# November 1996 NSLS NEWSLETTER

*Editor: Eva Z. Rothman*
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# NOVEMBER 1996 NSLS NEWSLETTER

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# IMPORTANT UPCOMING DATES

November 21, 1996
    Town Meeting
November 22, 1996
    UEC Meeting
January 17, 1997
    Deadline for submissions, March Newsletter
January 31, 1997
    Deadline for General User Proposals

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# November 1996 NSLS NEWSLETTER

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# *In Situ* Powder Diffraction at the NSLS

*P. Norby*

*Department of Chemistry, BNL*

Knowledge of the relations between structural and physical/chemical properties enables a better understanding of for instance reaction mechanisms. However, although knowledge of the room temperature structural properties is valuable, it can not immediately be extrapolated into the realm of reactions, as for example, catalysts at actual working conditions. Therefore, *in situ* methods have become increasingly important in order to extend the knowledge of structure-properties relationships. High intensity, tunable wavelength and low divergence are features that make synchrotron x-ray radiation a unique probe for *in situ* and time resolved studies. Two methods are dominant for *in situ* studies using x-ray synchrotron radiation, namely absorption, where local surroundings of the atoms are probed, and diffraction, where long range order can be investigated. In this article the focus will be on powder diffraction experiments.

The possibility of obtaining real-time structural and kinetic information at reaction conditions makes time resolved powder diffraction a powerful tool in a large number of technologically interesting research areas. These areas include catalysis, fuel cells, batteries, ion exchange, and syntheses. *In situ* powder diffraction studies of technologically important materials under working conditions are of increasing interest for industrial research and development. In addition, *in situ* studies are very valuable for the understanding of reaction mechanisms for basic research. Likewise, *in situ* studies of formation of materials using e.g. hydrothermal syntheses or solid state reactions, are important for obtaining information concerning crystallization mechanisms. Again, this is of great interest for basic understanding of nucleation and crystallization phenomena, but equally important in development of new materials, modification of the chemical/physical properties of materials and in materials design.

The study of materials in preassembled units, like batteries and composite wires is another area where *in situ* powder diffraction can give information otherwise not obtainable. Frequently, characterization of the materials prior to assembly or after disassembly does not yield the true picture of the state of the materials during processing or working conditions. Using high energy x-ray radiation makes it possible to penetrate container materials and get structural information about the active materials.

Information obtainable from *in situ* powder diffraction experiments include:

- Kinetics of crystallization, transformation and degradation.
- Existence of crystalline or amorphous intermediate phases.
- Crystallite size as a function of time/temperature.
- Observation of induction periods.
- Optimization of synthesis conditions.

- Real time crystal structure refinement.
- Phase identification and composition.

The remainder of this article is not intended as a complete survey of *in situ* powder diffraction experiments performed at NSLS, but rather an illustration of how information concerning crystal structure, phase changes and kinetics of crystallization and chemical reactions can be obtained from materials under processing or working conditions. An overview of industrial applications at the NSLS, which includes some *in situ* powder diffraction examples, was recently published in Synchrotron Radiation News [1].

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# *In Situ* Powder Diffraction at the NSLS:

# Time Resolved Studies at X7B

*P. Norby and J.C. Hanson*

*Chemistry Department, BNL*

During the last few years we have developed a time resolved powder diffraction facility at the Chemistry Beamline X7B at NSLS. Being a Chemistry beamline, the main emphasis has been on real-time *in situ* studies of syntheses and chemical reactions. Studies of phase transformations, crystal structure refinement using dehydration of zeolites, etc. have also been performed.

Diffraction methods can primarily be used for studies of reactions involving microcrystalline materials. However, amorphous intermediates can also be detected, either as an absence of diffraction peaks, or as very broad peaks. Crystallinity as well as progress of crystallization can be monitored using the peak width and intensity of diffraction peaks.

More detailed information concerning these experiments can be found in references [2-17]. and in the NSLS Activity Report.

It is very important to be able to extract structural information from powder diffraction data obtained using very short exposures. We have demonstrated that it is possible to refine crystal structures during dehydration of natural zeolites using a position sensitive detector. Using 5 minute exposures it was possible to obtain detailed information concerning the hydration state and cation positions[2]. Using imaging plate techniques we were able to solve the crystal structure of $Zr(HPO_4)_2$ and refine it, using the Rietveld method, from a 30s powder diffraction pattern[18]. Rietveld refinement of standard $ZrO_2$ and alpha-quartz demonstrated that imaging plate powder diffraction data yields reliable structural parameters[18,19]. We have recently studies absorption of HFC gases in zeolites, and have determined the position of the adsorbed molecule in the zeolite cage using Rietveld refinement of imaging plate powder diffraction data [17].

A simple but very versatile reaction chamber was developed for the *in situ* studies[3,4]. All reactions are performed in 0.5-1 mm quartz glass capillaries mounted on a goniometer head using a ferrule in a

Swagelock T-piece. For *in situ* studies of hydrothermal syntheses the capillary is closed in one end, and a pressure of nitrogen is applied using the connected tube (Figure 1 inset). This allows hydrothermal conditions to be obtained in the hot zone. Pressures up to 45 atm. can be used, which allows hydrothermal syntheses up to about 260ºC to be studied. This setup has been used also for studies of reactions between phenolates and $CO_2$, and for *in situ* studies of adsorption of gases on zeolites.

Using an open capillary reactions between solids and gases can be studied under flow conditions. Figure 1 shows a sketch of a two stringed flow system, where computer controlled valves allow shifting between gases. This makes kinetic studies of solid-gas reaction possible, and we have used it for studies of oxidation/reduction behavior of high temperature Solid Oxide Fuel Cell (SOFC) cathode materials at realistic working conditions, i.e. at temperatures of 700-950ºC. Profile refinement was used to determine the unit cell parameters as function of time after changing from high to low oxygen partial pressures. Kinetic parameters were extracted by fitting the unit cell volume as a function of time, which allow determination of e.g. diffusion coefficients.

We have used two types of detectors for the time resolved powder diffraction studies. An INEL CPS120 curved position sensitive detector was used initially, but we have recently constructed a Translating Imaging Plate (TIP) Camera designed especially for time- temperature- and wavelength- dependent powder diffraction experiments[20]. The design of the TIP camera was done in cooperation with Alex Darovsky, Beamline X3/SUNY@Buffalo. The principle of the TIP camera is similar to that of high temperature Guinier-Simon or Guinier-Lenne cameras except that imaging plate technique is used, and the camera has a flat plate geometry. Figure 2 shows a sketch describing the principle behind the TIP camera. The imaging plate (200x400mm Fuji) is placed behind a steel screen with a vertical slit typically 3mm wide. The imaging plate is mounted on an aluminum support, and is kept in place and flat using vacuum. The metal support is mounted on a slide, which allows the imaging plate to be translated behind the steel screen. The diffraction cones from the capillary sample are intercepted by the steel slit in front of the imaging plate, and when the plate is translated a continuous series of time resolved powder patterns are recorded. Combining the TIP camera with the *in situ* reaction cell results in a unique setup, where even fast chemical reactions can be followed as a function of time.

Figure 3 shows part of the imaging plate from a time resolved TIP experiment where phase transformations of $KNO_3$ were followed during repeated cycles of heating and cooling[9]. We were interested in the occurrence of the intermediate gamma-$KNO_3$ phase. This is a phase existing only in a short interval during cooling. The transformation temperature interval of existence is dependent on cooling rates and the thermal history of the sample.

Figure 4 is a 3-dimensional representation of a time resolved powder diffraction experiment following the hydrothermal conversion of zeolite LTA at 260ºC and 45 atm. pressure. The starting materials were zeolite Na-LTA and an aqueous solution of LiCl, and the product in this experiment is a mixture of zeolite Li-A(BW) and a cancrinite-like phase. In order to obtain kinetic information for the conversion, integrated intensities of diffraction lines can be used to estimate the progress of crystallization or degradation of the phases. Thus, information concerning rates of formation and degradation of the phases involved in the reaction, including intermediate phases, can be extracted. By combining transformation kinetics analysis with SEM pictures of partially converted zeolite LTA samples prepared on the diffractometer, it was possible to extract information concerning the transformation mechanism[15].

By performing time resolved powder diffraction experiments of hydrothermal syntheses at different temperatures it is possible to extract more detailed information, including apparent activation energies and information concerning reaction mechanisms. An example of this is a study of hydrothermal syntheses of zeolite LTA and sodalite using metakaolinite and $NaOH_{(aq)}$ as the starting materials[16,17]. Isothermal experiments were performed at various temperatures, the crystallization curves were fitted using a rate expression, and from an Arrhenius plot the apparent activation energies were determined.

*We would like to thank our many collaborators and users of the time resolved powder diffraction equipment for bringing interesting experiments to the beamline. This work was supported under contract DE-AC02-76CH00016 with the US Department of Energy by its Division of Chemical Sciences, Office of Basic Energy Sciences and by the Danish Natural Science Research Council.*

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# *In Situ* Powder Diffraction at the NSLS:

# Hard X-Rays at X27A

*T.R. Thurston*

*Physics Department, BNL*

Although the flux of hard x-rays at NSLS bending magnet ports is smaller that that available at insertion device beamlines or thrid-generation synchrotron radiation facilities, many useful *in situ* powder diffraction experiments can be performed on a properly equipped bending magnet beamline. During the past year, a hard x-ray powder instrument has been assembled and tested on X27A whihc utilizes a new type of high flux monochromator originally designed by teh NSLS medical group[21].

The monochromator consists of a bendable Si(111) crystal oriented in a Laue geometry. Bending the crystal creates a range of d-spacings which in turn causes a beam of x-rays with a large energy band pass to be diffracted. Because this monochromator operates in a transmission geometry, it only functions well at higher energies where the x-ray absorption by silicon is small, but under these conditions the diffracted flux can be orders of magnitude larger than that obtained with conventional monochromators. The rest of the instrument consists of a two-circle goniometer and the standard hardware which is a permanent part of the X27A beamline.

A number of *in situ* studies demonstrating the utility of this approach has been done. The instrument was constructed expressly for a CRADA between BNL and Intermagnetics General Corporation in which the structural properties of Bi-2223 high-$T_c$ superconducting wires are characterized. These wires are made by placing Bi-2212 powder and other precursor materials within a silver tube, drawing and rolling the tube into a tape hundreds of meters long, and then growing textured Bi-2223 superconductor material within this silver sheath in several processing steps involving high temperature heat treatments and further rollings. *In situ* hard x-ray diffraction experiments have been done with an x-ray compatible furnace to determine which processing conditions produce optimal texture and Bi-2223 phase development, and hence better current carrying capacity[22]. The x-ray energy for these experiments was set just below the silver absorption edge at ~25 keV for maximum penetration through teh silver sheath.

Under these conditions the superconductor Bragg peaks typically have intensities of ~10000 counts/sec, so the diffraction studies were straight-forward to perform. A side benefit of using hard x-rays wasa that the x-ray compatible furnace used int his work could be thermally well insulated (the hard x-rays readily penetrate through the insulating material as well as the silver), so a temperature stability of $\pm$0.2ºC at ~820ºC was attainable.

*In situ* studies of the structural changes in electrode materials within operating battery cells have also been done with this instrument. The difficulty overcome by using hard x-rays here is penetration throught eh surrounding electrolyte and battery casing materials,a s well as the electrode itself. To date, demonstration experiments have been done on two technologically relevant electrode materials at X27A: hydrogen uptake in an AB2-type nickel-metal-hydride anode and Li intercalation in the $LiMn_2O_4$ cathode of a Li-ion "rocking chair" cell. Diffraction signals up to ~50,000 counts/sec were easily attainable for both of these systems, thus showing the viability of this technique[23].

A final example of the many research problems addressable with this instrument is texture measurements of Nd-Fe-B-based permanent magnets. In this case neutron scattering measurements were unsuccessful because neutron absorption by the sample material was too large. However, hard x-rays studies on slices of magnet samples ~0.4mm thick were successful, and the results of these measurements could then be correlated with magnetic properties[24].

The work described above shows that there are many different materials research problems which can be addressed with this hard x-ray diffraction instrument. Further, the main factor limiting data quality for many *in situ* experiments is the background signal from external support materials. In these cases, this instrument makes an NSLS bending magnet port competitive with other hard x-ray sources, especially since these other sources are often heavily over subscribed.

*The author would like to thank Jerry Hastings and Peter Siddons for making generous amounts of beam time available on X27A, and for the use of the bent Laue monochromator crystal. The experiments described here were performed in collaboration with many people including P. Haldar, Y.L. Wang, and M. Suenaga (superconducting wires), S. Mukerjee, X.-Q. Yang, N.M. Jisrawi, J. McBreen, M.L. Daroux and X.K. Xing (batteries), and L.H. Lewis, V. Panchanathan, and D.O. Welch (permanent magnets). Special thanks are extended to Uli Wildgruber, who suggested that hard x-rays would be useful for studying high-$T_c$ wires in the first place, and has collaborated in experiments on both superconducting wires and permanent magnets.*

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# Chairman's Introduction

*Michael Hart*

As the end of Fiscal Year 1996 closed DOE finalized the list of competitive awards for beamline enhancements under the Presidential Scientific Facilities Initiative. The list was published on the Basic Energy Sciences www page at http://www.er.doe.gov/production/bes/WHATSNEW/SFIawards.html (BES home page is www.er.doe.gov/production/bes).

NSLS users secured twelve awards, a very encouraging number and most welcomed even though the average value was at the low end of the distribution of dollar requests. Coupled with the direct initiative funding to NSLS it is clear that we have seen a very successful move forward in the development and refurbishment of our light sources during Fiscal Year 1996. At the time of writing this Newsletter the Energy and Water bill has passed all but the final hurdle with an appropriation which we believe provides a second year of SFI funding. However, compared with FY 1995 the NSLS component seems to be only 25% to 30% of the FY 1996 step.

The full flavor of our users achievement can be seen in the list of awards, to;

Eric S. Jensen for *Ultra High Resolution Angle-Resolved Photoemission Spectrometer*
Malcolm Capel and Robert Sweet for *CCD Detector for Macromolecular Crystallography*
John W. Sutherland for *Monochromators for Elliptically Polarized Wiggler Beamline*
John D. Axe for *Magnets for Neutron and X-ray Scattering*
L. Doon Gibbs for *Mirror Upgrades for Beamlines X22B,C*
Russell J. Hemley for *High Pressure Infrared Beamline*
Peter Pershan for *Upgrade of Harvard/Brookhaven Liquid Surface X-ray Spectrometer*
Dale Sayers for *Upgrade of Beamline X11A*
Theodore Madey for *Upgrade for Core-Level Photoemission Spectroscopy*
Janos Kirz for *Enhancement of the Scanning Transmission X-ray Microscopy Facilities*
David Tanner for *Time-Resolved Far Infrared Spectrometer*
David Heskett for *Upgrade of Beamline X24A with Improved Electron Detector*

Congratulations to all the above, their collaborators and General Users. Commiserations to those who tried and almost succeeded. Let us all start work on the FY 1997 Scientific Facilities Initiative to build on this achievement.

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# A User's Perspective

*Peter W. Stephens*
*SUNY @ Stony Brook*
*UEC Chairman*

I want to start by thanking all those who wrote letters to their legislators and others in support of the Scientific Facilities Initiative in the fiscal 1997 budget. Evidently, our voices were heard, as the relevant congressional committees have (so far) kept the FY1997 SFI in the base. But I'm afraid that we cannot rest. President Clinton's off-year projection for FY2000 contains significant cuts, on the order of 25%, to the Department of Energy's Office of Energy Research. This would certainly have a devastating effect on our science, inasmuch as this is the source of funding for all of the DOE synchrotron radiation sources and research reactors. We have to get the message out that the Office of Energy Research provides an amount of basic research support comparable to the NSF. In August, I sent an appeal by electronic mail to the 2000 users in the NSLS data base, providing some more details and asking them to write to the President. (If you want a copy, please let me know: pstephens@sunysb.edu.) Curious to know how much activity that generated, last week I inquired through the same channel whether people had written or not. As of Monday September 16, there were 285 responses to the yes-or-no question, 70 of which said they had sent a letter. Another 15 said they still intended to, and the remainder said they had not.

The director of the Office of Energy Research, Dr. Martha Krebs, visited the NSLS town meeting in August and spent some time chatting with users. I think she was very pleased to see the breadth of scientific disciplines represented and the strength of our commitment to the NSLS. She certainly carried away the message that we plan on our NSLS to be an important part of our research programs for the foreseeable future, and we count on continued strong support from the DOE.

The NSLS and the UEC are taking action on some of the issues that came up in earlier user surveys. Two frequently-cited issues are the noise and clutter in the experimental areas. The noise level seems to be an insoluble problem. In the Fall of 1994, absorbing panels were installed on the walls and ceiling of the VUV Ring. Noise surveys made before, immediately after installation, and now are displayed on a poster by Frank Terrano in the NSLS entrance foyer. Professional advice is being sought on schemes for the X-Ray Ring and your comments and discussion of options are invited. One noteworthy improvement is that the NSLS has provided new chairs at all of the beamlines, replacing the duct-tape-laden castoffs that had previously cluttered the floor. We are grateful to have them. There is a new Task Force on Building Cleaniness, which will be working to hold the beamlines to reasonable standards and to do what they can to improve the custodial services delivered by the Laboratory.

A majority of users support the formation of beamline consortia based around certain widely-used techniques. The NSLS has pledged to help, but has repeatedly stated that the impetus to form any such group must come from the users themselves. It is apparent that the strongest motivation for such collaborations comes in the areas of shared equipment and supplies and common data acquisition systems. Referring to the latter, the NSLS has made a considerable contribution to the development of a new data acquisition system which has been adopted by nearly all of the EXAFS beamlines. This makes it much easier for users to move from one beamline to another. We are also working on expansion and improvements to the equipment pool. Many people have expressed an interest in obtaining access to short-term technical help. Some of the NSLS technicians are available to PRTs, depending on their workload, on an hourly cost basis. Contact the leader of the relevant group, e.g., Conrad Foerster for vacuum work, for availability. Another route is outside job-shop services. These can either be obtained through BNL by writing a PO on a capital or operating account (and paying 45% overhead), or by contacting local job-shop houses and hiring temporary personnel directly. The only BNL requirement is to process a guest appointment (through the NSLS user office) for the employee to gain access to the lab. I am happy to act as a clearinghouse for good/bad recommendations based on users' experiences. As for scientific help, in the form of paid consultants, a list of available people is compiled in the Industrial User Group entry of the NSLS world wide web page (www.nsls.bnl.gov, under "User Organizations"). The facility is studying the possibility of running the x-ray ring at a higher electron energy, 2.8 GeV, to provide a harder x-ray spectrum and to make possible certain gamma ray experiments. There have been studies of the storage ring operation, of beamline shielding, and of the impact on specific experiments. You can expect further information and solicitation of your input soon.

Finally, I want to take the opportunity to remind you that this is your user facility, and we are your User Executive Committee. If you have any comments or issues you want brought up to the NSLS, contact me or your special interest group representative.

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# NSLS Safety is Vitalizing

*W. Thomlinson*
*Associate Chairman for ES&H*

We all remember "fondly" the days of the Tiger Teams. Safety in our workplace was put on an equal priority with carrying out excellent research. The results of those efforts paid off in improved safety and awareness of good safety practices. Over time, however, BNL has noticed a gradual slippage in safety indicators. Although we continue to constantly work for a safe NSLS, we are sure that we too have slipped a bit. Earlier this year, BNL instituted a program of Safety Revitalization which was intended to not only reemphasize safety at the Laboratory, but to involve all staff and visitors in the process.

One of the central themes of the revitalization is the belief that a clean laboratory is a key part of having a safe laboratory. The first effort was a two day walk-through of the entire NSLS physical plant to identify messy, cluttered and neglected areas. The walk-through team at the NSLS consisted of management, NSLS and BNL safety personnel, a cross section of NSLS staff and users. The approach was not to focus on specific safety and housekeeping issues but, rather, to look at the "big picture". Several key issues were found which will be addressed in the upcoming ES&H Tier II/Self-Assessment of the NSLS this autumn. Among the issues needing attention are the general state of the laboratory spaces adjacent to the experimental floors, the clutter and mixed usage of many of the blue cubicles assigned to beamlines, the state of the laboratories in Building 510E and general housekeeping around beamlines. We think that we can show real progress in correcting these concerns within the next year.

Another central theme is the involvement of every individual in the revitalization process. To that end, the NSLS held a department stand-down. In a meeting of the entire staff, a comprehensive presentation of the state of safety at the NSLS was presented. The overview discussed the results of a recent ES&H questionnaire, trends in key indicators, the on-going programs in electrical and radiation safety, and the effects of recent changes in the personnel monitoring system (TLD dosimeters have replaced the old film units). A reminder of the rules governing the use of the badges follows this article.

Within days of the staff meeting, each NSLS section held a meeting to discuss safety issues of concern to individuals. The purpose was to allow staff members to raise concerns about any ES&H issue. Bill Thomlinson and Michael Hart were present to listen, take note of the concerns and occasionally answer questions. There were lively discussions of topics such as what clothes people wear at work, radiation safety, drinking water, snow clearance in winter, traffic on site, the need for new sidewalks, training programs, CPR and communication problems in safety issues. As part of the self-assessment this year, each comment will be receiving a response. The remarks have been assigned to staff members for study and initiation of the proper follow-up. By now, November, all of the comments should have been addressed.

The entire NSLS community will benefit from the cleaner, safer environment resulting from the revitalization efforts. It is important that everyone contribute to the maintenance of a safe workplace.

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# Radiation Badges:
# Tender Loving Dosimetry (TLD) !

*Nicholas F. Gmur*
*NSLS ES&H Coordinator*

During the recent NSLS Departmental and Divisional stand-down meetings, a great deal of emphasis was placed on the proper use of radiation badges. The new thermoluminescent dosimetry (TLD) badges are more sensitive than the old film-type badges. It is only through proper use of these badges that you, as an NSLS staff member or user, are going to receive accurate information on any dose you acquired while you were working here at the NSLS. The following items will remind you how to use your badge properly.

- You ***must*** wear your badge while in a Controlled Area, i.e. the VUV and X-Ray Experimental Floors as well as the posted second floor offices.
- Wear your badge ***between*** your neck and waist with the color band facing out. Do not cover your radiation badge with your BNL ID badge.
- Return your badge to its board at the end of ***every*** workday. It is only in this fashion that an accurate dose can be determined for the period you work at the NSLS.
- ***Never*** wear someone else's badge.
- If your badge is ***lost or damaged***, immediately contact your S&EP Representative at Ext. 2593 (permanent badges); contact User Administration or the Control Room (temporary badges).
- If you are found on the experimental floor without your radiation badge, you have two choices:
  a) leave the floor and retrieve your badge from your office or home; or
  b) contact User Administration or the Control Room for a temporary badge.
  ***Operations and Safety personnel will enforce this policy***.

  **Badge Exchange Days** are at the beginning of each month, notices are put up announcing badge exchange days. On those days, if you are a Permanent Badge holder, promptly pick up your new badge from your board and leave your old badge on the exchange rack (if available) or in the board's return bin. Failure to do this will delay analysis of your badge and could deplete the supply of badges needed for the following month. Temporary Badge holders should deposit their badges in any board's return bin and obtain a new badge from User Administration.

  If you have any questions, please contact Chris Weilandics (S&EP, Ext. 2593, Rm. 1-175) or Linda Feierabend (User Administration, Ext. 5763, Rm. 2-100).

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# DOW/NIST Materials Characterization Facility at U7A

*Daniel A. Fischer (Spokesperson U7A, NIST) and Benjamin M. DeKoven (DOW)*

A new soft-x-ray (C,N,O,F) materials science end station to study the structure and chemical nature of diverse materials at the National Synchrotron Light Source has been designed, built and commissioned. This experimental station, which delivers state-of-the-art intensity, resolution, and detection sensitivity is the result of a collaboration between NIST, The Dow Chemical Co., Brookhaven National Laboratory, and NIST Small Business Innovation Research (SBIR) awardees, International Radiation Detectors and Osmic Inc. On June 21, 1996 a dedication ceremony for the Dow/NIST materials characterization end station took place in conjunction with a poster session highlighting some of the many soft x-ray materials characterization experiments involving collaborations among Dow, BNL, and NIST scientists. The dedication ceremony was a celebration of the second year in the DOW/NIST soft x-ray materials analysis Cooperative Research and Development Agreement (CRDA). Management from NSLS, NIST, Dow Chemical, International Radiation Detectors, and Osmic Inc. had a first hand look at the new soft x-ray facility and the poster presentations.

The Dow/NIST materials science end station utilizes polarized ultra soft x-rays from the newly configured and upgraded NSLS U7A beamline (see the July NSLS Newsletter) as a search light for the determination of chemical bond identification, orientation and quantification. Utilizing electron and fluorescence yield detection methods the surface (5nm) and bulk (200nm) structure and chemistry can be compared and contrasted simultaneously utilizing ultra soft x-ray absorption spectroscopy (USXAS). The materials characterization end station facility is a direct result of DOW / NIST co-funding synchrotron radiation technology in a value driven partnership with industry and government scientists. Practical industrial problems are currently being investigated, such as model catalyst systems, polymer surfaces and their interfaces, self assembled monolayers and high Tc superconducting tapes. For example, the Dow Chemical Company has successfully competed for an NIST Advanced Technology Program (ATP) award, "Breakthrough Process for Direct Oxidation of Propylene to Propylene Oxide". As part of this ATP project Simon Bare *et al* (Dow) and Dan Fischer (NIST) are using the Dow/NIST materials end station at U7A for characterizing new catalyst active sites and overlayers in conjunction with the detector development projects described below.

NIST has funded two SBIR phase I and II grants focused on state of the art improvements in energy resolution and collection efficiency for the detection of soft x-ray fluorescence in conjunction with the materials characterization efforts at U7A, described above. Our goal is to develop a practical non destructive *in situ* probe for powder catalyst samples, in order to characterize the bonding of hydrocarbons to the catalyst and bond cleavage at elevated temperature and pressure. The Osmic Inc. SBIR grant is for "Development of focusing multilayer collectors for soft x-ray fluorescence yield near edge spectroscopy". The key to this improvement is a normal incidence focusing multi-layer mirror collector for wavelength dispersive collection of soft x-rays from the sample. The multi-layer mirror will concentrate the x-ray flux from a large solid angle in a narrow wavelength band pass, in order to discriminate fluorescence soft-x-rays. By utilizing the fluorescence yield multi-layer technique, measurements on materials will be greatly enhanced due to reduced backgrounds. The International Radiation Detectors SBIR grant is for "Soft x-ray silicon photodiodes for synchrotron radiation surface analysis". IRD is developing two silicon photodiode detectors. First, for incident beam intensity monitoring during experiments in the presence of a gas atmosphere. The second is a low noise, large solid angle, energy dispersive fluorescence yield detector. Both companies have delivered working prototypes which have been tested by other independent evaluation methods or institutions and found to be superior to existing instrumentation. Taken as a pair, these SBIR prototypes will improve our elemental sensitivity and greatly reduce the background in the surface structure measurement of catalysts and ceramic materials such as silicon nitride.

Following are some additional examples highlighting soft x-ray materials characterization experiments involving Dow, NIST, BNL, and IBM.

(1) The Dow Chemical Company (B. M. Dekoven *et al*), NIST (D. A. Fischer *et al*) and IBM (C. S. Bhatia *et al*) are using USXAS to probe the surface and bulk of selected lubricants applied to various magnetic hard disk substrates in order to determine the bonding and orientation of the lubricant layers at a molecular level. USXAS is an ideal non destructive tool for studying the disk lubricant system since it has both elemental and chemically sensitivity, with great selectivity in carbon bond type. In addition the average orientation of chemical bonds may be measured via the polarization anisotropy of the soft x-ray absorption spectra. Current investigations are focusing on lubricant aging and wear (i.e. head disk crashes), lubricant surface chemistry, and hard disk surface composition.

(2) The Dow Chemical Company (B. M. DeKoven *et al*) and NIST (D. A. Fischer) have utilized the bulk sensitivity of fluorescence yield (200 nm) to study a buried metal polymer interface, highlighting the use of USXAS to provide new detailed chemical information which will be useful in predicting and optimizing the adhesion in metal / polymer interfaces. An example is DVS bis-BCB (divinyl siloxane bis-benzocyclobutene) which is used for dielectric layers in new generation multilayer interconnect devices (multi-chip modules). For this application, an understanding of the nature of the bonding and complexing which occurs at the metal / polymer interface is needed. Fluorescence yield is particularly useful in studying the buried interface between DVS bis-BCB coated with aluminum (10 to 100 nm) and other metals because the fluorescence from the buried interface is able to penetrate the overlying metal film. In contrast, the relatively short depth sensitivity (5 nm) of traditional electron yield USXAS makes it useless for the buried interface study since it would only probe the outer 5 nm of the aluminum overlayer. We have collected fluorescence yield carbon K near edge spectra for evaporated aluminum coated (50 nm thick) DVS bis-BCB (50 nm thick) as well as the identical bare polymer. To enhance the metal / polymer interface sensitivity we examined difference plots (coated polymer minus bare polymer) and found a dramatic enhancement in polarization dependence after the formation of the metal / polymer interface. This polarization enhancement at the interface region implies that the aromatic ring planes of DVS bis-BCB are highly oriented towards the surface normal. These preliminary results highlight the use of USXAS to provide new detailed chemical information which may be useful in predicting and optimizing the adhesion in metal / polymer interfaces.

(3) A. Moodenbaugh (BNL - Dept. of Applied Science) and D.A. Fischer (NIST) are utilizing the bulk

sensitivity of fluorescence yield (200 nm) oxygen K USXAS to study the electronic structure of high $T_c$ superconductors. The high $T_c$ superconductors are a family of oxide compounds which range from electrical insulators through metallic conductors. The variations in electrical properties are controlled by controlling doping, using either oxygen content (in $YBa_2Cu_3O_{(7-d)}$) or substitution ($Sr^{(2+)}$ for $La^{(3+)}$ in $La_{(2-x)}Sr_xCuO_4$ ). The oxygen K near edge spectra can provide insight into electronic structure. Common features have been identified among studied high $T_c$ superconductors. These "prepeaks" vary in intensity with doping. In particular, the hole peak near 528 eV is observed in all studied systems and grows with carrier concentration. In addition, most systems show a hybridization peak near 530 eV, which decreases as conductivity increases. Both peaks typically are observed in samples with optimum $T_c$.

In order to identify these peaks with particular oxygen content, high resolution studies were used to resolve peaks in the oxygen K near edge spectra. Additionally, single crystals are being used to gain more information on the various oxygen bonds. Stable, low noise, high resolution fluorescence yield oxygen K near edge spectra are easily achieved at U7A. Fluorescence yield has an additional advantage relative to electron yield in high $T_c$ materials whose surface composition may change over time because the fluorescence method is much less surface sensitive than the often used electron yield technique.

Beside the basic inquiry, the fluorescence yield method has been used as an analytical probe for Bi-based superconducting tapes. First a series of polycrystalline samples of varying measured oxygen content was prepared. Tapes were heat treated similarly, but no straightforward method of monitoring change in oxygen content is available for these small packages. Oxygen K near edge structures, superconducting transition temperatures, and heat treatment conditions were compared between polycrystals and tapes to confirm that the mechanism of $T_c$ variation in the tapes is change of oxygen content.

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# November 1996 NSLS NEWSLETTER

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# 2.8 GeV X-Ray Ring Studies

*Roger Klaffky*
*X-Ray Ring Manager*

Over the last six months there have been studies to demonstrate the feasibility of running the X-Ray Ring at 2.80 GeV and to determine the impact of 2.8 GeV operation on experimental beamlines.

In the Spring a ramp from 744 MeV injection energy to 2.8 GeV was established and skew quadrupoles were adjusted to maintain the small vertical beam size present during 2.584 GeV operation. In August it was demonstrated that 250 mA of beam could be reliably ramped to 2.8 GeV, as had been previously predicted from RF power considerations. In the future, studies will be conducted to determine if the dual hybrid RF power amplifier installation on RF System 2 in December will increase the 250 mA operational limit. The maximum allowable current at 2.8 GeV is 340 mA, as determined by ring vacuum chamber heating limits.

Careful beamline measurements were performed by Lars Furenlid on the X19A beamline at 100 mA beam current at both 2.584 GeV and 2.80 GeV. The ratio of the third harmonic to fundamental was determined from rocking curves of the X19A second Si(111) monochromator crystal. The factor by which this ratio increases at 2.8 GeV is shown in Figure 1 for fundamental energies ranging from 2.5 KeV to 5.0 KeV. A molybdenum coated collimating mirror at a 3 milliradian incidence angle upstream of the monochromator begins to cutoff x-rays at approximately 18 KeV. These measurements are in good agreement with calculated flux ratios at 2.584 and 2.80 GeV. The flux and brightness ratios are shown in Figure 2 for 250 mA 2.80 GeV beam compared to 350 mA 2.584 GeV beam, the latter being the new standard operational beam conditions after beryllium windows are installed during the December shutdown. In another study, Sue Wirick and other beamline personnel at X1A determined that there was sufficient range in the Soft X-Ray Undulator gap motion to carry out x-ray imaging at the carbon edge without a degradation in image quality. Users wishing to determine the effect of 2.8 GeV operation on their experiments should request x-ray studies time. The Users Executive Committee has formed a 2.8 GeV Subcommittee with Carl Zimba (UEC Imaging Special Interest Group Representative), Peter Siddons (NSLS Experimental Support/Beamline R&D Head), and Steven Whisnant (UEC LEGS Special Interest Group Representative) as its members.

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# November 1996 NSLS NEWSLETTER

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# Focus on.........The Accelerator Test Facility (ATF)

*Ilan Ben-Zvi*
*NSLS*

Within the NSLS Users' Facility there is another Users' Facility that is perhaps not known to many of the NSLS users. The research conducted at this facility is proposal-driven and peer-reviewed, and users come from universities, industry, and national laboratories around the world - just like the VUV Ring and X-Ray Ring of the NSLS. However, the Accelerator Test Facility (ATF) is a unique User's Facility dedicated to long range R&D in accelerator and beam physics. The NSLS has an active R&D program in all areas of accelerator based photon sources from their design to their end use. The production of coherent radiation is being pursued worldwide, and this area is in the forefront of NSLS' program at the ATF.

There are four components to the NSLS work at the ATF: RF photocathode gun development; the microundulator FEL oscillator; the High Gain Harmonic Generation (HGHG) FEL; and a Compton back-scattering x-ray Source.

The ATF program in RF guns is recognized internationally as cutting edge R&D. One example is the recent measurement of slice emittance in a 10 ps electron bunch. Another is a brightness record, achieved in a micro-bunch of 2x108 electrons, 370 femtosecond long with an emittance of 0.5 mm mrad. The generation and careful manipulation of such beams is an area of intense activity in accelerator physics, being the key to X-Ray FELs. The Microundulator FEL Oscillator Experiment is designed to probe FEL physics at the short wavelength limit of electron beam quality, while the HGHG-FEL experiment (spokesperson - Li Hua Yu, NSLS) constitutes a Proof-of-Principle for the generation of Deep-UV(DUV) and X-Ray FELs based on single pass, sub-harmonically seeded FEL amplifiers driven by Ti:sapphire lasers. The resulting radiation from such an FEL retains the pulse length, as well as the wavelength and bandwidth stability of the seed laser. These are important considerations for the end user of the FEL.

Taking advanced source development in another direction, femtosecond x-ray pulses will be generated by Compton back-scattering of the ATF's 1 TW CO2 laser (under construction in St. Petersburg, Russia) from its 70 MeV electron beam. Preliminary estimates indicate that it should be possible to generate elliptically polarized 8 KeV x-rays at the rate of 108 to 109 photons per pulse that can be as short as a couple hundred femtosecond.

Another experiment at the ATF which has recently broken a record is the Inverse FEL Acceleration Experiment (spokesperson - Arie van Steenbergen, NSLS). This experiment, which is like an FEL amplifier operated in reverse, is motivated by the interest to develop a compact, high-gradient electron accelerator.

While the ATF is a small facility, it is rather complex, including the generation and control of high power laser and high brightness electron beams (synchronized to a picosecond) and a complete, sophisticated computer control system. To accomplish the large variety of tasks in the development and operation of this system one needs a range of expertise: Xijie Wang and Vitaly Yakimenko, (accelerator, gun, diagnostics), Marcus Babzien and Igor Pogorelsky (lasers), Robert Malone (computer and control), Bill Cahill (technical supervisor and usersÕ coordinator), Bob Harrington (mechanical and optical systems) and Mark Montemagno (electronics). Xijie is also the operations supervisor of the ATF and heads the new gun development collaborations (one with SALC and UCLA and another with KEK and Sumitomo in Japan). Igor is the head of the terawatt CO2 laser project. The ATF is benefiting from the support of the mechanical, electrical and ES&H sections of the NSLS. The cognizant engineers for the ATF are Joe Sheehan (electrical section) and John Skaritka (mechanical section) who are involved in every aspect of the ATF program and contribute invaluable expertise. In addition to the NSLS staff mentioned above, there are also consultants (at present Karl Kusche) and visitors (at present Fumio Sakai and Jin Yu). Many other people at the NSLS and the rest of BNL are associated with various experiments conducted at the ATF - the list is just too long for this occasion, but the reader may look for more information, including the many students (both current and alumni), in the ATF's World Wide Web site, with access from either the National Synchrotron Light Source or BNL Home Pages.

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# November 1996 NSLS NEWSLETTER

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# Facility Report

*Mike Kelly*
*NSLS Building Manager*

During the summer of 1996, the Task Force on Building Cleaniness was formed. Its goal is to improve general housekeeping conditions at the NSLS by re-establishing cleanliness and good housekeeping practices on the NSLS Experimental floor and surrounding areas. In addition to identifying and addressing housekeeping issues, the task force will conduct housekeeping inspections. This task force replaces the former NSLS Housekeeping Committee. The task force has already begun work on several issues, which are summarized below:

## Custodial Improvements

We have 4.5 custodians assigned to Buildings 725, 726, 727, 728, 729 and the NSLS trailer park. We have lost 1.5 custodians over the past two years due to cost control initiatives implemented by BNL. In order to cover as much territory as possible, the custodians are working on the following schedule:

Building 725

Experimental FloorTrash removal
Monday, Wednesday, and Friday

Building 725

Upstairs Trash removal
Tuesday and Thursday

Building 725

Bathrooms, conference rooms, kitchen/lunch rooms
Cleaned daily

Building 726, 727, 728, 729

Cleaned Tuesday and Thursday
Trailer Park Cleaned Tuesday and Thursday

The areas around the beamlines are vacuumed and mopped once a week unless the user declines, and stripped and waxed twice a year. The custodial supervisor will educate the custodial force on sensitive items around beamlines. She will also ensure that better cleaning occurs around equipment stands and other objects that are difficult to sweep around.

## Housekeeping

Standards have been established for a number of practices, such as equipment storage, and any special rules will be posted in the appropriate places around the experimental floor. In addition, all users and staff are urged to date any notices they post on the bulletin boards. Notices will be removed after 60 days to keep the bulletin boards from being overcrowded with old material.

## Recycling

Members of the task force have volunteered to pick up scrap metal at all beamlines once a week. The area outside the stockroom will be expanded to better handle scrap cardboard and metal, with easier access to the dumpsters and clearer instructions.

## Safety Related Issues

Improperly run wiring at beamlines is being handled at Tier One inspections. Unsafe wiring will be brought to the users' attention for correction.

Finally, in September, on Michael Hart's authorization, all the "old grey chairs" on the experimental floor were replaced with multi-adjustable work station chairs. In addition to improving the general appearance of the beamlines we have hopefully improved the comfort of NSLS users.

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# November 1996 NSLS NEWSLETTER

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# The 1996 Annual Users' Meeting at the National Synchrotron Light Source

*Peter W. Stephens*
*SUNY Stony Brook*
*Meeting Chairperson*

The NSLS users held their 1996 Users' Meeting on May 21st, 1996 at Brookhaven National Laboratory, where they had an opportunity to celebrate another year of scientific and technical achievement, to welcome Michael Hart as the new NSLS Chairman, to hear the latest news about Department of Energy support for its scientific facilities, and to visit once again with old friends. Three workshops on various scientific and technical subjects met on May 20, and three more on the 22nd. The workshops have continued to grow as an important part of the annual get-together; of the 368 people who registered for any part of the meeting, 80% attended a workshop.

The user community saw the vision for the future held by the new NSLS Chairman, Michael Hart. Reminding his audience that the NSLS operates with the high-brightness Chasman-Green lattice, he noted that the opening of third-generation synchrotron radiation sources in Europe (ESRF and Elettra) has not reduced the demand nor the productivity of second generation facilities, such as the SRS at Daresbury and HASYLAB in Hamburg. Since many experiments are limited by such factors as flux, detector capability, and source stability, they will not necessarily be improved by the newer accelerators, and the NSLS can expect to play an important scientific role for the foreseeable future. Many of these points are discussed in the recently released white paper, "NSLS Into the 21st Century".

Hart also told the users that he has reactivated the NSLS Science Advisory Committee, and has instructed them to initiate the process of tenure review for each beamline. He pointed out that the mission of many of the PRTs has changed substantially since they were created, and that the PRT system does not necessarily provide the best-supported access for all users. The NSLS is working to broaden the access available to the user community for such popular measurement techniques as EXAFS, powder diffraction, and reflectivity.

William Oosterhuis of the Department of Energy described the Scientific Facilities Initiative within the Office of Basic Energy Sciences. In the present fiscal year, $57 million was made available to the DOE programs at twelve facilities. This gave the NSLS a $5 million increase in funding, its first real increase in five years. Consequently, the NSLS has increased its beamline support staff by 20%, and made significant upgrades to the injection system, water supply, computer controls, and the accelerators themselves. Oosterhuis congratulated the user community in communicating the importance of the SFI to Congress. He indicated that the SFI is in the President's FY'97 budget (although 2% less than this year) so that we can expect to continue activities started this year.

The keynote speaker was Malcolm Browne, Pulitzer prize-winning Senior Science Writer of The New York Times. His talk, entitled "Lamplighters and Tool Makers", described two complementary modes of scientific progress: the abrupt discovery and the methodical advancement of new knowledge. He drew a number of interesting conclusions about the role of science and technology in society.

The talks about scientific topics began with Stephen Harrison of Harvard University and the Howard Hughes Medical Institute. He spoke about the role of protein crystallography in understanding how genes are switched on and off in the cell. He described his group's work on the transcription factor NF-*B, specifically on the complex of one protein dimer with a strand of DNA. This work, performed at beamlines X4A and X25, used the multiple wavelength anomalous diffraction (MAD) technique for solving the structure. It is also highlighted in a report entitled "Structural Biology Research at the NSLS" (BNL Informal Report 62916, May 1996).

Peter Siddons of the NSLS spoke about precision machining of plastic (PMMA photo-resist) using hard x-rays. The NSLS group concentrates on making three-dimensional parts, in contrast with the rest of the world's micromachining efforts, which are mostly on thin, planar structures. Among other points, he described the serendipitous discovery of a technique for making re-entrant cavities, illustrated with a tiny hexagonal wine glass. The ultimate goal is to develop these techniques to the point where they could be economically feasible on a production-line basis rather than a technical curiosity.

The next talk moved to a different part of the electromagnetic spectrum - the use of infrared radiation to elucidate high-temperature superconductivity. Dmitrii Basov of the Brookhaven Physics Department spoke about three regimes that are observed in layered cuprates as a function of oxygen concentration: insulating, "conventional" superconductivity with a gap (range of frequencies with zero conductivity), and a pseudo-gap state in which the infrared conductivity is suppressed but nonzero. In these materials, the pseudogap occurs at rather high temperature, and grows deeper as the temperature is lowered, with no abrupt change at the superconducting transition.

Gary Prinz of the Naval Research Center spoke about magnetoelectronic devices, in particular about the role of synchrotron radiation in their development. Rapid progress in this field is leading to significant applications in sensors, memories, and logic elements. These can only be pursued with the detailed understanding of the magnetism of electronic states, especially at surfaces and interfaces, which comes from elegant ultraviolet spin-resolved photoemission and magnetic circular dichroism experiments.

John Fulton of the Pacific Northwest Laboratories discussed recent work on ion solvation in supercritical water. This has been suggested as an attractive method for cleaning and disposing of hazardous materials, and it is important to have a detailed understanding of the fundamental differences between usual solution chemistry and solutions in supercritical water. *In situ* EXAFS experiments are able to reveal such information as the fact that Sr^(2+) is coordinated by half the number of water molecules in supercritical water than at standard temperature and pressure. These experiments also have significant relevance to catalysis and to geochemistry.

Polymer diffraction patterns usually have relatively few, broad peaks, whereas one normally associates synchrotron-based powder diffraction as a high resolution technique. Nevertheless, DuPont scientist Kenn Gardner described several significant advantages to the use of synchrotron radiation for his work in polymer crystallography. Particularly important is the fact that the lineshape is accurately known, whereas laboratory measurements need good standards (which do not exist), e.g., to distinguish paracrystalline disorder from finite-size broadening for low angle peaks.

Erik Johnson of the NSLS concluded the series of scientific talks with the tantalizing question, "Could you Use a Couple of Orders of Magnitude?" He proposes to deliver that improvement in the intensity of pulsed ultraviolet and infrared light at the Source Development Laboratory. This is a broadly-based project of research and development on an ultraviolet free electron laser amplifier and on the use of very short electron bunches for FEL and coherent synchrotron radiation experiments.

Two other important features of the Users' Meeting were the scientific poster session, with a reception at the Brookhaven Center, and the equipment show in the Berkner Hall lobby. Both were popular opportunities to sniff out some of the latest work at the facility, and to see some of the exciting new tools and ideas for synchrotron radiation research.

By way of administrative business, the NSLS Users' Association elected three new general members to the Users' Executive Committee: Joel Brock of Cornell University, Ian Robinson of the University of Illinois, and Thomas Russell of the University of Massachusetts. They also approved a charter revision so that future Special Interest Group elections will be held by electronic mail, and approved a new SPIG for industrial users. In executive session, the UEC elected Brock to be its Chairman-elect.

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# November 1996 NSLS NEWSLETTER

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# New Infrared Programs on the VUV Ring

*Gwyn P. Williams*
*NSLS*

The NSLS is in the process of expanding its infrared (IR) capabilities. By next Spring, a total of six IR beamlines should be operating, compared to the two (U4IR and U2B) in place now. The additional beamlines will be installed on new ports, each providing the large acceptance angles necessary to overcome diffraction effects. In addition Larry Carr has recently joined the beamline R&D group and brings considerable infrared experience. These new programs all evolved out of experiments conducted at U4IR under the NSLS General User program.

Reviewing briefly the history, the first infrared light was taken at U4IR on May 4, 1987, operated by the original PRT involving AT&T (now Lucent Technologies), Exxon, and Fairleigh-Dickinson University, as well as the NSLS. A program for General Users started in 1990 and today comprises 75% of operations. Three of these users, the Carnegie Institution of Washington (Geophysical Laboratory), SUNY at Stony Brook (Physics) and Grumman Aerospace (Research Ctr., now Northrop Grumman ATDC) collaborated with the NSLS to develop the conventional VUV port at U2B for use in both the near and middle infrared. Use of U2B for microspectroscopy by Grumman, in collaboration with Spectra-Tech and the NSLS, led to the funding of a Cooperative Research and Development Agreement (CRADA) which provided the initial impetus for the present infrared expansion. This was followed by four additional new programs. One of these is a BNL "New Initiative", involving the NSLS and Physics departments in a program to study "bad metals", and two are "Scientific Facility Initiative" programs which involve the Carnegie Institution of Washington (high pressure and microscopy studies) and the University of Florida (far-infrared and time-resolved spectroscopy). Finally, there is a new program from the Albert Einstein College of Medicine to study biological materials.

The far-infrared program will be on the new port U12IR, which involved replacing the ring vacuum chamber by the one shown in Figure 1. Also in the photograph are many of the people who were involved with the project. This port delivers 90 vertical by 90 horizontal milliradians of light and was installed in the VUV Ring during the 1995 Fall shutdown ( Figure 2). Light was extracted from this port in the Summer of 1996, after installation of the primary mirror optics. This beamline will deliver infrared radiation to a Bruker 113v interferometer or a lamellar grating interferometer (to be installed by the University of Florida). In addition to the spectrometry, the University of Florida will also implement a synchronized, mode-locked, Ti:sapphire laser for pump-probe spectroscopy studies.

The mid-infrared programs, including microspectroscopy, will be on ports U10 and U2. These ports deliver 40 vertical by 100 horizontal milliradians of light. The U10 chamber is shown in Figure 3, and was installed in the Fall of 1995. Installation of the U2 chamber is scheduled for the Fall 1996 shutdown. Both U2 and U10 will each supply two independently operating beamlines of approximately 40 by 40 milliradians. Once the remaining transport optics are installed, the microspectrometer presently at U2B will be moved to U10B, and should immediately experience a 5 to 10 fold improvement in signal. Current plans call for a Bruker 66v (vacuum) Michelson interferometer to be placed at U10A for wide spectral range studies of complex metals at low temperatures. Another Bruker 66v will be set up at U2A, along with a microscope attachment, for studies of materials at ultra-high pressures. Lastly, U2B will have a Nicolet Magna Michelson interferometer operated in vacuum, with a sample measuring chamber and cryostat customized for biological specimens. The existing U2 beamline will be removed to make way for these new lines. These five new beamlines and upgrades, in conjunction with the upgrades to the soft-xray lines U4, U5UA and U7A described by Steve Hulbert in the last Newsletter (July 1996), plus upgrades to U13 and possibly U12, will provide many state-of-the-art experimental capabilities. In many cases, such capabilities can only be achieved with a stable, lower energy, high current machine such as the NSLS 800 MeV VUV Ring.

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# November 1996 NSLS NEWSLETTER

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# Chipmunks: A Way to Monitor Radiation

*Nicholas F. Gmur*
*NSLS ES&H Coordinator*

**What is a "Chipmunk"?** A Chipmunk is a radiation monitor which determines local dose due to gamma and neutron radiation.

**How Are Chipmunks Used at the NSLS?** Chipmunks are placed in locations around Building 725 where it is deemed advisable to monitor short and long term radiation trends and patterns. This includes areas in the vicinity of the LINAC, the booster ring, the VUV Ring, the X-ray Ring, the front ends of beamlines and certain second floor offices. The output of the Chipmunks currently in place is accumulated in history files and subsequently analyzed as needed. Their alarms are also used to warn people in their vicinity that radiation levels are elevated.

How to Recognize a Chipmunk?

Each Chipmunk has an analogue dial on the front indicating dose in millirems per hour. A series of 3 colored lights are associated with various dose ranges:

**Light Color**.....................**Dose (mRem/Hr.)**
Green................................. less than 1 - 2.5
Yellow................................2.5-20
Red.......................................>20

NOTE: above 40 mRem/Hr. the chipmunk alarm will sound continuously. (Please ignore "Occupancy in Hrs./Day" on the chipmunk shown above right.)

**What to Do if a Chipmunk Gives Off an Alarm in Your Area?** Under normal conditions Chipmunks will give off occasional chirps and show a green light under background radiation conditions. If the rate of chirps increases and you see that either the yellow or red light is illuminated, do the following:

1. Move away from the vicinity of the Chipmunk and the probable source of radiation.
2. Telephone the Control Room at Ext. 2550 and inform the staff that a Chipmunk in your area is giving off an alarm. Specify the exact location of that Chipmunk and, if you can see it from where you are, indicate the level of the alarm (yellow or red).

**Questions?** Contact an Operations Coordinator on digital pager 5824.

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# November 1996 NSLS NEWSLETTER

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# New Security Procedures at the BNL Main Gate:

Due to heightened concerns over security at the Laboratory procedures at the Main Gate have changed.

**To get through the Main Gate most efficiently, please take note of the following:**

- Users who are on site frequently are urged to get a BNL car sticker for the fastest access to the BNL. Car stickers can be obtained from Brookhaven Center or through the User Administration Office.
- If you do not have a car sticker, you will be required to present your BNL picture ID card before being allowed on site. Remember, you should be carrying your BNL ID card with you every time you come to BNL!
- If you have do not have either a car sticker or a valid BNL ID card, you will be asked the purpose of your visit. NSLS users should state they will be performing research at the NSLS. You will also be asked for the name of a contact person on site - a BNL employee or someone on the beamline who is expecting you. Security will call the contact person for confirmation.

**New NSLS users, who would not have received this Newsletter, will still have to provide a contact name when they arrive at the Main Gate.** The User Administration Office will ensure each General User is informed about the procedures in their beam time confirmation letter, and the NSLS asks the Beamline PRTs to spread the word among their new collaborators.

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# NOVEMBER 1996 NSLS NEWSLETTER

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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 Friday, January 31, 1997 for scheduling May through August 1997

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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. (Forms are available from the User Administration Office or here. NEW PROPOSAL FORM AS OF 1/13/97) 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 Friday January 31 in order to be considered for the May - August cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on January 31. 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 by contacting the General User Program Coordinator. Office hours are Monday through Friday, 8:00 am to 5:00 pm Eastern Time.

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**NSLS Home Page........... BNL Home Page...........**

# November 1996 NSLS NEWSLETTER

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# Computer Repairs at the NSLS:

All hardware related problems should be called in to x5093. This includes hardware upgrades such as addition of memory or disk drive, installation of a board, etc. The NSLS Administration Group will handle all communications and paperwork with the BNL Computing and Communications Division (CCD). The NSLS will be tracking the progress of each job, and the information will be used to determine whether improvements to the system are needed.

All software-related problems should be directed to CCD at x5444. The CCD Page (http://www.ccd.bnl.gov/pcrc/pcrcprod.htm) details the software that they support. If CCD can not help please call x5093 and the problem will be addressed.

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# November 1996 NSLS NEWSLETTER

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# Activity Report 1995 is Available:

Pick up your free copy next time you are at the NSLS. Or, send your request along with your name and mailing address to Nancye Wright (wright1@bnl.gov, fax 516-344-7206).

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# November 1996 NSLS NEWSLETTER

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# X-Ray Data Booklet:
# The "Little Orange Books"

Unfortunately, the Center for X-Ray Optics is no longer printing this useful little booklet. However, much of the pertinent information can be found on the World Wide Web at the Center for X-Ray Optics (CXRO) Home Page: http://www-cxro.lbl.gov/

Clicking on the topic titled "X-Ray Interactions with Matter" should display most of the information previously found in the booklet.

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# Identification and Tagging of Equipment

The Department of Energy requires that all capital equipment at BNL have bar codes or tags to indicate ownership. If your organization does not have tags (logo's, etc., blank tags are available at no cost in the NSLS Stockroom.

Please obtain tags, fill in your organization in the space provided, and apply to all unidentified equipment belonging to your organization. The serial numbers on the blank tags are for your optional use in record-keeping.

BNL's Supply & Materiel Division will be conducting periodic inspections to ensure proper identification of all equipment. If, during the inspection, untagged equipment is found, a tag will be applied. If you have any questions or need assistance please call Armand DiFilippo at extension 3645.

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# November 1996 NSLS NEWSLETTER

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# X-ray Microtomography PRT

NSLS has, in collaboration with DAS (BNL Department of Applied Science) and CCD (Computing and Communications Division), developed a third-generation microtomography capability which currently operates on beamline X27C. CCD has constructed a 3-d viewing theater for this and other projects. We are interested in broadening the scope of applications for this instrument and forming a PRT to solidify its foundation. Anyone interested in joining such a team should contact Peter Siddons (siddons@bnl.gov, ext. 2738)

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