and moderate pressures. Each is characterized by hydrogen bonding that gives rise to many of the unusual properties of normal ice (I_h) and water. However, it was predicted over 25 years ago that under pressure ice should transform to a very different structure in which conventional hydrogen bonding is lost and the hydrogen atoms are symmetrical disposed between the oxygens (**Figure 3**). Infrared spectroscopy provides an ideal means in which to monitor the hydrogen bonds; at very high pressures an intense radiation source is required to observe the behavior. Recent synchrotron infrared measurements at beamline U2B have found that ice is indeed profoundly altered at very high pressures [5] . At 600 kilobars, synchrotron infrared reflectivity measurements show that the material transforms to a new structure that has the spectroscopic signature of the symmetric state (**Figure 3**). Moreover, the positions of the oxygen atoms were confirmed by synchrotron x-ray diffraction carried out at X17C. The new form of ice is stable to record pressure of 2.1 Mbars, where it is over four times denser than ice I_h. Under these conditions, ice is no longer a molecular solid but is best described as a dense ionic oxide. Very recent work has revealed additional new physics: the transition is associated with a soft phonon mode that undergoes a large number of interactions with other vibrational levels (Fermi resonances) before and after the transition to the symmetric structure [6].

We are currently upgrading this infrared beam line and instrumentation to make it an fully dedicated facility for high-pressure infrared spectroscopy. The facility will consist of a versatile high-pressure beam line capable of a broad range of measurements from the far-infrared to the visible spectrum at the U2A beamline. This new facility will give significantly higher IR brightness, particularly at long wavelengths, in comparison to the existing, partially dedicated beamline (U2B). A new FT-IR (Bruker 66v) is being installed at the beam line along with a recently completed high-pressure (long working distance) microscope as well as a commercial, high-magnification infrared microscope for micro-infrared measurements of samples at low-pressure . With the existing high-pressure x-ray facility at the NSLS, the new instrumentation will permit synchrotron IR, synchrotron x-ray, and other optical experiments on the same high-pressure samples. Systematic high-pressure measurements addressing a range of problems in condensed-matter physics and chemistry, Earth and planetary science, and materials science will be performed. These experiments include high-pressure studies of dense hydrogen and related planetary materials; minerals of the Earth's crust, mantle, and core; geochemical reactions; glasses and melts; surfaces and interfaces; whole-rock samples; and new high-pressure technological materials.

High-Pressure X-Ray Studies

Laser-heating with diamond cell samples provides the only means in the laboratory for creating temperatures of thousands of degrees at static megabar pressures which characterize the Earth's deep interior. *In situ* x-ray diffraction with laser-heated silicate [7] and iron [8-10] samples in diamond cells has been successfully obtained up to 3500 K and 1.3 Mbar at X17C, a high-energy wiggler beamline dedicated to diamond cell studies. To improve accuracy of these studies, however, requires a considerably more sophisticated facility. The Geophysical Laboratory of Carnegie Institution Washington has collaborated with the MRSEC of Arizona State University and GSECARS of the University of Chicago to develop such a facility at X17B1 which is free from the geometric handicaps of X17C and can implement recent advances in laser-heating, diamond cell, and synchrotron x-ray technology. Major progress in shaping and defining the temperature distribution in a laser-heated diamond cell sample has been made with a "double hot-plate" system which coordinates the optimal laser characteristics, sample configuration, and optical arrangement. With glancing-angle Kirkpatrick-Baez mirrors, the spatial resolution and sensitivity of the x-ray diffraction microprobe have also been greatly improved to enable precise measurements of a sample under uniform high pressure-temperature conditions. The X17B1 beamline is being reorganized to focus on high-pressure applications, with time allocations of 25% to the high-pressure multianvil PRT of SUNY Stony Brook, 25% to diamond cell PRT, 25% to X17B2, and 25% to General Users. Accurate determinations for phase diagrams and *P-V-T* equations of state of Earth materials can be made at X17B1 along the entire geotherm from the crust to the core. **Figure 4** shows the new high *P-T* phase diagram of iron based on x-ray diffraction observations.

The X17C beamline has long been a workhorse for diamond cell developments and experiments. Phase diagrams and equations of state of polycrystalline samples at uniform *P-T* conditions up to 1100 K and 1 Mbar have been obtained with x-ray diffraction of polycrystalline samples in resistively-heated diamond cell [12, 13] . Characterization of ultrahigh-pressure behavior and identification of new high-pressure phases of light elements or microscopic samples have been conducted routinely at X17C with polycrystalline [14] and single-crystal x-ray diffraction [15, 16] . Recent development of a novel diamond cell technique using high-strength beryllium as the sample gasket has opened a new frontier. The x-ray transparent Be gasket allows measurements of rheological properties, including preferred crystallographic orientation, shear strength, and single-crystal elasticity tensor [17] . Since these properties are directly comparable to seismic data, for the first time we are in the position of critically constraining models of Earth's core based on experimental measurements at megabar pressures. Moreover, detailed rheological studies of the elastic deformation of diamond anvils and plastic deformation of the sample and gasket provide valuable information for improving the diamond cell designs [18] . Due to the overwhelming attenuation of x-radiation below 12 keV by diamond anvils, megabar experiments have previously been limited to high energy sources. The use of Be gasket also extends the limit down to 5 keV and opens a vast area of megabar research, including high-energy resolution XFS, EXAFS, and inelastic scattering. These proposed studies, to be performed in collaboration with Dr. Chi-Chang Kao of the NSLS, hold great promise of revealing electronic and vibrational properties at ultrahigh pressures. In the energy range of 5-15 keV, focused white beam at X25 and X26C and monochromatic beam at X21 are superior than the high-energy beamline X17.

Megabar Facility Center at the NSLS

In ultrahigh-pressure research, the power of an integrated approach is far greater than the sum of individual techniques. Comprehensive understanding of high-pressure phenomena relies on the combination of all complementary measurements. It is most desirable, and often essential, to study the same sample at the same pressure and temperature with various spectroscopic and diffraction techniques. Above one megabar, diamond anvils break on releasing pressure. To perform a separate experiment for each type of measurement not only multiples the cost and time, but also introduces severe errors in data correlation. The NSLS is unique by virtue of its comprehensive synchrotron x-ray and IR capabilities, but it has lacked other basic diamond cell instrumentation needed for routine operation and analysis. To realize the full potential of the high-pressure advances, we are building a Megabar Facility Center at the NSLS with a fully integrated capability, including on-site laboratories for gasket and sample preparation, cryogenic and laser-heating methods, state-of-the-art optical calibration of temperature and pressure, and Raman and Brillouin spectroscopy. The laboratories will be located with easy access to both the x-ray and IR beamlines. After completion in 1998, the Center will be an international resource for users in the high-pressure field.

The commissioning of the AP-PRT beamline at X27C was supported by NSLS, SUNY at Stony Brook, AP-PRT memberships, and grants from DOD (DURIP program) and NSF (GOALI program).

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[1] R. J. Hemley, H. K. Mao, A. F. Goncharov, M. Hanfland and V. V. Struzhkin, *Phys. Rev. Lett.* **76**, 1667 (1996).

[2] A. F. Goncharov, I. I. Mazin, J. H. Eggert, R. J. Hemley and H. K. Mao, *Phys. Rev. Lett.* **75**, 2514 (1995).

[3] I. I. Mazin, R. J. Hemley, A. F. Goncharov, M. Hanfland and H. K. Mao, *Phys. Rev. Lett.* **78**, 1066 (1997).

[4] R. J. Hemley, I. I. Mazin, A. F. Goncharov and H. K. Mao, *Europhys. Lett.* **37**, 403 (1997)

[5] A. F. Goncharov, V. V. Struzhkin, M. Somayazulu, R. J. Hemley and H. K. Mao, *Science* **273**, 218 (1996).

[6] V. V. Struzhkin, A. F. Goncharov, R. J. Hemley and H. K. Mao, *Phys. Rev. Lett.*, **78**, 4446 (1997).

[7] C. Meade, H. K. Mao and J. Hu, *Science* **268**, 1743 (1995).

[8] S. K. Saxena, L. S. Dubrovinsky, P. Haggkvist, Y. Cerenius, G. Shen and H. K. Mao, *Science* **269**, 1703 (1995).

[9] C. S. Yoo, J. Akella, A. J. Campbell, H. K. Mao and R. J. Hemley, *Science* **270**, 1473 (1995).

[10] C. S. Yoo, J. Akella, A. J. Campbell, H. K. Mao and R. J. Hemley, *Science* **275**, 94 (1997).

[11] R. Boehler, *Nature* **363**, 534 (1993).

[12] Y. Fei and H. K. Mao, *Science* **266**, 1678 (1994).

[13] Y. Fei, C. T. Prewitt, H. K. Mao and C. M. Bertka, *Science* **268**, 1892 (1995).

[14] T. S. Duffy, R. J. Hemley and H. K. Mao, *Phys. Rev. Lett.* **74**, 1371 (1995).

[15] P. Loubeyre, R. LeToullec, D. Hausermann, M. Hanfland, R. J. Hemley, H. K. Mao and L. W. Finger, *Nature* **383**, 702 (1996).

[16] M. S. Somayazulu, L. W. Finger, R. J. Hemley and H. K. Mao, *Science* **271**, 1400 (1996)

[17] A. K. Singh, H. K. Mao, J. Shu and R. J. Hemley, *Phys. Rev. Lett.* submitted (1997).

[18] R. J. Hemley, H. K. Mao, G. Shen, J. Badro, P. Gillet, M. Hanfland and D. Häusermann, *Science*, **276**, 1242 (1997).

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ESH at the NSLS: A Very Serious Time

W. Thomlinson, NSLS
Associate Chairman for User Programs/Environment, Safety & Health

Since the inception of the NSLS, the Department has placed the safety of all users and staff at the very highest priority. That is clearly demonstrated by the great safety record that we, as a community, have amassed over the years. It has been accomplished with a lot of effort and proper distribution of financial and personnel resources. Now, as we all know, BNL is in serious trouble because of the tritium plume and the concurrent critical report to the Department of Energy by its Office of Oversight. The results are contained in the Integrated Safety Management Evaluation (ISME) of Brookhaven National Laboratory report. I would like to describe the NSLS situation with regard to those two major issues and how we will be reacting to the future ES&H (Environment, Safety and Health) changes that will be put into place at BNL.

Coming on the heels of our intensive Tier II Self- Assessment, the NSLS participation in the ISME was an exhausting trial for many of our dedicated staff. The ISME review took place over a period of two months, absorbing man-months of effort by the ES&H staff and others called upon by the review team. The NSLS was one of the primary facilities which was reviewed (the others being the AGS, HFBR, and Chemistry). We provided input through the release of many documents, long interviews with ISM staff, participation in numerous meetings and summary reviews with the ISME team. The intent of the review was to study how DOE Headquarters, the DOE Area Office at BNL, BNL management, and the departments met the criteria established for management of ES&H according to the ISM approach. As is common knowledge from the final report, DOE, the Area Office and BNL management did not fare very well. The contract to manage BNL is now out to bid and a shake-up of the organization for ES&H, starting with the DOE and continuing with line management through the BNL departments, has begun.

The NSLS is highlighted in that report through one primary example of poor ES&H practice, namely an incident in which a Plant Engineering worker was found to be working electrically hot at the NSLS without following proper procedures. The situation was complicated but basically arose through a misunderstanding between the NSLS and PE regarding supervision of employees. The finding was in fact a serious example of employees not having up to date training, not following applicable guidelines, and not having clearly defined supervision. This example was used throughout the report to illustrate many deficiencies of BNL ES&H management. One should not judge the review of the NSLS from that one issue. Although the most public document is the ISME Report, there is a second part of the review published as the Field Report. It is in that document that somewhat more detailed findings of the various departments are to be found. A careful reading of the Field Reports, in fact, shows that on balance the NSLS ES&H programs are functioning very well.

As in any critical report, even positive comments must be followed with "yes, but ....." examples. This report is no different. For example, the NSLS was given positive reviews for its conduct of operations by the operations staff, the review of experimental safety approval forms, beamline reviews and work control of major maintenance programs and operations. In each case, however, deficiencies were noted and we plan to work on them over the upcoming months. An example of deficiencies noted was the observation of an NSLS user handling liquid nitrogen without proper gloves and eye protection. Our program will respond immediately to those which we can easily integrate into the daily life of the NSLS without major policy changes. Many of the changes involve procedural and work control issues. Some will be transparent to users, but others may require some changes in the way our users operate at the NSLS. Major responses must await guidance from BNL with regard to the changing management, changing ES&H culture, and changing ES&H organization and operations. In any event, the user community will be kept informed of, and involved in, the process. In response to the tritium plume problem, the NSLS has also been directly affected. Our capital and ARAM budgets have been drastically reduced, preventing some of our planned ES&H and scientific upgrades from advancing. Nevertheless, we are committed to operating the machines and user programs in as safe a manner as possible. In another response to the plume, BNL has committed itself to a Review of Potential Environmental

Release Points from BNL Facilities in order to determine the existence of any unknown potential sources of ground water contamination. The NSLS, like all BNL departments and divisions, is heavily involved in that process. Being a young facility with mostly new buildings and few "historical relics", our task has been reasonably straightforward. However, we have taken it extremely seriously and have had the help of our technical, ES&H, and user staff and the Suffolk County Department of Health. This is a critical step for BNL in regaining public trust in our operations.

The NSLS accepts the findings of the ISME Report and will work with BNL and DOE to improve our own ESH programs. As always, we will strive to have the best possible ES&H program. Our mission of providing the highest quality operations of the facility for the user community will be compatible with that program. We are committed to achieving this goal while maintaining efficient integration of our scientific users into the NSLS research community.

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Microbeam Diffraction at the NSLS

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I.C. Noyan, *IBM Research Center*

Introduction

Recently, several groups at the NSLS have performed experiments using microbeam diffraction. Although one may think that third generation synchrotron sources with their high brilliance beams are necessary for meaningful results, we will give ample proof that microbeam diffraction using spots on the order of 5 to 20 mu m in diameter is quite feasible and is an important part of several research programs centered at the NSLS.

Microbeams are used at the NSLS for various types of measurements, such as fluorescence microprobe (X26A), soft x-ray imaging and spectroscopy (X1A), coherent (speckle) diffraction (X25, X20C), high-pressure energy-dispersive-diffraction using diamond anvil cells (X17B1, X17C), and microbeam diffraction (X3A2, X17C, X16C, X26C). This article will deal with only the last of these areas. It will discuss the issues surrounding the design of microbeam diffraction instrumentation and describe results from several recent experiments. The range of styles of these experiments illustrates the number of different approaches that can be taken. The technique itself has many uses, such as identification of phases or atomic composition in mixtures made up of several domains of different structures, study of inclusions or defects or other types of heterogenous structures, determination of variations in strain at different locations in a small feature in a patterned device, etc. With very high flux rates, the variation of structure or strain in real time, as a function of temperature or current or some other variable, is sometimes feasible. At an NSLS bending magnet beamline, with a ring current of 300mA, a crude estimate of the diffracted signal from a 1mu m cube of aluminum at 10m from the source with no focusing yields an integrated count rate of between 1 and 10 photons/sec. This is clearly the limit of experimental feasibility, but two conditions can make this sort of experiment possible. Relaxing the volume of scattering material to a cube 5 microns on a side yields a 125 times greater counting rate. Alternatively, a Kirkpatrick-Baez focusing mirror pair, tapered capillary, or other concentrating optics collecting 100 microns x 100 microns of incident radiation onto a square micron aluminum grain would also increase the count rate to acceptable levels.

The challenge in microbeam diffraction lies not merely in the production of an x-ray beam having dimensions of sub-micron to tens of microns, but in delivering the beam to an identified region of interest on the sample, keeping it there during the diffraction experiment, getting the diffraction information out (energy and/or direction of the diffracted beams), and properly interpreting the data. The area of interest on a sample can be identified by optical or spectroscopic means. Optical microscopy has limitations of about 0.5 micron because of the wavelength of visible light. Spectroscopic identification of the target feature or nearby fluorescence markers is in some ways easier because the same x-ray beam is used for locating the feature and doing the diffraction experiment. In some cases identification techniques and feedback mechanisms are required to keep the area of interest in the x-ray beam, or to keep the x-ray beam in the area of interest. At the same time, thermal and mechanical stability of the entire system (beam-conditioning and sample positioning) is of paramount importance. If the sample is supported one meter above a table, just a 2º C change in temperature can cause a 2 micron displacement in the position of the sample.

A variety of techniques exist for making an x-ray beam of small dimensions. There is a trade-off in flux gain versus beam divergence, so that it is an advantage to start with the smallest combined source size and beam divergence (brilliance) possible. With slits and pinholes, alignment can be simple, white or monochromatic radiation can be used, and beam divergence is not increased, so that the sample can sit several centimeters away. However, there is also no gain in flux since the beam is not concentrated. Single tapered glass capillaries currently hold the record for smallest focused hard x-ray beam (0.1 micron)[1] and can have a flux gain of 10-1000X. Because of their concentrating power, they produce a highly convergent beam (about 2.5mr for 1Å x-rays in borosilicate glass) and must be placed very close to the sample. The intensity distribution within the microbeam depends upon the detailed capillary shape and the divergence is dependent on that as well as the critical angle. Capillaries can be used to focus either white or monochromatic radiation. They have been used for microbeam diffraction experiments by Columbia University and IBM researchers at Beamline X26C. Kirkpatrick-Baez mirrors provide another means for focusing white or monochromatic x-rays. Alignment and adjustment are critical, and like capillaries, these optics introduce horizontal and vertical beam divergence. Advantages over other microfocusing optics are their combined achromaticity and relatively long (10cm) working distance. These have been tested at X17C and X26C by visitors from University of Chicago (Geo-CARS)[2] and used by Bell Laboratories scientists at X16C. Fresnel zone plates, used on Beamline X1A for soft x-ray imaging experiments, also are used to concentrate beams, and are limited by the narrowest width (and highest aspect ratio) of the outermost ring that can be made by lithography[3,4]. The focal length of zone plates depends upon the x-ray wavelength and thus they are useful only as monochromatic sources. Bragg diffraction combined with Fresnel focusing yields higher efficiency flux concentration in Bragg-Fresnel optics. These hold much promise, but involve complicated, state-of-the-art fabrication techniques[5]. Again, they are useful only for monochromatic experiments, and have an energy range from about 6-30keV.

There are a number of methods for obtaining diffraction information in a microbeam experiment. To specify a point (h,k,l) in reciprocal space, three parameters are needed: the magnitude of momentum transfer, (q=4pisin(2theta/2)/lambda) and two angles of sample orientation. Monochromatic radiation has the advantage that the energy is known accurately, and once diffraction angles are determined, accurate lattice spacings can be calculated. A disadvantage is that there are far fewer reflections to detect, and the sample always has to be rotated to bring any of them into the Bragg condition. However, there is a great advantage in not having to rotate the sample, since the concentricity of circles ("sphere of confusion") on a well-aligned diffractometer is no better than 20 microns unless it is specially designed. Another complication that arises from sample rotation is the change in illuminated area at various tilts. If white beam radiation is used, it is not necessary to rotate the sample in order to set up diffraction conditions for different crystalline grains or for different sets of planes in a single grain. In this case, two of the three parameters needed can be determined from the peak position on an area detector, typically a CCD camera. The third parameter, the photon energy, can be determined by scanning the incident photon energy.

An alternative way to determine the photon energy is to energy analyze the outgoing photons for a given Laue spot. This is possible if one has a detector arm with 2 degrees of freedom that allows mounting of optical elements, such as beam collimation that defines the scattering volume, this is not easily done with a CCD. The Columbia group uses an energy-dispersive detector [Si(Li) or Ge] mounted on such a detector arm, but with a restricted range of motion for one of the angles. The Bell group has used a single crystal analyzer mounted on such a detector arm with a scintillation detector. Linear or two-dimensional detectors can add great efficiency to the search of the Ewald sphere for the scarce diffracted beams, and they may be essential for determining orientations of individual grains and for locating Laue peaks to be scanned with an energy- or wavelength-dispersive detector. In a microbeam experiment, the signals can be so small that a detector having a large dynamic range is essential, as well as good shielding, both electronically and physically. Reduction of background signal is imperative and can be a challenge in white-beam hutches.

The interpretation of a microbeam diffraction experiment can also be quite challenging. The local characteristics of the sample dominate the diffraction profile. If the irradiated volume is greater than the size of a single grain, assumptions must be made about how many grains contribute to the observed diffraction and whether they have identical behavior and characteristics. For micro-structural phase determinations, it is crucial that the illuminated volume (including penetration depth) be no more than the size of the phase domain. This can be difficult when using white or high energy monochromatic incident beams.

The following sections give a brief desciption of some of the results from microbeam diffraction research projects at the NSLS.

In-situ Identification of Natural Diamond Inclusions

White synchrotron beam was used at X17C by P.G. Conrad, R.J. Hemley, H.K. Mao, L.W. Finger, J. Hu, and J. Shu at Carnegie Institute of Washington, B. Harte, and M. Hutchison at University of Edinburgh to obtain energy-dispersive x-ray diffraction from natural diamond inclusions[6]. The Brazilian samples are of geophysical importance because they are thought to have originated from the Earth's lower mantle. A 11x15 micron beam was produced by Kirkpatrick-Baez mirrors. The samples were mounted on a chi circle, and a search for single-crystal peaks was made by scanning omega from -20 to 45 degrees, and chi from 0 to 180 degrees. Once an orientation matrix for the diamond was determined, it was used as an aid for identifying peaks unique to the inclusions. Searches have revealed a range of crystalline character for the inclusions, from polycrystalline to single crystal to poorly crystallized. In one diamond, five inclusions have been identified. Three predominately exhibit diffraction lines of (Mg, Fe)O with a Mg/Mg+Fe ratio averaging 0.76. Two of the inclusions are CPX with a few lines of (Mg, Fe)O.

Kinetics of Absorption-induced Transition in Pd-(H,D)

E.F. Skelton, P.L. Hagans, S.B. Qadri, and D.D. Dominguez from NRL and J.Z. Hu from Carnegie Intitute of Washington have recently used 60+ keV photons from X17C to measure the *alpha*-to-*beta* structural phase transition in Pd-(H,D), induced by the electrochemical absorption of deuterium[7]. A spot size of around 10mm was used to illuminate $18x10^{-12}$ m^3 of a 1mm diameter Pd wire, and the Pd(422) peak was monitored as a function of position and time from the edge to the core of the wire. Under an electrochemical loading current of about 100ma, the *alpha*-to-*beta* transition moved from the edge to the core at 48 nm/sec. The alpha phase was also found to persist about 30% longer in the core than on the surface.

Skelton and coworkers have also used microbeam diffraction to measure stress in sub-micron diameter single crystal bismuth filaments[8] and structural inhomogeneities in $YBa_2Cu_3O_{7-delta}$ and $Nd_{2-x}Ce_xCuO_{4-y}$ high T_c superconductors[9]. In the superconductor work, small changes in the lattice were found over a spatial scale of 10 microns.

Strain Measurements on Aluminum Conductor Lines

P.-C. Wang and G.S. Cargill III (Columbia Univ.), and I.C. Noyan, E.G. Liniger, C.-K. Hu, K.Y. Lee (IBM) have used white beam Laue diffraction on beamline X26C with a Si(Li) detector for strain measurements on passivated aluminum conductor lines with 10 mu m spatial resolution[10]. Real-time measurements were made of strains developed during electromigration using a symmetrical reflection geometry to take advantage of the (111) fiber texture of the aluminum films. On-chip tungsten pads were used as "internal" diffraction standards to correct for angular or energy calibration shifts during the experiments. By measuring differences in d-spacings with time at many locations along 200 micron long lines, the strains developed during electromigration could be measured with greater precision than the absolute d-spacings. The precision of the individual d-spacings measurements was limited by uncertainties in diffraction angles, since only a few grains diffracted into the detector aperture at each location along the line. An example of the d-spacing changes at different locations along a conductor line caused by passing $1.4x10^5$ A/cm^2 through the line at 260ºC for nine hours is shown in **Figure 1**, together with the biaxial stresses in grain boundaries deduced from the d-spacings. Values of the stress gradients, *e.g.* the slope of the dashed line in **Figure 1**, were determined for different current densities and times, and these are shown as data points in **Figure 2**, together with results from model calculations which are shown as lines for three different values assumed for the effective diffusion coefficient D_{eff}. Good agreement is obtained for the intermediate value of D_{eff}. Electromigration-induced strain measurements are continuing for other line lengths and widths, for other conductor line metals and alloys, and for other passivations.

Using a KB mirror pair designed by A.A. MacDowell producing a spot size of 3x30 mu m, the Bell group at

X16C has obtained count rates of 300 photons/sec from a 1 mm wide wire of Al aligned with the narrow beam direction across the wire. Typically grain sizes were of the order of the wire dimensions. Also on a blanket film, they have demonstrated the ability to locate four peaks consistent with a single grain, without moving the sample, enabling the extraction of the full 3-D strain information.

Strain, Period and Composition of Patterned Multi-Quantum Well Lasers

Using an x-ray microprobe, K. Evans-Lutterodt, E.D. Isaacs, M.A. Marcus, A.A. Macdowell, W. Lehnert, C.-Y. Kim, and H.K. Kao, (Bell Laboratories), have demonstrated the ability to map strain and quantum well period of selective area growth structures, with a spatial resolution of 3mu m[11]. Information from this technique has been used to enhance the design of an InP-based electroabsorption modulated laser, which has a data transmission rate of 2.5 Gbit/sec.

With the technique of Selective Area Growth (SAG), local perturbations of materials properties are achieved by patterning features on the substrate, and then proceding with the crystal growth in exactly the same fashion as for un-patterned wafers. The patterned features are typically SiO_2 pads that affect the local concentration at the growing surface, hence modifying the growth locally. This results in a localized modification of the quantum-well thickness and consequently the local band gap, and enables monolithic integration of lasers and electro-absorption modulators. For the electro-absorption modulated laser, a typical structure is a pair of SiO_2 pads, separated by 20 microns, that define the laser region, as shown in **Figure 3a**. The quantum-well spacing and strain in between the pads will be substantially different from a region far away from the pads, with a resulting bandgap that allows electrical modulation of transmitted laser intensity.

Characterizing SAG structures with standard, large spot-size x-ray diffraction presents special difficulties because materials properties such as quantum-well spacing vary significantly over length scales as short as 10 microns. What is needed is to be able to perform x-ray diffraction with small spot sizes, on the order of 20 microns. **Figure 3a** shows typical dimensions of the oxide pads, and shows the physical locations of the x-ray scans in **Figures 3b and 3c**. The data in **Figures 3b and 3c** have an x axis which is percentage mismatch relative to the InP (004) substrate peak, which is the strong peak at 0% mismatch. **Figure 3b** shows a specular reflectivity scan for a region far away from the pads, as indicated. **Figure 3c** shows a similar scan that was taken in between the pads. The quantum-well thickness is extracted from the spacing between the satellite peaks, and the strain can be deduced from the centroid of the envelope of satellite intensities. As can be seen from the data, there is a substantial difference between the two regions, and by studying these differences as a function of the dimension W in **Figure 3a**, one improves in understanding the SAG process. This problem is well suited to the NSLS because the volume of material probed is of the order 20x20x0.1micron, and by scaling the crude estimate made earlier, the signal is clearly above background, even with a simple pinhole for the optics, as observed experimentally.

References

[1] D.H. Bilderback, S.A. Hoffman, and D.J. Thiel, *Science*, **263**, 201 (1994).

[2] P.J. Eng, M.L. Rivers, B.X. Yang, and W. Schildkamp, SPIE Proceedings, **2516**, 41 (1995).

[3] P.S. Charlambous, P.A. Anastasi, R.E. Burge, K. Popova, SPIE Proceedings, **2516**, 2 (1995).

[4] A.A. Krasnoperova, Z. Chen, F. Cerrina, E. Difabrizio, M. Gentili, W. Yun, B.P. Lai, E.S. Gluskin, SPIE Proceedings, **2516**, 15 (1995).

[5] A.A. Snigirev, and V. Kohn, SPIE Proceedings, **2516**, 27 (1995).

[6] P.G. Conrad, R.J. Hemley, H.K. Mao, L.W. Finger, J. Hu, J. Shu, B. Harte, and M. Hutchison, *NSLS 1996 Activity Report*, (1997).

[7] E. Skelton, P.L. Hagans, S.B. Qadri, D.D. Dominguez, and J.Z. Hu, *NSLS 1996 Activity Report*, B-150 (1997).

[8] E. Skelton, J.D. Ayers, S.B. Qadri, N.E. Moulton, K.P. Cooper, L.W. Finger, H.K. Mao, and Z. Hu, *Science*, **253**, 1123 (1991).

[9] E. Skelton, A.R. Drews, M.S. Osofsky, S.B. Qadri, J.Z. Hu, T.A. Vanderah, J.L. Peng, and R.L. Greene, *Science*, **263**, 1416 (1994).

[10] P.-C. Wang, G.S. Cargill III, I.C. Noyan, E.G. Liniger, C.-K. Hu, and K.Y. Lee, "X-ray Microdiffraction for VLSI," MRS Symp. Proc., **427**, 35 (1996).

[11] E.D. Isaacs, K. Evans-Lutterodt, M.A. Marcus, A.A. Macdowell, W.J. Lehnert, J. Vandenberg, S.Sputz, J.E. Johnson, J.A. Grenko, L.J.P. Ketelsen, C.Pinzone, R.Glew, W.Yun, Z.Cai, W.Rodrigues, H.-R. Lee, and B.Lai , Proc. Electrochem., Soc., submitted (1997).

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Focus on .... Long- Range R&D Group

Jerry Hastings and *Erik Johnson*

Brookhaven National Laboratory has played an important role in accelerator based photon source development and utilization for almost 30 years now. The dawn of modern high brightness synchrotron light sources can arguably be marked by the development of the storage ring lattice design of Ken Greene and Rena Chasman which is the basis for all such machines, starting with the NSLS. The driving interest and common thread behind all the subsequent efforts at the NSLS has been to provide the best possible tools for conducting research based on the light these sources generate. This tradition persists with insertion device development on the NSLS storage rings and the program at the Source Development Laboratory to develop a sub-harmonically seeded free electron laser source for UV radiation down to 100 nm and below. As the state of the art continues to advance, the need to integrate the source, optics and experiment have become ever more evident. In 1996 Michael Hart formed the Long-Range R&D (LRRD) group from members of the long standing Beamline R&D section with Jerry Hastings as its head. The principle function of the LRRD group is to work closely with other sections in the NSLS organization, other departments at BNL, and outside user interests in developing and reducing to practice state of the art accelerator based radiation sources for research.

The seed of this activity was planted ten years earlier in 1986, when the NSLS staff formed an insertion device team to utilize the X13 straight section for research and development in novel insertion devices and insertion device beamline components. The earliest version of the beamline used the "Mini-Undulator" (the prototype for the X1 soft x-ray undulator) as its source, and provided a test-bed for instruments and experiments developed by the NSLS, and for other facilities. Both the Advanced Light Source at LBNL and the Advanced Photon Source at ANL incorporate instruments first developed and tested at X13 including most recently the "Elliptically Polarized Wiggler", developed in collaboration with the APS and the Budker Institute of Nuclear Physics. This device and the science it could potentially support garnered a 1996 Scientific Facilities Initiative Award for John Sutherland and his colleagues. A new monochromator is being constructed and experiments are planned which exploit the novel properties of the EPW to push the limits of polarization sensitive measurements.

From the inception of the insertion device team, it was realized that the specific properties of the NSLS X-Ray Ring lattice, in particular the very small vertical beam size, could be exploited to study and implement small gap magnetic devices. Reduction of the spacing or gap between the magnetic elements in an insertion device permits a similar reduction in the length of the magnetic period. This in turn opens the possibility of a hard x-ray undulator, with all of its potential advantages on the X-Ray Ring. The NSLS first demonstrated the viability of this concept with the prototype small gap undulator (PSGU) project that was headed by Peter Stefan.

The PSGU was designed to examine in detail the interaction between a small gap magnet and the normal operations of the NSLS storage ring. To do so, the magnet is outside the NSLS vacuum enclosure and both a variable gap vacuum chamber and variable gap magnet system were provided. This permitted independent studies of the effects of small, extended apertures and small magnetic gaps on electron beam in the X-Ray Ring. The concept indeed proved successful, providing first harmonic undulator radiation at 5Å. Based on these results, an R&D project was initiated to build an in-vacuum undulator (IVUN) that would eliminate the `wasted' space taken by the variable gap vacuum chamber. By doing so, both the magnetic gap and period could be reduced still further, resulting in a higher energy fundamental for the undulator. This project is a collaboration with the SPring-8 project in Japan. The Japanese have pioneered in-vacuum undulator technology and are responsible for the development of the magnetic arrays in IVUN. The NSLS effort for the remainder of the system and system responsibility is again headed by Peter Stefan.

This device, operating at a magnet gap of 3.3 mm is calculated to produce first harmonic radiation at 2.7 Å wavelength. Installation of the device was completed during the May 1997 shutdown and commissioning studies will begin over the next several months. If all proceeds apace, this device will have an important impact on insertion device beamlines at the NSLS. Of the eight straight sections on the X-Ray Ring, at present only five are available for insertion devices. The other three are occupied by injection and RF cavities. Because of the short period of IVUN, the overall length of the magnet is also short (less than 1 meter). In fact, short enough to fit between the RF cavities in the X9 and X29 straight sections. A successful IVUN will therefore make available two additional insertion device beamlines to the user community with substantially improved brightness over our current hard x-ray devices.

Free Electron Lasers (FEL's) are now widely considered to be the next major step in accelerator based photon sources. This view, held at many laboratories, owes much to the theoretical and experimental work of NSLS physicists stretching back more than a decade. Of particular note are the development of high current - high brightness electron beam technologies at the Accelerator Test Facility, and the theoretical framework for high gain FELs and subharmonically seeded single-pass FELs. To capitalize on these developments and bring them nearer to user applications, the NSLS founded the Source Development Laboratory (SDL) with Erik Johnson as the project manager. The objective of the SDL is to facilitate the coordinated development of sources and experiments to produce and utilize coherent sub-picosecond synchrotron radiation.

The centerpiece of current SDL activities is an ultra-violet FEL (UV-FEL) which will operate to wavelengths below 100 nm, and will produce radiation with substantially shorter pulses than are available from the UV ring (as brief as ***5*** ***fs***!) with peak power ranging from hundreds of Megawatts to as high as 100 Gigawatts for the shortest pulses. The bandwidth of the FEL can to some extent be controlled within the constraints of the Fourier transform limited nature of the source (trading pulse length for bandwidth). In terms of peak power, this source will open territory completely uncharted by existing tunable sources. The SDL will be a site for `proof-of-principle' studies providing a new tool for the study of nonlinear and ultrafast phenomena. The actual time table for the SDL is very sensitive to the available resources, but there is no lack of determination on the part of the NSLS staff to make it part of the *Long-Range* future for the NSLS community.

References and Further Reading

These projects, and the related Department R&D are described in overview form in the most recent NSLS Activity Report (1996) BNL 52517.

More detailed information can be obtained from the following publications and references therein: "Lighting the Way to the Future: An Anthology of Improvements, Developments, and Research by NSLS Staff and Collaborators", (also located in Activity Report 1996, "Research Anthology" chapter is a PDF file.)BNL 63477, and "Lepton Accelerators and Radiation Sources: R&D Investment at BNL" BNL 64214.

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VUV Ring Report

Stephen Kramer

VUV Ring Manager

A major upgrade during the Winter 96-97 shutdown of the VUV Ring included the replacement of one of the dipole magnet beam chambers to accommodate a new infrared beam port. The conventional U2 port which had been used with the infrared microscope has been replaced with a larger aperture (100H X 48V mrad.) U2IR port. This new port will provide greater flux for this diffraction limited infrared beamline, complementing the other three high flux infrared beam ports on the VUV Ring. U2IR will not only reduce the backlog of infrared users, but create an opportunity for greater user investment in new infrared experimental facilities.

The vacuum chamber replacement, although simple in principal, was a major reconstruction effort. Based on the 95-96 shutdown experience, we knew it required a two-month shutdown and careful planning by the Mechanical and Vacuum Groups. Overall, the chamber replacement required: the removal of massive amounts of lead and concrete shielding, removal of one quadrupole, one sextupole and two trim magnets, the installation of one new beam position monitor electrodes and the installation of the photon beam defining apertures for two beam ports (one new and one existing port). The VUV Ring had been designed with a minimum of radiation shielding; additional shielding was installed as the loss points were identified, but this made access to the Ring components more difficult. Adding to the complexity this time was a leak in the ceramic-to-metal braze joint in the bump magnet, just downstream from the dipole chamber that was being replaced. Luckily this slow leak was discovered in October 1996, giving just enough time to plan for its replacement. All of this, plus the existing dipole magnets, had to be realigned to better than a 0.2 mm resolution. The trickiest part of the whole operation was the *in situ* welding of the two new chambers (the dipole and ceramic gap) to the existing vacuum chamber by machinists from the Central Shops and the NSLS Mechanical Group. These four welds had to be vacuum tight and remain tight during the bake out and subsequent beam loading.

Turn on of the ring went very rapidly and with no real surprises. The ring was back under vacuum by January 3,1997 and stored beam was established by 23:00 on January 16, 1997. Two Amp-hrs of integrated current were accumulated by 17:30 the next day, at least four days ahead of schedule, in order to allow conditioning of the vacuum chamber with beam over the three day (holiday) weekend. Within a week, 100 Amp-hrs had been accumulated and the lifetime was back to within 20% of the pre-shutdown value. By Monday January 26th, the lifetime was within 10% of the pre-shutdown value and the VUV Ring was declared operational, one day before the scheduled conditional operations and a week before scheduled operations. Two additional injections per night were added until February 10th in order to enhance the gas desorption and cleanup of the vacuum chamber. During the re-shielding process in this section, additional shielding material was added below the U1/U2 dipole magnet. This has helped reduce the radiation levels on the VUV floor during injection.

Since the VUV Ring has been back in operation, the major improvement effort has been to reduce the phase noise on the beam. Adding an RF feedback system to the 52 MHz RF amplifier has had some success, but the stability required more work than anticipated since the 4th harmonic system may need a similar feedback in order for the entire system to be stable. During the spring shutdown, work on the 52 MHz RF cavity will help reduce the impedance of some of the higher order modes in that cavity and allow higher cavity power operation by reducing the coupling of some higher order mode suppression antennae to the 52 MHz field in the cavity.

The plan to run pump-probe experiments on some of the infrared beamlines using short pulse laser beams, mode locked to the synchrotron beam, will require extensive operation of the VUV Ring with short beam bunches. The desired bunch length is about 250 psec FWHM, but this short bunch is not possible even at low current with the present ring lattice. To achieve this desired bunch length the VUV Ring would have to operate at an energy of 550 MeV, resulting in a lifetime decrease by a factor of 15 to 20, if the present lattice is maintained. A new VUV Ring lattice which provides greater longitudinal focusing would allow operation at the 800 MeV with shorter bunches, but with lifetimes closer to the normal operating conditions. The lattice would reduce the momentum compaction factor of the beam by giving up the achromatic feature of the Green-Chasman lattice. Not only would this change reduce the bunch length of the beam, but it would increase the horizontal emittance of the beam making the horizontal beam size larger and therefore increasing the lifetime relative to the smaller horizontal beam size lattice. As current is added to the bunch, the bunch length increases naturally for both lattices. By reversing the effect of the 4th harmonic RF cavity and increasing the longitudinal focusing on the beam, the bunch length could instead be decreased. Since the shorter bunches may tend to have less stability than the longer bunches due to the higher frequency components of the beam, it will be quite a challenge for the RF Group.

A test of such a new lattice has been performed at low current per bunch and **Figure 1** shows the shorter bunch length of 256 psec FWHM compared to the bunch length of 400 psec for the normal (achromatic) lattice. The new lattice without the 4th harmonic cavity countering the natural bunch lengthening was tested in April and May 1997. At 500 mA in 7 bunches, the lifetime for this new lattice decreased by less than a factor of two compared to the lifetime in normal operations (4th harmonic cavity stretching the bunch length to 1.5 to 2 nsec FWHM). The horizontal emittance increased by about a factor of 2.5 and a similar increase in the vertical emittance was measured, since the linear coupling was held constant. If the natural bunch lengthening with increasing current can be fully countered by the 4th harmonic RF system, then an additional reduction in lifetime of about a factor of three will result, for a net reduction of a factor of less than six in lifetime.

The above net reduction in lifetime is considerably less than the factor of 15 to 20 loss for 550 MeV operation with the achromatic lattice. This should reduce the conflict between the timing users and the rest of the VUV users. However, the ultimate current that can be stored in the beam with this lattice will depend on the heating of the ceramic gaps in the ring by the short bunches. This will have to be measured once the 4th harmonic system is working to keep the bunches short. Also needing to be studied will be the impact of the increased emittance for the dipole and undulator beamlines and the effect of dispersion in the undulators on these beamlines and their ability to tune during operations. The availability of this new more compatible lattice will help to unite these divergent user needs and should help strengthen the VUV user community as a whole, by allowing new experiments to take place without turning off the present strong user program for significant periods.

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X-Ray Ring Status

Roger Klaffky

X-Ray Ring Manager

The extensive electrical and mechanical work completed during the December 1996 shutdown was described in the March 1997 NSLS Newsletter. Operations commenced on the X-Ray Ring on January 23. On February 1, there developed a water-to-vacuum leak in the RF1 cavity, causing the cavity pressure to jump from the 10^{-10} Torr range to the mid 10^{-07} Torr range. Helium leak detection failed to identify which water-cooled component was leaking. At an emergency user meeting the decision was made to replace the RF coupling loop, the most likely defective component, during the February 10 and 11 studies period and to continue operating at 250 mA until that time using the other three RF cavities. The repair was carried out February 10, followed by RF and beam conditioning on February 11. Operations resumed on schedule at noon on February 12. By February 15, conditioning with operational beam had lowered the cavity pressure to the 10^{-09} Torr range.

After an initial commissioning period in January, the new dual transmitter on the RF2 cavity became operational. At present each of the transmitters is operating at half power (50 kW) with increased reliability. During the December 1997 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 more than 150 kW into the cavity. This additional power will enable reliable 438 mA operation at 2.584 GeV or 250 mA operation at 2.8 GeV. Improved windows for the other RF systems are presently on order. The new cavity has several desirable features. There are no welded ports or joints, cooling channels are machined in the outside skin, and there is an improved mushroom cooling. The cavity will be shipped to the NSLS in July, after an acceptance test in Germany.

In February, an effort to fully characterize the beamline shielding requirements at 2.8 GeV began. Studies periods were utilized for radiation surveys by S&EP personnel to record in detail which beamline components and hutch walls require additional shielding. The initial surveys were completed in April so that users could make use of the May shutdown period to install shielding. A 21,000 pound shipment of (3'x5'x1/16") sheets was delivered early in May, and a work area was set up outside the NSLS Stockroom for cutting the lead sheet. Procedures for handling the lead and discarding lead scraps, contaminated covering material, gloves and aprons were outlined in the April 16 NSLS ES&H Highlights. Users are encouraged to begin their shielding effort early because obtaining final operational approval of the shielding is an iterative process requiring a series of radiation surveys followed by shielding efforts. There will be radiation survey time during studies periods. In the Fall, beamlines which have received shielding approval will be able to operate during the two designated 2.8 GeV weeks in September and November. Radiation surveys will be scheduled during these periods on lines still in the process of adding shielding.

The major X-Ray Ring task during the May shutdown was the removal of the PSGU from the X13 straight and the installation of the IVUN(In-Vacuum UNdulator) which is a short-period magnet array (30.5 periods, with an 11 mm period length) developed at SPring-8. The associated vacuum chamber and mechanical systems were developed at the NSLS. IVUN is designed to produce 4.6 keV radiation in the fundamental, at a magnet gap of 3.3 mm, with useful photon fluxes in both the 2nd and 3rd harmonics. The magnet gap is adjustable between 2 mm and 10 mm.

Several other shutdown tasks were completed. Upgraded beryllium windows were installed on the X4C and X6B beamlines. The remaining 13 upgraded beamline windows required for high current (438 mA) operation will be installed during maintenance periods and the December 1997 shutdown. Safety metering transformers were installed on 480 Volt RF and magnet power supplies to verify isolation from source power before carrying out repair work. Also, the full cooling capability of the NSLS high pressure and low pressure copper water systems was restored after heat exchangers for these systems were cleaned out. Finally, the helium cryotransfer line for the X17 superconducting wiggler was replaced, allowing the X17 beamlines to resume operation on June 9. A leaking transfer line had shut these lines down since the middle of April.

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A User's Perspective

Joel D. Brock

Cornell University

UEC Chair

Everything is new again. Spring is here. The UEC always changes at this time of year - at every annual meeting, a new UEC is formed, a new chair takes over, and the cycle continues. But this year is completely different. Everything has changed.

In a move aimed at restoring the public trust from the citizens of Long Island, Secretary of Energy Federico Pena recently terminated DOE's contract with Associated Universities, Inc., the nonprofit organization that has managed BNL for its entire 50 years. The specific issues driving this unusual action were clearly the environmental concerns at BNL. However, BNL's "university atmosphere" was blamed in a DOE safety report for not providing the level of "discipline and control" needed to ensure the protection of the public, the workers and the environment. This suggests that still bigger changes may be on the horizon. The new contractor may well interpret this as a call for a change in the entire character of BNL.

How will all this affect NSLS users? At this point, we simply don't know. To be sure, there will be changes in the way the NSLS does business. Phrases such as the need for "a complete change of culture" have crept into the vocabulary of BNL's management. Two examples from the DOE safety report illustrate the issue. The first example is the complaint that "researchers, who typically have the least ES&H experience, receive less than two hours [of ES&H] training annually." The second describes a safety violation at the NSLS wherein a Plant Engineering electrician was working near an energized panel at the NSLS. Detailed procedures had not been followed and the training and approvals were out of date. From the nature of these examples we can clearly expect increases in the time we all spend in safety training.

In the broader context, we should expect a reorganization of the laboratory management. We don't know anything about what the new management structure might look like yet. But change is not necessarily good or bad. It is frequently unsettling, but there is tremendous opportunity during times of change. I believe that we, the users, should approach the current situation with an open mind and a willingness to try to make things work. In every instance, we must act positively to make the new system as safe and as efficient as possible. Constructive suggestions for improvements in the system are far more valuable than criticisms of the old system. We need to be particularly careful not to take shots at each other. This is not the time for the NSLS Administration to crack down on the users and make them toe the line. Similarly, this is not the time for users to go after the Administration and make demands for additional services. This is the time to come together and work in concert to build the best possible future we can for the NSLS and BNL.

So how do we do this? I believe that there are two roots to the problems facing the NSLS today. These issues are certainly not unique to the NSLS. All branches of science currently face the same issues in some form. The issues are (i) the current constraints on federal funding of science, and (ii) poor communications with our neighbors. Both issues can be dealt with in a straight-forward fashion. The solutions, however, will require significant effort from us.

First, funding does not need to be the crippling issue it has become. Federal support of research pays huge dividends to the economy. According to an editorial which appeared in the New York Times on May 15, 1997, ***"Each dollar spent on basic research permanently adds 50 cents or more each year to national output - an impact that is many times larger than the permanent gains from increases in ordinary business investment."*** Investing in basic research makes good financial sense for the country. We need to communicate this message to our elected officials - personal visits and letters are known to have the greatest impact, but even telephone calls, e-mail, and FAXs are useful.

Second, the neighbors of the lab are getting incorrect and/or incomplete information. When dealing with them it is paramount to keep in mind that these are intelligent, rational human beings who are responding in a completely predictable fashion to the limited and often distorted information they have. The reality is that they are AFRAID of what is happening here at the lab and feel that they have no control over what is happening. Finally, there is nothing special about the folks who happen to live on the physical boundary of BNL. Similar people live near my home in Ithaca, NY and I'm sure they live near you as well. Their fears are real and need to be addressed with respect and compassion. It is our duty as scientists to speak to them and to speak to them in language that they understand. We must make our language accessible to average citizens without college degrees in technical subjects. We must not talk down to them. If media reports are inaccurate, we must attempt to correct them. We have to do this job because there is no one else to do it.

This job will require an investment of time and effort. At the most modest level, we must be willing to keep up to date on the FACTS. (See http://www.bnl.gov/NEWS/bnl_news.html) Have you read the DOE safety report? Have you read BNL's response? Have you read the EPA super-fund site description and remediation plan? Do you know what is actually in the plumes and where they originate from? Do you have a simple example in mind that describes the levels of solvents/tritium in the ground water and the corresponding health hazards? In short, do you understand the issues? At the next level, are you willing to write your elected officials and explain to them what you feel is important? Are you willing to write a letter to the editor either expressing dissatisfaction with or commending the accuracy of the paper's reporting? Finally, are you willing to speak with your neighbors? Are you prepared to justify to your neighbors what you do for a living?

During the upcoming year, the UEC will do its best to support users efforts in these areas by facilitating the transfer of information. Whenever possible, we will post the URLs of important BNL, DOE and EPA documents so that users can obtain the original source material. We will also attempt to alert users when issues are before Congress that affect the scientific community. However, each of us must take the initiative and utilize that information wisely.

Users' Executive Committee

Chair: Joel Brock (Cornell University)

Vice-Chair: John Parise (SUNY Stony Brook) *

Past Chair: Peter Stephens (SUNY Stony Brook)

Barbara Illman (U. of Wisconsin-Madison) *

Ian Robinson (U. of Illinois)

Thomas Russel (U. of Massachusetts)

Paul Stevens (Exxon Research & Engr.)*

*** New members, elected 5/20/97**

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Fax: (516) 344-3238
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Brookhaven National Laboratory
Bldg. 725D
Upton, NY 11973-5000

e-mail: exafs@bnlls1.nsls.bnl.gov

www.nsls.bnl.gov/BeamRD/services/exafs.HTM

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New Color-Coded BNL ID Cards for NSLS Users

If you are a foreign national who holds a guest appointment with the NSLS, please bring your current GREEN BNL ID card to the NSLS User Administration Office. It will be exchanged for a new RED card. Office hours are Monday through Friday, 8:00 am to 5:00 pm.

Users who are U.S. citizens are being issued PURPLE cards as they register. A similar exchange program for existing users (old GREEN cards replaced with new PURPLE cards) will take place in a few months.

Foreign nationals are not required to complete special paperwork in advance of their visit. They must, however, bring a valid passport with them in order to work at BNL.

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Electronic Images in Lobby Directory

The electronic user and staff directory, on the touch screen terminals located in the NSLS lobby, has been enhanced to display photo images. These digital images replace the old NSLS staff picture board which used to hang in the lobby, and includes photos of our users as well. To date there are approximately 2,000 images residing on the database, which come from the new photo ID system that was implemented last August. Pictures of all NSLS staff as well as users who have been issued ID badges since August 1996 are on the system, which gets updated weekly with new registrants. Check out the directory - whether you are looking for someone you are not familiar with or just to see how you look!

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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

Tuesday, September 30, 1997 for scheduling January - April 1998

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 Tuesday, September 30 in order to be considered for the January - April cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on September 30. 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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Space Charging To Reduce BNL Overhead Rates

Beginning October 1, 1997 (FY 1998) BNL will implement a new space charging arrangement throughout the Laboratory whereby all Departments and Divisions will pay an allocation based on square footage and type of space occupied. The new space charging scheme is designed to remove building maintenance costs from the BNL overhead area which will serve to lower the G&A rate and provide incentives to optimize use of space throughout the Laboratory. The current G&A rate of 46.5% is projected to decrease to 34.0% beginning in October.

The NSLS will not pass along these new space charges to users, but will allow all users to remain at the NSLS and benefit from the lower G&A rate. Users who are currently paying for office and lab space outside of Building #725 will continue to do so.

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User Account Information

Effective October 1, 1997, all accounts will receive the same G&A and material burden rates. The anticipated FY 98 rates are 34% for G&A and 6.5% for material burden.

Since these rates will apply to all types of funding, there is no longer a need to identify accounts as capital ("89" accounts). If there is a need to purchase capital equipment items valued over $5,000, use object classes 217 and 219, which are designated for capital procurements. Object class 217 must be used for capital items over $25,000 and object class 219 is required for items under $25,000. A capital purchase (item cost over $5,000) will solely receive a G&A charge on its material burden cost; therefore, the purchase price of the item is exempt from G&A fees.

Please note that all accounts with remaining balances (uncosted) as of September 30, 1997 will be subject to these new rates.

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CALL FOR EXPERIMENTAL SUMMARIES (ABSTRACTS)

FOR THE 1997 NSLS ACTIVITY REPORT

http://www.nsls.bnl.gov --> Science --> ESS

E-Mail and Disk Submissions Accepted Until September 26:

Anyone who decides not to use the ESS (Electronic Submission System) this year, for whatever reason, can still **submit a via E-mail or on disk** (WordPerfect, Mac or PC Word file, or ASCII text). The catch is that your submission must be post-marked **no later than September 26, 1997.** We would like to accommodate those who prefer submitting without the ESS, but because it introduces extra processing on our end and an extra round of editing on your side, we must limit the submissions to this time period.

ESS Submissions Between September 3 and October 31

* NEW submissions accepted until 5 pm Friday October 17.

*** EDITS to existing submissions allowed until 5 pm Friday October 31.**

Detailed information and instructions can be found on the Web on the ESS pages (http://www.nsls.bnl.gov, "Science --> Experimental Summary Submission"). Note that many people are going to submit at the last minute and the system will be extremely busy and slow near the deadlines. There is not much we can do to change this system limitation - please save yourself anxiety and try to submit earlier!

After October 31, 1997:

We must enforce this strict deadline in order to produce the Activity Report according to schedule and in time for the Users' Meeting. **October 31 is a hard deadline.** Although the system will remain open, any submissions entered after October 31 will be automatically rolled over into the 1998 Activity Report. The November Newsletter will have information about when the abstracts will be available for viewing on the Web.

CALL FOR PUBLICATION REFERENCES

for the period

September 30, 1996 through December 31, 1997

Our publication references are an important element in how funding agencies judge the productivity of the NSLS. All NSLS users are obligated to send the NSLS a complete publication reference for any paper based in whole or in part on research done at the NSLS. Each beamline PRT submits a list of their own papers as part of their Annual Beamline Progress Report, but the NSLS must also collect references by General Users and other collaborators in order to present the complete picture of the work being done here.

Please take a few moments to e-mail or fax us your publication references. If you are not sure whether you had already sent us your list, send it in anyway - we always check on this end to avoid double-listings. Send your references to Nancye Wright at wright1@bnl.gov or FAX 516-344-7206

DO YOUR PART TO SUPPORT THE NSLS!

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