National Synchrotron Light Source
Annual Report 1991
Volume I
(For the period of October 1, 1990 through September 30, 1991)

Editors:
S. L. Hulbert and N. M. Lazarz

April 1992

The National Synchrotron Light Source Department
is supported by the
Office of Basic Energy Sciences
United States Department of Energy
Washington, D.C.
EDITORS’ NOTE

The Editors of the 1991 NSLS Annual Report would like to recognize Lydia Lever for her dedicated effort in typing and unending patience in compiling the NSLS Staff and User Publication lists, the latter being a major tangle of differing formats and degrees of completeness (what a mess). We also recognize Lisa Lo Sordo for painstaking typing of the lists of VUV and X-ray abstracts.

This year’s Annual Report is divided into two volumes. Volume I contains scientific highlights, operational summaries, and reports from workshops and NSLS projects, while Volume II contains all contributed VUV and X-ray abstracts. By making this division, Volume I becomes a compact summary of FY1991 work conducted at the NSLS, which can be distributed to visitors as well as to NSLS beamlines and users. Volume II will be provided as a reference volume to each NSLS beamline and to the libraries of synchrotron radiation facilities around the world. In an effort to save trees, Volume II will be provided to individuals only upon request (contact NSLS User Administration).

As you will discover, the NSLS FY1991 scientific highlights have been compiled by a collaborative group of NSLS scientific staff members and Special Interest Group representatives, with our thanks. An effort has been made to make these summaries more interesting by including a number of figures with informative captions. These figures include all beamline scientific highlights contributed by users. Beamline operational highlights remain in their own section (V); we thank all contributors and renew our request for greater participation next year (note that the cover photographs are selected from the contributed highlights).

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Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, VA 22161

NTIS price code:
Printed Copy: A09; Microfiche Copy: A01
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FRONT COVER PHOTOGRAPHS

a - The National Synchrotron Light Source, Building 725.

b - Scanning electron microscope photos of 0.1 micron lines and spaces in a 60 nm thick layer of PMMA resist, patterned using 3 different fabrication techniques by the Advanced Lithography Research Department of AT&T Bell Labs at beamline U13UB. See Section I - Lithography, Microscopy and Tomography.

c - Three 2×2 μm² images of an Au test object, as viewed in the X1A Scanning Transmission Electron Microscope (STEM). See Section I - Lithography, Microscopy and Tomography.


e - The crystal structure of the superconducting Buckminsterfullerence compound K₃C₆₀. The raspberries are C₆₀ and the smaller isolated spheres are potassium cations. This structure was solved by Peter Stephens and coworkers at SUNY and UCLA, from powder diffraction data taken at beamline X3. See Section I - X-ray Scattering and Crystallography.

f - Andy Warkentien (Technical Specialist in the NSLS Mechanical Group) and John Flannigan (Computer Analyst in the NSLS Computer Controls Group) adjust the liquid helium refrigerator to optimize the performance of the X17 Superconducting Wiggler Cryogenic System.
g - Movable exit slit chamber for the U13UA beamline, sandwiched between long welded bellows and mounted on a 880 mm-travel linear slide. The distance from the sample chamber to the center of the U13 insertion device is 23 meters. S. L. Hulbert (NSLS, Beamline R & D Section).

h - The Phase I Superconducting X-ray Lithography Source (SXLS) Storage Ring.

i - VUV front-end fast valves during assembly.

j - Tony Lenhard (NSLS Beamline R & D Section) inspecting an adjustable crystal holder for a double crystal monochromator. These monochromators are currently installed at X19A, X12B/C and X7.

k - Crystal holders for a double crystal monochromator. T. Oversluizen (NSLS Beamline R & D Section).

l - Molecular structure of Buckminsterfullerence, $C_{60}$. D. Cox (BNL Physics) and P. Heiney and coworkers (U. Penn) at beamline X7A have carried out high resolution powder diffraction experiments which revealed the existence of an orientational ordering transition in $C_{60}$ at 250K and an analogous transition in $C_{70}$. See Section I - X-ray Scattering and Crystallography.

m - Schematic of the water cooled copper monolith body of the NSLS developed beam position monitors. The tungsten blades which are used to sense the beam position are also shown. E. Johnson and T. Oversluizen (NSLS Beamline R & D Section). See Rev. Sci. Instrum. 60, 1947 (1989).

**BACK COVER PHOTOGRAPHS**

n - The U4IR beamline on the VUV Storage Ring includes three Nicolet interferometers covering the wavelength region from 1 micron to 1 mm. See Section V - Beamline Operational Highlights.

o - A 44 year old male sustained an acute myocardial infarction on Feb. 19, 1991. On Feb. 25, cardiac catherization revealed an 80% stenosis of the Right Coronary Artery (RCA) – see left hand Cine (film) image. Balloon angioplasty was performed to re-open the RCA – see middle Cine image. Transvenous coronary angiography was performed on Aug. 22, 1991 at the Synchrotron Medical Research Facility (SMERF) showing that the RCA was still patent – see right hand SMERF image, Left Anterior Oblique 40° view. Beam parameters: X17 superconducting wiggler field was 3.0 Tesla; ring current was 206mA.

p - Computed microtomography (CMT) image of the internal structure in a 1 cm fragment of the Allende carbonaceous meteorite. This 2-dimensional image is one of 40 slices in a 3-dimensional tomogram obtained in the superconducting wiggler beamline (X17B1). The high energy wiggler radiation is essential to penetrate these large, dense objects. CMT of meteorites is valuable because of the ability to determine the positions of the pseudo-circular objects (silicate-bearing chondrules that formed in the early solar nebula) prior to developing sampling strategies and allocation plans. The techniques being developed at X17B1 will be particularly useful in studying rare meteorites collected in Antarctica, some of which come from the Moon and probably Mars.
Developers of the X1A SPEM (left to right) D. Kern, IBM, E. Anderson, LBL, E. Johnson (back), NSLS, J. Kirz, SUNY at Stony Brook, S. Hulbert, NSLS, and H. Ade, SUNY at Stony Brook. H. Ade and J. Kirz were the principal investigators of this collaborative project which received a 1991 R & D 100 Award.

Using a soft x-ray undulator source and a Fresnel zone plate to produce a focused x-ray probe, researchers at X1A have obtained 50 - 75 nm resolution images of the ZnS-based P31 phosphor grains shown here. C. Jacobsen, S. Lindaas, S. Williams, and X. Zhang (SUNY at Stony Brook). See Section I – Lithography, Microscopy and Tomography.

Laue crystal used in monochromators.

A Couette Shear Cell at beamline X10A is designed for \textit{in-situ} x-ray scattering of macromolecular fluids under flow. The purpose of this research program is to explore new non-equilibrium steady-state structures of complete fluids that are produced under shear flow, due to the continuous input and dissipation of energy. See Section V – Beamline Operational Highlights.

Beamline U3A during re-assembly. The large chamber at right-center houses the new monochromator. Two of three calibration chambers are located left (downstream) of the monochromator. See Section V – Beamline Operational Highlights.

X-ray scattering at beamline X22C. Upper figure: The diffraction pattern obtained for Pt(111) at high temperatures in the reconstructed phase. Lower figure: “ring scans” taken around the (1,0) reflection as a function of temperature. A. R. Sandy, S. G. J. Mochrie (MIT), D. M. Zehner (ORNL), G. Grübel, K. G. Huang, and D. Gibbs (BNL Physics). See Section I – X-ray Scattering and Crystallography.

Two X1A-SPEM images of a microfabricated device, acquired simultaneously by monitoring the sample current (representative of the sum of all electrons liberated) together with the intensity of the electron energy analyzer. D. Kern, IBM, E. Anderson, LBL, E. Johnson, NSLS, J. Kirz, SUNY at Stony Brook, S. Hulbert, NSLS, and H. Ade, SUNY at Stony Brook. See Section I – Lithography, Microscopy and Tomography.
Section I
Introduction
INTRODUCTION

Denis McWhan
NSLS Chairman

The National Synchrotron Light Source is probably the largest user facility in the world with 3000 users from 400 institutions. The number of users continues to grow with no sign of saturating, and the number of publications based on work done at the NSLS during FY91 was 435. There is more than one Physical Review Letter published each week which is based on synchrotron radiation and approximately 60% of these come from the NSLS. This only represents one of the many fields in which forefront research is done at the Light Source. The Friday lunch seminars feature two informal 20 minute talks of new things being done on the X-ray and VUV Rings and attract an audience of close to 50 people. It is impossible to walk around the floor and not find something new and exciting that is being done.

The impressive improvement in orbit stability is clearly the highlight of this years accelerator physics program. The real time, global harmonic feedback is operating routinely on both the X-ray and VUV Rings in the vertical and on the X-ray Ring in the horizontal. The vertical orbit is stable to better than 20 microns during a fill, and this has made possible new classes of experiments. A 4th harmonic cavity is operating in the VUV Ring and tuned so as to double the length of the electron bunch. This results in a doubling of the lifetime on the VUV Ring. A third klystron was added to the linac so as to increase the injection energy to 130 MeV, and a major program is underway to improve the operation of the booster. The controls group has made major strides in understanding and dramatically improving the communications rate between the micros and the workstations. Implementing Ethernet communications will lead to improved reliability and performance. Studies of the water system have led to an understanding of its deficiencies, and a plan to improve the stability of the water temperature is underway.

Major improvements have been made in the work environment for the users of the NSLS. A rearrangement of various groups and facilities has led to a consolidation of user services. A new user administration office is located right upstairs from the front door, and the new electronic registration system painlessly takes care of all the new users. We have established a permanent memorial to the accelerator physicists whose work forms the basis of all low emittance storage rings with the Chasman-Green Memorial Library/Lounge which is adjacent to the experimental floor. The library/lounge provides a place to discuss new results, to consult a reference book or to take a nap, and should provide a focus of activity on the floor. Adjacent to the library are the computerized stockroom, the offices of the floor manager, control room supervisors, safety engineers, and safety and environmental personnel, and the canteen/kitchen. Most of the people and facilities that the users need are now located in one area. The user shop has been improved and is now staffed during working hours. More staff has been added to keep the facility beamlines at a
state of the art level and to develop new techniques. The Directorate of Brookhaven National Laboratory has shown its strong commitment to the NSLS by providing money to expand the building between X1 and X5 on the X-ray Ring. We are optimistic that line item funding will be obtained this year for an addition devoted to structural biology research between X5 and X11. With these additions we will come close to being able to provide secure setup space adjacent to the experimental floor for each Participating Research Team.

The superconducting x-ray lithography source (SXLS) is moving forward. The Phase I project involved building a storage ring with conventional iron magnets. This ring was commissioned and extensive accelerator physics studies have been done during the year. The ring has stored currents of 1.5 Amperes, which is three times the design specification. Construction of the Phase II ring, which will use superconducting 180 degree dipole magnets, is underway.

The design of a Free Electron Laser user facility, operating in the ultra-violet, has continued and has been extensively reviewed by a blue ribbon FEL panel. Preconstruction R&D, suggested by the panel, is beginning at the Accelerator Test Facility with collaborations involving the NSLS and Chemistry departments at BNL and the Grumman Aerospace Company. We look forward to important accelerator physics developments being made using the X-ray and VUV Rings, the SXLS, and the ATF.

The 1991 NSLS Users’ Meeting was held on May 20–22. The first day was devoted to five workshops on Atomic and Molecular Science, Computational Tools in XAFS, Electronic and Chemical Phenomena at Surfaces, Imaging, and Surface Structure. The number of workshops has been increasing each year, and this year they attracted more than 220 people. The main Users’ Meeting was highlighted by an overview of the NSLS by Denis McWhan and ten scientific talks on topics ranging from X-ray Projection Lithography to Mossbauer Spectroscopy. The meeting was attended by over 310 people, which is down just slightly from 1990. There was a vendor display with 18 vendors and a poster session with over 60 posters of research conducted at the NSLS. Summaries of the main Users’ Meeting and the five workshops can be found in Section II of this report.

Both the VUV and X-ray rings have been running well. The X-ray ring has seen significant improvements in beam stability with the addition of both vertical and horizontal feedback systems, improvements which have been high on the users’ priority list for some time. Further improvements in

USERS’ EXECUTIVE COMMITTEE

Mark Rivers
Univ. of Chicago
UEC Chairperson
the VUV feedback system are scheduled for the near future. A comprehensive program of upgrading the booster ring is under way, which should improve the speed and reliability of injection into both storage rings.

The NSLS has been successful in securing General Plant Projects (GPP) money for two building additions, including an expansion of the experimental floor in the region between X1 and X5. This space will be used in part for new user laboratories. The biology initiative to expand the ring between X5 and X11 is still under active consideration by Office of Health and Environmental Research (OHER). These additions should allow every X-ray beamline to have an amount of dedicated setup space.

It appears that the NEXRAD advance weather radar system will definitely be coming to BNL, to be installed on a site about 3/4 mile from the NSLS with operations scheduled to begin in 1994. There is a written agreement that NEXRAD will provide whatever screening is required to reduce RF radiation levels to 2 V/m inside the NSLS and other BNL buildings. Additional local shielding will be installed if any experiments are still adversely impacted.

There has been good progress on a number of “quality of life” items affecting users. The reorganization of NSLS space is now almost complete. The new user library/lounge and the much more convenient location of the User Administration Office are among the benefits of these changes for the user community. The Staff Services Division has made a number of improvements in the areas of housing and food services. These include expanded dinner services and the addition of a bar and video games at the Brookhaven Center. A number of apartments are being renovated each year now. The dormitories will be upgraded with microwave ovens in each of the kitchens and VCRs on the TVs in the common rooms. Staff Services is actively working with DOE to overcome accounting obstacles to allow the use of credit cards to pay for housing at BNL and food service at the Center.

The present members of the UEC are:

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<th>Name</th>
<th>Position</th>
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<tr>
<td>Mark Rivers</td>
<td>Chairperson</td>
<td>Univ. of Chicago</td>
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<tr>
<td>Neal Shinn</td>
<td>Vice Chairperson</td>
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<td>Peter Johnson</td>
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<td>BNL Physics Department</td>
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<tr>
<td>Brant Johnson</td>
<td>Secretary</td>
<td>BNL Applied Science Dept. and Atomic &amp; Molecular Science SPIG Rep.</td>
</tr>
<tr>
<td>C.T. Chen</td>
<td>Member</td>
<td>AT&amp;T Bell Laboratories</td>
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<td>Paul Cowan</td>
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<td>Argonne National Laboratory</td>
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<td>Eric Jensen</td>
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<td>John Kirkland</td>
<td>Member</td>
<td>Naval Research Laboratory</td>
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<td>Gabrielle Long</td>
<td>Member</td>
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<td>Larry Sorenson</td>
<td>Member</td>
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<tr>
<td>Brian Stephenson</td>
<td>Member</td>
<td>IBM Research Center</td>
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<tr>
<td>Susan White-DePace</td>
<td>Ex. Officio</td>
<td>NSLS User Administrator</td>
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Special Interest Group (SPIG) Representatives

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<th>Name</th>
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<tr>
<td>Robert Bartynski</td>
<td>UV Photoemis-</td>
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<td>Charles Bouldin</td>
<td>XAFS</td>
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<td>Michael Dudley</td>
<td>Topography</td>
<td>SUNY at Stony Brook</td>
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<td>Jonathan Hanson</td>
<td>X-ray Scattering</td>
<td>BNL Chemistry Dept.</td>
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<td>&amp; Crystallography</td>
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<td>Stephen Lindaas</td>
<td>Students &amp; Post-Docs</td>
<td>SUNY at Stony Brook</td>
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<td>Andrew Sandorfi</td>
<td>Nuclear Physics</td>
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<td>Earl Skelton</td>
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<td>Shawn Williams</td>
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<td>SUNY at Stony Brook</td>
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<td>Microscopy/Tomography/X-ray Fluorescence</td>
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The 1992 Scientific Program Advisory Committee (SPSC) chaired by Roger Klaffky and Randy Alkire has been very active. This committee is responsible for identifying significant items which affect the scientific and working environment of the NSLS.

One of the items the UEC has asked this committee to work on is the audio noise level on the experimental floor. The sentiment of the UEC was that users would be willing to abide by noise abatement regulations, such as soundproofing of pumps and equipment racks if it would significantly reduce the noise levels on the floor.

The Space Committee of the UEC is one of the most important committees, recommending policy and implementing space assignments for user labs and offices. This chairmanship of this committee has been handled for many years by the Exxon PRT, first by Rich Hewitt and more recently by Kevin D’Amico. I would like to thank them both for their considerable efforts over the years. The new chairman of this committee is Brian Stephenson of IBM.

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1991 SCIENTIFIC PROGRAM SUPPORT COMMITTEE

Randy W. Alkire
ANL

Roger Klaffky
NSLS

SPSC 91 Co-chairpersons

The 1991 Scientific Program Support Committee was formed in October 1991 and was charged with the task of making recommendations to the UEC that would address user related problems at the NSLS. All aspects of life at the NSLS were considered, including ring operations and quality of life issues. The committee chose to focus on about a dozen topics and final recommendations on these topics will be made in the SPSC 91 report, scheduled to be completed in May 1991. The committee members were:

A. Ackerman - NSLS
R. Alkire - ANL (Co-Chair)
D. Costas - IBM
N. Gmiir - NSLS (Secretary)
S. Hulbert - NSLS
J. Kirkland - Sachs Freeman Assoc.
R. Klaffky - NSLS (Co-Chair)
A. MacDowell - AT&T Bell Labs
Ring operations, computing user support facilities, safety and security were the main topics addressed by SPSC 91. In addition, the committee continued to look at several issues examined by previous SPSC committees. Highlights from these investigations will be discussed here, and the complete list of recommendations can be found in the SPSC 91 final report.

Beam stability on both storage rings has been a major topic of concern with the past SPSC committees, and with the installation of global orbit feedback on both rings, beam stability has improved dramatically. However, the orbit to which the beam is locked by the global feedback is determined at the beginning of each fill by a global orbit correction algorithm that minimizes the r.m.s. sum of the deviations from this orbit of all of the pick-up electrodes (PUEs) around the ring. Since this minimization scheme is global, rather than local, there can be significant fill-to-fill deviations at a given point in the ring. The committee is tracking fill-to-fill changes in beam position at every X-ray Ring dipole magnet in order to determine baseline characteristics for later comparison. A procedure for locally correcting the orbit in every X-ray straight section is being implemented which should reduce fill-to-fill orbit deviations on the adjacent bending magnets.

Xyplex (9600 baud terminal server to ethernet) connections are currently available on the VUV Ring and plans for installation on the X-ray Ring are in progress. Some problems with use of the Xyplex system were determined to be caused by too little memory on the Xyplex boards, which have been solved by installing expanded memory boards.

Significant progress has been made by the committee in getting the NSLS trailer park hooked up to Ethernet. All necessary cable has been installed and the final fiber optic link will be completed by early spring 1992.

The past year has seen a rearrangement of space on the NSLS floor, yielding a more centrally located user administration office, easier access to periodicals, fax and copy machines in the Chasman-Green Memorial Library/Lounge, and more centrally located storage space. Further improvements will be recommended this year regarding the shipping of large items and the user machine shop. Shipping will be made easier and most cost effective by stocking boxes large enough to ship items such as Camac crates, avoiding costly crating charges. The user machine shop steward will begin making recommendations of which raw materials to be put in the stockroom for use in the user machine shop, such as bar stock, Unistrut, and aluminum plate. User training in the machine shop will also be offered to acquaint general users with safe machine shop practices.

Safety is being expanded as a topic by the SPSC and questions regarding scheduling of work by outside contractors, workmen training, and waste disposal are being investigated.

Noise, a topic looked at by all previous SPSC committees, is a problem that will not go away without a significant influx of money. However, the committee has recently purchased sound insulation materials in order to examine the possibility of reducing noise locally by insulating electronics racks and computing equipment.

Theft of equipment at the NSLS has been a major concern of users in the past, primarily during extended shutdown periods. Results of user surveys and an extensive look at police reports show that building 725 does not have a problem with theft, although the same cannot be said for 510. The committee has also looked at personnel security at the NSLS trailer park, and plans are currently underway to install additional lighting around the park to increase illumination in the area.

Other areas in which the users can expect improvement include more frequent filling of vending machines (with a wider variety of food, eg. vegetarian), more routine use of pocket pagers to let users know when ring problems occur, and extended repair services offered through third party services, accessible to users with an NSLS account.
I. Valence electronic excitation

Beamline U11 is dedicated to gas phase studies involving valence electronic excitations. Of special interest are chemical dynamics in reactions and in van der Waals clusters.

With the use of synchrotron radiation in photoionization mass spectrometry, chemical reaction products are identified, and the photon energy dependence of their yields are measured. The major channels for the reaction between N and the C₆H₆ radical are suggested to be:

\[ N + C_2H_5 \rightarrow H_2CN + CH_3 \rightarrow C_2H_4 + NH. \]

A study has been made on the photoionization spectral yields of the radicals H₂CN and D₂CN, generated by the reactions of N with CH₃ and CD₃, respectively.

Recent photoionization and photodissociation experiments on van der Waals clusters include the monomer and clusters of thiophene, \((C_4H_2S)_n\) \(n=1,2,3\); CF₃Br and its mixed dimer with CH₃OH; and complexes of O₂ with C₆H₆ and C₆F₆. In the last study, a surprising result is that \((C_6H_6\cdot O_2)^+\) is formed by the non-dissociative photoionization of the van der Waals dimer \((C_6H_6\cdot O_2)\) alone. On the other hand, C₆H₆O⁺ and C₆F₆O⁺ are only produced from larger clusters by photoionization-induced intracluster reactions. The yield curve for C₆H₆O⁺ in Fig. 1 suggests the mechanism by which the ion is produced from \((C_6H_6)_2O_2\). The observations are consistent with the calculated structures of \((C_6H_6)_2O_2\) and C₆H₆•O₂ shown in Fig. 2. With a newly developed instrument, it is found that the kinetic energy of \((C_4H_6\cdot SO_2)^+\), produced from the dissociative photoionization of the van der Waals cluster \((1,3-C_4H_6)_n(SO_2)_m\), is surprisingly larger than the van der Waals bond strengths.

Fig. 1 Yield curve for the production of C₆H₆O⁺ from the van der Waals mixed trimer \((C_6H_6)_2O_2\). The window resonance corresponding to the \(\left(1^4\Sigma_u^+\right)^3\delta_a\) Rydberg state of free oxygen is prominent at 580 Å, showing, for the first time, the involvement of autoionization in intracluster reactions where only polyatomic molecules are present. The inset shows the first onset, at 14.10 ± 0.05 eV, which is far above the thermodynamic threshold. However, it is beautifully consistent with direct ionization of a C₆H₆ moiety to produce a bound, excited benzene ion corresponding to the \(^2^2\Sigma^+\) or \(^2^B_2\) state, which then transfers its excitation into the neighboring O₂, dissociating it with capture of one of the oxygen atoms. Elimination of a C₆H₆ moiety disposes enough energy to stabilize C₆H₆O⁺. J. R. Grover (BNL Chemistry Dept.), G. Hagenow (Freie Univ. Berlin), and E. A. Walters (U. of New Mexico).
II. Core electronic excitation
The dynamics of core electron excitation and de-excitation process in small molecules constitute the main research on Beamline U15. These are probed by ion yield measurements at different angles and electron-ion coincidence measurements. Very recently, the high resolution and high flux on U13UA have enabled refined measurements to be made.

The symmetry and photo-absorption asymmetry parameter $\beta$ of core-hole excited molecular states are investigated by looking at the angular dependence of photo-fragmentation. Ion yield spectra are recorded simultaneously by two retarding field analyzers with their axes oriented perpendicular and parallel to the electric vector of synchrotron radiation. The excitation of a series of diatomic molecules (N$_2$, O$_2$, NO and CO) near the N and O K-edges shows the general pattern of a pre-edge $\pi^*$ resonance and a $\sigma^*$ resonance. In addition, the $\beta$-spectra of NO at the N K-edge and the O K-edge are found to be qualitatively similar to the corresponding $\beta$-spectra of N$_2$ and O$_2$, respectively. A second study is directed towards triatomic molecules with different symmetries, namely, CO$_2$ (D$_{\infty h}$), N$_2$O (C$_{\infty v}$) and H$_2$O (C$_2v$); the ion yields and the anisotropy parameters at the O K-edges as well as other atomic edges have been determined.

The chemistry induced by core electron excitation is studied by electron multiple ion coincidence measurements. The behavior of CO upon O 1s photoionization is studied by mass spectrometry in coincidence with 465-475 eV Auger electrons, which produce two CO$^+$ states with known energies. Their fragmentation to C$^+$ and O$^+$ leaves these atomic ions in highly excited electronic states, as deduced by the difference in the parent ion energies and the kinetic energies of the atomic ions. The distribution of energies in other fragmentation pathways giving dipositive ions is also obtained. In a study of NH$_3$ upon the creation of a N1s core hole, the mass spectrum in coincidence with 369-379 eV Auger electrons gives the dissociation dynamics of NH$_3^{2+}$ in its ground singlet and first excited electronic states. A recent development is the detection of ion pairs, and a study is made on the fragmentation of methanol following the excitation of C 1s to the continuum (see Fig. 3).
Chemically important pathways of core hole decay of large molecules may often include fragmentation involving neutral species in an excited state. A complete characterization of the dynamics of core hole decay of such molecules would not be possible without information about the neutral decay products. An ideal technique for observation of the neutral fragments is the observation of luminescence from excited states of such fragments (see Fig. 4). From a knowledge of the excitation energy of the atomic species and the final state energies of the Auger decay products it is possible to determine the probable final states that result in the observed fragments.
On Beamline U13UA, two recent gas phase experiments have been done. For N 1s photoabsorption in N₂, angular-dependent ion yield measurements have identified excitations to π and σ symmetry states and refined a previous peak assignment obtained by high-resolution absorption spectroscopy on U4B (see Fig. 5).

The second experiment demonstrates the effect of site and state specificity in molecular core hole decay. The C K-edge resonances in acetone in Fig. 7, obtained by total ion yield, show 1s → π* and 1s → 3p excitations from different carbon atoms. The identification is made possible from the Auger electron spectra at resonance together with electron-ion coincidence data (see Fig. 6).

Fig. 5 Ion Yields from N₂ at 0° and 90°, and the anisotropy parameter, β. spectra. The β spectrum is constructed from the ion yields at the two detectors and the degree of light polarization. Excitations to symmetry electronic states appear only at 90°, and excitation to Σ symmetry electronic states appear only at 0°. Resonance peaks that have not been resolved by conventional high resolution spectroscopy can now be revealed in the two different detectors. D. Y. Kim, K. Lee, C.-I. Ma, M. Mahalingham, and D. M. Hanson (SUNY at Stony Brook), and S. L. Hulbert (NSLS).
III. High resolution core level excitation and de-excitation

On the newly completed X1B beamline, core level spectroscopies in small molecules have been done. The combination of undulator radiation and high-resolution soft x-ray optics has resolved features in the O K-edge photoabsorption spectra of O$_2$ more clearly than before. The capability for core level photoelectron spectroscopy at vibrational resolution is also demonstrated, as shown in Fig. 7. The vibrational fine structure in the decay of core hole states in gas phase N$_2$ reveals information about the coupling between electronic states and nuclear motion, both evolving on a time scale of $\sim 10^{-14}$ s. The first observation of vibrational selection on core hole decay is shown in Fig. 8. The high intensity has also enabled the observation of electronic decay in the 1s$^3$3p and 1s$^{-1}$ 3s core hole Rydberg states, together with the much more intense 1s$^{-1} \pi^*$ decays. A comparison yields information on the Coulomb interactions between the spectator Rydberg electron and the rest of the system.

![Fig. 6](image_url) (a) C K-edge resonances in acetone, showing the 1s $\rightarrow \pi^*$ resonance of the carbonyl carbon (C$_1$) and the 1s $\rightarrow$3p resonance of the methyl carbon (C$_2$). The assignment is made from the Auger electron spectra at resonance (b,c) together with electron-ion coincidence data. For example, the spectral shape of (c) resembles the Auger spectrum of methane, which implies that C$_2$ is the methyl carbon atom and spectator decay dominates the core hole relaxation. By elimination, C$_1$ is the carbonyl carbon atom. C.-I. Ma, K. Lee, D. Y. Kim, and D. M. Hanson (SUNY at Stony Brook) and S. L. Hulbert (NSLS).

![Fig. 7](image_url) C 1s photoelectron spectrum of methane taken at a photon energy of $\sim 304$ eV on X1B, clearly resolving the vibrational peaks. K. J. Randall, J. Feldhaus, A.L.D. Kilcoyne, A. M. Bradshaw (Fritz-Haber-Institut.), W. Eberhardt, J. E. Rubensson (KFA Jülich), Y. Ma (PNL), Z. Xu, and P. D. Johnson (BNL Physics Dept.).
IV. X-ray Photoionization

On beamline X24A, the double photoionization of He at 2.8 KeV has been studied. The ratio $\text{He}^{++}/\text{He}^+$ is found to be $1.6 \pm 0.3\%$, presenting the first accurate measurement at photon energies greater than 200 eV. This measurement is important because the photoionization of He is the testing ground for understanding electron correlation.

On beamline X26C, initial studies have been made of the double differential cross-sections of Compton-ejected electrons from Ne, using 10-15 keV photons obtained by filtering white light with a thick Be window and 3mm Kapton. A spherical sector electrostatic spectrometer has been used to detect electron kinetic energies up to 4.5 keV. Modifications to the gas jet, the scattering chamber, and plans for a magnetic spectrometer to detect up to 100 keV electrons, are under way.

Fig. 8 (a) The decay of the first vibronic level of the $1s^{-1} \pi^*$ state of gas phase $\text{N}_2$, obtained on beamline X1B. The sharp peak at 16.7 eV corresponds to the first vibrational level of the final state with a $1\pi_u$ hole, and the weaker progression starting at 15.6 eV corresponds to the first vibrations of the final state with a $3\sigma_g$ hole. (b) Previously recorded spectrum without vibrational resolution in the excitation. J. E. Rubensson, W. Eberhardt (KFA Jülich), Y. Ma (PNL), Z. Xu, and P. D. Johnson (BNL Physics Dept.).
Energy dispersive diffraction techniques at NSLS are principally used for high pressure research. Extreme pressures, in excess of those found at the center of the earth (≈360 GPa), are developed in the submillimeter regions between the flattened tips of opposed diamond-anvils. Larger volume high pressure capabilities are available in the cubic press, operational on X17B. During the past year, some diamond-anvil cell energy dispersive diffraction studies were pursued on X7A, but the bulk of the work was performed on the beamline designed for this technique, X17C.

Using X7A in the white beam mode, Wang et al. from the University of Hawaii studied the isothermal compression of spinel (Fe₂SiO₄) at pressures up to 24 GPa. They observed an apparent stiffening of the spinel lattice above 18 GPa, which they attributed to nonhydrostatic conditions. In a separate study, Reichlin et al. from Lawrence Livermore National Laboratory (LLNL), in collaboration with Hemley et al. from Carnegie Institution of Washington (CIW), found that monatomic metallic iodine transforms to a bcc structure at 55 GPa, which prevails to at least 181 GPa. At this pressure, the volume compression, V/V₀, is measured to be 0.356.

Zhao et al. from SUNY at Stony Brook, using the cubic press on X17B, measured both the thermal expansivity and the isothermal compressibility of NaMgF₃. At ambient pressure and a temperature of 765°C, a phase transformation was observed from the orthorhombically distorted perovskite structure (Pbnm) to a cubic phase (Pm3m). Results from these measurements allow deductions about the iron content of the earth's core.

A broad spectrum of energy dispersive diffraction work has been performed on X17C, ranging from structure determinations of crystals with volumes as small as 28 attoliters to studies at pressures approaching 300 GPa and at temperatures ranging from 50 to 900 K.

Loubeyre et al. from Universite Paris, collaborating with scientists from CIW, measured the phase diagram and equation of state of helium over the range from 50 to 300 K and from 4 to 20 GPa. Relations between the fluid phase and the two condensed phases, fcc and hcp, were determined from energy dispersive measurements on polycrystalline samples (see Fig. 9).

![Fig. 9. Phase diagram of ⁴He measured at high pressure using X17C. The large symbols indicate where single crystals had been grown from the melt by the isochoric method. P. Loubeyre et al. (Universite Paris).](image-url)
In a similar study, but on a single crystal sample, Mao et al. from CIW confirmed that hydrogen crystallizes in the hcp structure up to at least 42 GPa. Data from this sample allowed fitting the Birch-Murnaghan equation of state to over 40 GPa (see Fig. 10).

A number of high pressure investigations were pursued on a variety of materials. From CIW, magnesiowsitite, \( (\text{Mg}_{0.6}\text{Fe}_{0.4})\text{O} \), was examined to 30 GPa and 800 K by Fei et al.; Hu et al. collected data on possible high pressure calibrants, W and Pt, to hundreds of GPa (see Fig. 11); Vos et al. performed measurements to 10 GPa and elevated temperatures to chart the phase diagrams of \( \text{N}_2 \), He, and \( \text{N}_2\text{-He} \) mixture (see Fig. 12); and Meade et al. performed studies on silica glass to 42 GPa, the results of which can be interpreted as an increase in the short and intermediate range order with increasing pressure (see Fig. 13).

**Fig. 10.** Pressure-volume relation of solid hydrogen at room temperature, measured at X17C up to a pressure of 42 GPa. Fitting the data to the Birch-Murnaghan equation of state yields an isothermal bulk modulus \( K_0=0.492 \text{ GPa} \) and \( K=4.3 \). H. K. Mao et al. (Carnegie Institution of Washington).

**Fig. 11.** The energy dispersive x-ray diffraction spectrum of polycrystalline tungsten and platinum at \( P=289.00 \pm 8.46 \) GPa, \( T=294 \text{K} \), and \( 2\theta=20.011^\circ \pm 0.007^\circ \). The spectrum was obtained in 23 minutes at X17C. J. Z. Hu et al. (Carnegie Institution of Washington).
In a separate study, Skelton et al. from NRL, in collaboration with Finger et al. from CIW, obtained energy dispersive diffraction data from a cylindrical single crystal of Bi having a diameter of only \( \approx 0.067 \) \( \mu \text{m} \). Provisional refinement of these data reveal a new phase of bismuth, Bi-X, with an fcc lattice \((a=10.65 \pm 0.05\text{Å})\) belonging to the space group F-43m.

Qadri et al. from NRL examined phase transitions in \( \text{Zn}_{1-x}\text{Fe}_x\text{Se} \) and \( \text{Hg}_{1-x}\text{Fe}_x\text{Se} \) (see Fig. 14).

Akella et al. from LLNL studied the equation of state of Nd to 90 GPa.

![P-T phase diagram of \( \text{N}_2 \), He, and \( \text{N}_2\)-He mixture measured at X17C.](image1)

![Structure Factor Silica Glass](image2)

![The structure of SiO\(_2\) glass at high pressure.](image3)

![The equation of state of \( \text{Zn}_{1-x}\text{Fe}_x\text{Se} \) (x=0.17) measured at X17C.](image4)
LITHOGRAPHY, MICROSCOPY, AND TOMOGRAPHY

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Lithography

The three operating x-ray lithography beamlines continued active programs during FY 1991. Beamlines U2 (IBM) and U6 (IBM) are primarily concerned with proximity printing although some of the radiation induced effects are of general interest. U13UB (AT&T Bell Labs) is primarily concerned with projection printing.

U2 operated reliably with the Karl-Suss stepper being used for several production runs in conjunction with personnel from Grumman Aerospace Corporation.

U6 was used in several different studies, all of which can be classified as radiation induced effects. These included studies of mask distortions, of new resists, and of radiation damage effects to the circuits being printed. Specific examples are the effects on the emitter gain of bipolar transistors and the susceptibilities of different gate dielectrics and other insulating elements to degradation due to the creation of residual electron traps. The effects of annealing on radiation induced damage was also a part of the study, and it was found that in general, induced charged or bipolar traps in the materials anneal out well compared with neutral traps. Thus it is desirable to choose materials which are less prone to the creation of neutral traps.

At U13UB several studies of the physics of reflective optics and diffraction limited imaging took place. Two instruments, a Schwarzschild reduction optic and an Offner 1:1 ring field system, were used to print 0.1 micron lines and spaces using both transmission and reflective masks (see Fig. 15).

The reflective masks are part of the program involving multilayers which will also be applied to reducing the operating wavelength of the ring field camera system from its present value of 46 nm to 14 nm.

Multilayers were also studied at U8 (IBM) for use in a soft x-ray telescope which was used by collaborators at the Harvard-Smithsonian Center for Astrophysics to photograph the sun.

Schwarzschild Camera Images in PMMA Using Patterned Reflection Masks

![Image of Schwarzschild Camera Images in PMMA Using Patterned Reflection Masks]

The U13UB researchers have for the first time compared the performance of Mo/Si multilayer coated reflection masks on rugged, stable substrates to that of a transmission mask using a 20:1 reduction Schwarzschild optic at 14 nm. They found that there is no observable difference among reflection masks patterned using different fabrication techniques, as shown in these scanning electron microscope photographs of images of 0.1 micron lines and spaces in a 60 nm thick layer of PMMA resist. (a,b) Printed from reflecting masks patterned by absorbing layers deposited on top of a multilayer reflector. (c) Printed from a reflective mask patterned by reactive ion removal of reflective coating. There is no observable difference between reflection and transmission mask results. R. Freeman, J. Bjorkholm, D. Tennant, L. Eichner, T. Jewell, A. MacDowell, L. Szeto, D. Taylor, W. Waskiewicz, D. White, D. Windt, and O. Wood (AT&T Bell Labs).

Fig. 15 The Advanced Lithography Research Department of AT&T Bell Laboratories is involved in an experimental program on Beamline U13UB to determine the feasibility of using soft x-rays at wavelengths near 14 nm to carry out advanced projection lithography. It has been suggested that soft x-ray projection printing can be accomplished using a reflecting mask fabricated on a rugged, stable substrate. The substrate must exhibit minimal thermal distortion, since the power incident on the mask may be hundreds of milliwatts. This gives reflecting masks a great potential advantage over the membrane masks currently used in x-ray proximity printing.
Microscopy

The x-ray microscopy program at X1A saw several developments during FY 1991, both in terms of technique development and application. The scanning transmission microscope was upgraded to a resolution of 45 nm using a new zone plate and scanning stage (see Fig. 16).

Some of the biological samples imaged with this improved setup have been muscle, calcium deposits in human cartilage, zymogen granules, in which it was possible to estimate the protein content, and chromosomes (see Fig. 17).

Not only can the beam be almost routinely focused to these dimensions, but using the high brightness, it was possible for the first time to detect luminescence from a sample as the micro-focused beam was scanned over it (see Fig. 18).

In another new application, it proved possible to tune the photon energy to the energy corresponding to transitions from the 1s level to the aromatic $2\pi^*$ of carbon and to other nearby final states in a polymer mixture, and so image carbon in a specific chemical state. These experiments involve adjusting the focus of the sample since the zone plate images different wavelengths at different distances. Finally, a series of experiments began in which a series of new detectors were tested with the aim of doing phase contrast imaging. In a demonstration of the application of x-ray microscopy, a scanning photoelectron microscope picture was successfully taken of a microelectronic device.
device at a one-dimensional resolution of 100 nm and a two-dimensional resolution of 400 nm. The contrast in the image was due to the different elements of which the circuit was composed and their different chemical states (see Fig. 19).

Fig. 18 Scanning luminescence x-ray microscopy (SLXM) is based on the use of the very small focused probe of a scanning x-ray microscope to stimulate visible light emission from dyes usually used in conventional fluorescence microscopy. Using a soft x-ray undulator source and a Fresnel zone plate to produce a focused x-ray probe, researchers at X1A have obtained 50–75 nm resolution images of the ZnS-based P31 phosphor grains shown here. The resolution was limited not by the focused x-ray probe (they have resolved 36 nm structures in the Scanning Transmission X-ray Microprobe (STXM)) but by dark noise and low net efficiency (70%) in the luminescence detection system used for this first investigation. This technique should make possible the imaging of dye-tagged sites of biochemical activity at 20–60 nm resolution in wet, unsectioned, and perhaps even initially living cells. A possible extension of the technique for 3D imaging at the transverse resolution of the x-ray microscope is described, where visible light collection optics might be used to obtain <500 nm axial resolution. C. Jacobsen, S. Lindaas, S. Williams, and X. Zhang (SUNY at Stony Brook).

Fig. 19 Top: Chemical and elemental composition of a microfabricated device as determined with the X1A Scanning Photoemission Microscope (SPEM). Elemental aluminum (a), silicon (b), and oxygen (c) are identified and mapped by selecting characteristic electron energies associated with each element in turn. Silicon atoms bound in silicon dioxide molecules (d) have an energy that is slightly shifted from the pure silicon level. In this way, two different chemical states of silicon have thus been distinguished. Bottom: Additionally, the instrument can acquire two images simultaneously by monitoring both (a) the sample current (representative of the total electron yield (TY)) and (b) the intensity of the electron energy analyzer (in this case the oxygen KVV Auger yield). Areas with low oxygen concentration appear dark in (b) but bright in (a). Note that there are streak-like features near the bottom of (a) which are not present in (b), which is attributed to changes in work function of sub-surface material. Some microelectronic gates are imaged in the top right corner of (a) and (b), although the gates themselves cannot be resolved owing to insufficient two-dimensional resolution (= 0.4 μm). The photon energy is 690 eV, the array size is 140 x 140, with a pixel size of 0.25 μm and a dwell time to 200 ms (images are median filtered). H. Ade, J. Kirz (SUNY at Stony Brook), E. Johnson, S. Hulbert (NSLS), E. Anderson (LBL), and D. Kern (IBM).
Tomography

Several interesting images were taken at X2 (Exxon), X23 (NIST), and X26. X2 researchers recorded images of calcium deposits in cartilage tissue (see Fig. 20), images of the spatial distribution of metal atoms in a supported catalyst system, and a study of the settling of particles in a heavy oil.

Synthetic calcite was imaged at X26C and compared with electron microprobe analysis. This evaluation of the usefulness of the synchrotron images will continue.

Finally, some exotic samples were imaged at X23, from crystals of triglycine sulfate, mercuric iodide and lead tin telluride grown in Spacelab III and on the Space Shuttle to synthetic diamonds. Also studied were nonlinear optical crystals of L-arginine phosphate which have a potential application in optical devices. In all cases images of superb contrast were obtained as can be seen in the abstracts.

Fig. 20 Microtomography investigation of the spatial distribution of calcium deposits in cartilage tissue at X2B. The sample was mounted in an epoxy substrate and cut to a cube approximately 1.5 mm on an edge, and was imaged at $h\gamma \approx 15 \text{ keV}$. Despite the low optical depth of the epoxy and tissue material, the relatively large mineral deposits required the high photon energy in order for them to be penetrated with sufficient signal-to-noise to permit reconstructions. The relative size and orientations of the deposits was sought. It is clear from the data that the Ca deposits varied up to sizes in excess of 150 $\mu$m. In addition, the roughly spherical particles appear to be arranged in a well defined pattern, much like beads on a string. Further experiments are planned to investigate the location of deposits relative to physical features such as the neighboring bone tissue. This figure is a volume rendering of the cartilage sample showing the tissue matrix (lighter color) and the Ca deposits, which are the dark nodules which appear to be strung together. K. L. D'Amico, J. H. Dunsmuir, S. R. Ferguson (Exxon), and C. J. Buckley (King's College, London, UK).
NUCLEAR PHYSICS

Andrew M. Sandorfi
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Beamline X5, the Laser Electron Gamma Source, has completed its second full year of operations — and a full year it was. Several significant improvements were made to the gamma-ray beam and to the experimental equipment for nuclear physics measurements. Many new collaborators have joined in making these improvements, and more importantly, in performing four separate major experiments during the past year.

The gamma-ray beam was produced for about 160 days. Of this total, approximately 20% was used for beam and detector development. The remaining 80% of the beam time was devoted to four experiments.

L3: Photodisintegration of the deuteron at energies from 200 to 320 MeV to study the role of the nuclear tensor force (38 days);

L4: Three body final states in the photodisintegration of $^3$He to determine the importance of three body forces (48 days);

L5: Neutral pion photoproduction on the proton to determine the deformation of the delta isobar, the first excited state of the nucleon (18 days);

L6: Three body photodisintegration of the deuteron to determine properties of the nucleon-delta interaction (28 days).

Experiments L4 and L6 are part of our outside-user program and were performed in collaboration with a group from Renssellaer Polytechnic Institute (RPI).

We have replaced the laser with a higher power version, so that it now delivers 7 watts of power in the ultraviolet from 333 nm to 364 nm, which produces gamma-rays up to 319 MeV, and 2 watts deeper in the ultraviolet from 300 nm to 336 nm, which produces gamma-rays up to 349 MeV. Operation at this higher energy is a new capability, and it will become a common operating mode for LEGS. The higher energy allows us to make measurements on and above the delta resonance, which will significantly enhance new measurements of the delta deformation planned for the coming year.

A new cryogenic target, capable of liquefying $^1$H, $^2$H, $^3$He, and $^4$He, was constructed and installed in the target area, principally by collaborators from the University of Rome and RPI (Annalisa D'Angelo, Carlo Schaerf, and Kamran Vaziri). An array of 48 plastic scintillator bars, 1.5 meters long, was mounted above the target by the University of Virginia (Richard Sealock, Steven Thornton, Richard Lindgren, and Cole Smith). The entire detector and target stand is mounted on rails so that it can be removed intact from the beam to allow experiments to be run in alternation, with minimal disassembly.

Keith Mize, the first graduate student to work at LEGS, received his PhD degree from the University of South Carolina this year. His thesis work (experiment L1) on the effect of the nuclear tensor force on beam-polarization observables in deuteron photodisintegration, was published in Physical Review Letters in September. Three other graduate students, all from Renssellaer Polytechnic Institute, have finished their thesis experiments at LEGS this year (experiments L4 and L6) and are now immersed in the analysis of their data. A visiting scientist from Grenoble, Dominique Rebreyend, is spending the year at LEGS to learn details of the construction and operation of the Compton backscattered gamma-ray beam, as well as collaborate in experiments. He has been working at ESRF on the design of a similar gamma-ray facility called GRAAL.

The major effort at beamline X5 for the next year will be a series of measurements to determine the deformation of the first excited state of the nucleon, the delta resonance at 320 MeV. Essentially all quark models of the nucleon invoke a tensor force between the quarks. This non-central force breaks the otherwise spherical symmetry of the nucleon, resulting in a deformation and static quadrupole moment for the delta. The delta is excited mainly by M1 photons, but if it has a quadrupole deformation, then it can also be excited by an E2 photon. The dominant decay channel is pion emission. Sensitivity to the small E2 component is enhanced by observing the photo-pion production with linearly polarized photons and determining the ratio $\frac{d\sigma_{E2}}{d\sigma_{M1}}$. (24)
of cross sections with the polarization parallel to and perpendicular to the reaction plane. Preliminary results of these measurements are shown in Fig. 21, along with previous data and the results of two independent calculations, each with and without a quadrupole deformation of the delta. The calculation of Davidson, et. al., which have approximately the correct energy dependence, can be brought into agreement with the data for a mixing ratio of about 4%. This would imply a larger deformation than expected in most models. Unfortunately, the agreement is not as good at other reaction angles. New experiments at X5 will provide improved determination of other parameters upon which the calculations depend, as well as further reducing uncertainties in polarization observables such as those shown in Fig. 21.

Nuclear physics research on beamline X5 is performed by a collaboration of physicists from BNL, INFN Frascati, Universita di Roma, University of South Carolina, University of Virginia, Virginia Polytechnic Institute and State University, Tel Aviv University, and Rensselaer Polytechnic Institute.

![Fig. 21. Ratio of cross sections for production of neutral pions by linearly polarized photons on hydrogen with the incident photon polarization parallel and perpendicular to the reaction plane. The solid circles are the average of two separate experiments, L2 and L5, made at LEGS (X5). The calculations demonstrate the sensitivity of the measurement to the quadrupole deformation of the delta resonance.](image)
(1) Angle-resolved photoemission spectroscopy continues to be one of the most important experimental techniques in the study of electronic structure of materials. During the past year, angle-resolved photoemission has been used to study the electronic band structures of a variety of bulk solids (U3, U7B, U12B, U16A), surfaces (U4A, U7B, U16A), adsorbate covered surfaces (U4A), thin metal overlayers (U7B, U16A), metal-semiconductor interfaces (U12B), as well as high temperature superconductors and related materials (U3). Spin-resolved angle-resolved photoemission also continues to provide important information on surface and interface magnetism. The following are a few representative examples:

(1.1) The bulk electronic band structures of two experimentally very demanding systems, Li (U12B) and Gd (U16A), were measured.

Along the $\Gamma \rightarrow N$ direction of the Brillouin zone of BCC Li, both the bandwidth and the shape of the dispersion were found to deviate strongly from the behavior predicted by the free electron model.

Better agreement with the experimental results was achieved by taking into account the exchange-correlation in the framework of the local density approximation (LDA). Almost exact agreement is achieved by further including the quasiparticle self-energy (see Fig. 22).

In the bulk band structure of Gd(0001), along the $\Gamma \rightarrow A$ direction, two bulk band states with predominantly $6s$ character were observed. The splitting between the two states is interpreted as a manifestation of indirect exchange between the nonoverlapping $4f$ states via conduction electrons. The temperature dependence of the splitting yields a critical exponent $\beta$ of 0.378, which agrees very well with the results obtained from the magnetization measurement of bulk Gd (see Fig. 23).

(1.2) Spin-resolved angle-resolved photoemission was used to study the magnetic and electronic structure of Fe/Ag interfaces. A spin-polarized interface state was observed in the Ag/Fe(001) system. It shows a distinct quantum size effect, a single new electronic state being observed for each layer, up to three atomic layers. These results may have important implications in the understanding of the oscillatory indirect exchange coupling between two ferromagnetic layers separated by thin layers of nonmagnetic transition metals (see Fig 24).
Fig. 23 (a) Normal emission electron energy distribution curves (EDCs) for Gd(0001) at a photon energy $h\nu = 38$eV as a function of temperature in the range $0.35T_c < T < T_c$, where the Curie temperature $T_c = 293K$. (b) Temperature dependence of the exchange splitting of the $-1.6$eV binding energy peak in (a). The solid line is $(1 - T/T_c)^\beta$, where the best fit critical exponent $\beta = 0.378$ agrees well with bulk magnetization measurements of Gd. B. Kim, A. B. Andrews, J. L. Erskine (U. Texas at Austin), K. Kim, and B. N. Harmon (Iowa State U.).

Fig. 24 Spin polarized (a) and spin integrated (b) angle-resolved photoemission spectra taken using beamline U5U at normal emission, $h\nu = 52$eV, and 70° angle of incidence, for Fe(001) epitaxially covered with 0, 1, 2, and 3 monolayers of Ag. The Fe(001) surface state in the clean Fe spectrum (indicated by arrow) evolves into the Ag/Fe interface state in the 1, 2, and 3 monolayer Ag/Fe spectra (indicated by arrows). N. B. Brookes, Y. Chang, A. Clarke, P. D. Johnson, and M. Weinert (BNL Physics Dept.). See Phys. Rev. Lett. 67, 354 (1991).
Valence band photoemission

There have also been a large number of angle-integrated valence band photoemission experiments reported. Among them, one of the most important developments is the extension of resonant photoemission to the soft x-ray region. There is also an increase in the use of the Cooper minimum in the photo-absorption cross section to selectively probe the valence band partial density of states of individual components in alloys. Finally, high resolution photoemission has been used to study the temperature dependent Kondo resonances in heavy Fermion systems.

Resonant photoemission has long been an invaluable tool in the study of the electronic structure of correlated systems due to its capability to identify different electronic configurations in the valence band photoemission spectra. In their study of CuO, the authors demonstrated that for 3d transition metal systems, carrying out the measurement near the 2p absorption edge instead of the more commonly used 3p absorption edge has several advantages which simplify the data interpretation. Among these advantages are larger resonant enhancement due to stronger absorption and larger spin-orbit splitting compared to the lifetime width of the core hole (see Fig. 25).

The prevailing theory of photoemission of heavy Fermion compounds is the Kondo impurity theory, which predicts a Kondo resonance in the spectrum located an energy $kT_K$ ($T_K$ is the Kondo temperature) above the Fermi level for the single 4f electron Ce compounds, and $kT_K$ below the Fermi level for the single 4f hole Yb compounds. The theory also predicts that the spectral weight and width of the resonance should scale with $T_K$. Temperature dependent high resolution photoemission studies of a number of Ce and Yb compounds were carried out by the Los Alamos group at U3. Contrary to previous findings, the authors argue that the observed temperature dependence in their spectra can be fully accounted for by the temperature dependent broadening of the valence band (phonons) and of the Fermi function (see Fig. 26).

Clearly, this is one of the areas that will benefit most from the continuing efforts in the development of high resolution monochromators.

A combined high resolution valence band photoemission and soft x-ray absorption study of the
(a) Divalent Yb 4f 7/2 core level in YbCu$_2$Si$_2$: data (dots), Lorentzian fit (solid line), and resolution broadened Kondo resonance model prediction (dashed line). (b) Temperature dependence of photoemission spectrum in (a). The U3C researchers conclude from this and other data that the Kondo resonance model fails in all of its major predictions: (i) the temperature dependence can be explained by Fermi function and phonon broadening, (ii) the lineshapes and widths are simple core-level-like and much broader than predicted, (iii) the valence is a constant function of temperature, and (iv) the spectral weight is independent of the Kondo temperature (not shown in this figure). A. J. Arko, J. J. Joyce, J. M. Lawrence, R. J. Bartlett, P. C. Canfield, Z. Fisk, and J. D. Thompson (LANL). See Phys. Rev. Lett. 68, 236 (1992).

Fig. 26 (a) Photoemission spectrum of the bulk Yb 4f 7/2 core level in YbCu$_2$Si$_2$: data (dots), Lorentzian fit (solid line), and resolution broadened Kondo resonance model prediction (dashed line). (b) Temperature dependence of photoemission spectrum in (a). The U3C researchers conclude from this and other data that the Kondo resonance model fails in all of its major predictions: (i) the temperature dependence can be explained by Fermi function and phonon broadening, (ii) the lineshapes and widths are simple core-level-like and much broader than predicted, (iii) the valence is a constant function of temperature, and (iv) the spectral weight is independent of the Kondo temperature (not shown in this figure). A. J. Arko, J. J. Joyce, J. M. Lawrence, R. J. Bartlett, P. C. Canfield, Z. Fisk, and J. D. Thompson (LANL). See Phys. Rev. Lett. 68, 236 (1992).

Fig. 27 Valence band photoemission (left) and C 1s absorption (right) spectra from well-annealed high quality K$_x$C$_{60}$, x=0.3,6. thin films grown under ultrahigh vacuum conditions at beamline U4B. Spectra taken at other values of x are found to be linear combinations of these three stable phases. The Fermi edge for the K$_3$C$_{60}$ film (left) is well defined and the density of states at the Fermi level suggests that this material may be a BCS-like superconductor. The C 1s absorption spectra (right) exhibit large non-rigid-band shifts as a function of x which, combined with the anomalous occupied band width (1.2eV) for K$_3$C$_{60}$, implies that there is significant mixing of the electronic states of K and C$_{60}$ in the superconducting phase (see Fig. 27).

(3) Core level photoemission

In core level photoemission, the sensitivity of the core level binding energy to oxidation state, chemical environment, and valence electron distribution has been used to monitor chemical reactions, and to study surface and interface electronic properties. These studies include semiconductor surface etching reactions (U8) and oxidation of tungsten and aluminum surfaces (U16B).

There were also an increasing number of high resolution core level photoemission studies performed during the last year. Higher energy resolution not only enables ever smaller core level shifts to be resolved, but it also makes possible the study of linewidth and lineshape in greater detail. For example, in a series of
core level photoemission studies
of alkali metals reported by U4A,
the thermal shift of core level
binding energies as well as the vi-
brational broadening of the core
levels were measured. The au-
thors found that for Na(110) the
width of the surface core level has
a larger temperature dependence
than that of the bulk core level,
indicating a smaller effective
Debye temperature for the surface
atoms, which is consistent with an
earlier proposal of the existence of
a soft mode perpendicular to the
surface (see Fig. 28).

(4) Auger-photoelectron
coincidence

Auger-photoelectron coincidence
spectroscopy (APECs) has been
demonstrated to have the follow-
ing unique advantages compared
to ordinary photoemission: re-
duction of core hole lifetime broad-
ing, elimination of uncorrelated
secondary background, better sur-
face sensitivity, and separation of
overlapping spectral features.

In a study of the lineshape of the
Si L_{2,3}VV Auger line of SiO_2
carried out by the Rutgers university
group (U14A), the major features
in the coincidence spectrum are
found to be reproduced quite well
by an earlier calculation, as shown
in Fig. 29. However, additional
spectral weight is observed near
68eV and in the threshold region
(87eV). The additional spectral
weight near threshold is assigned
to Auger transitions involving Si
orbitals with d character, whereas
the spectral weight near 68eV is
assigned to a correlation satellite
(see Fig. 29).

It should be noted that the coinci-
dence technique is essential in ar-
riving at these assignments. For
example, the alternative interpre-
tation that the additional spectral
weight near threshold is due to
substoichiometric oxide was ruled
out because the spectrum shown in
Fig. 29 included only Auger
electrons which were coincident
with 2p photoemitted electrons of
the stoichiometric oxide.

Fig. 28 Vibrational broadening of bulk
and surface core levels of Na(110) mea-
sured at beamline U4A. The square of the
measured Gaussian widths of the surface
and bulk Na 2p core levels (squares) are
plotted as a function of temperature from
77K to 230K. Above the Debye tem-
perature, the phonon contribution to the
core level width increases as T^{1/2} (solid lines).
The zero temperature intercept for the bulk
core level agrees well with the indepen-
dently known instrumental width, indi-
cating that inhomogeneous broadening con-
tributions are small. The surface core level
widths exhibit a small inhomogeneous con-
tribution and, more importantly, a larger
slope than the bulk core level, which im-
plies that the surface atoms have a smaller
effective Debye temperature than bulk
atoms. This data supports the model of
Jackson (Surf. Sci. 43, 431 (1974)) in which
a soft mode perpendicular to the surface
results in a reduced effective surface Debye
temperature. D. M. Riffe, G. K. Wertheim,
and P. H. Citrin (AT&T Bell Labs).

Fig. 29 Line shape of the Si L_{2,3}VV Auger
spectrum of stoichiometric SiO_2 measured
by Auger Photoelectron Coincidence Spec-
troscopy (APECs) using beamline U14A.
Upper figure: ordinary Si 2p core level pho-
toemission spectrum from a 7Å film of SiO_2
on Si(111). The data is well fit by a series of
chemically shifted Si 2p core levels associ-
ated with substoichiometric oxides as pro-
posed by Himpsel (Phys. Rev. B 38, 6084
(1988)). Lower figure: background sub-
tracted Auger spectrum of stoichiometric
SiO_2 obtained in coincidence with Si 2p
core electrons emitted only from stoi-
chiometric SiO_2, i.e. at 41.85±0.5 eV ki-
netic energy (hv = 150eV). This coin-
cidence Auger spectrum generally agrees
with the calculation (dotted line) of Ramak-
er (Phys. Rev. B 21, 4608 (1980)). The dis-
agreement near threshold is attributed to
Si orbitals of d-character participating in
the Auger transition and not, as previously
suggested, to substoichiometric oxide gen-
erated by beam damage (the latter is ex-
plicitly eliminated by measuring in
coincidence with only stoichiometric SiO_2).
R. A. Bartynski, A. K. See (Rutgers U.),
C.-C. Kao, and S. L. Hubert (NSLS).
(5) Time resolved photoemission

Photoemission from laser-excited states has long been the exclusive domain of laser pump, laser probe techniques. For the first time, excited state photoemission has been demonstrated using a laser pump, synchrotron probe technique at beamline X24C (see Fig. 30). The laser transiently populates a surface state pocket at \( \bar{X} \) in the surface Brillouin zone of GaAs(110), which can be observed as a peak at a kinetic energy of 5.7 eV using synchrotron radiation at \( h\nu = 8.4 \) eV. The broad tunability of the synchrotron probe at these photon energies makes this technique useful for excited state photoemission in a wide range of materials. The X24C researchers have also measured the time decay of laser-induced surface photovoltage (SPV) shifts of the Si 2p core level on Si(111) surfaces, and the elimination by the SPV of inhomogeneous broadening caused by band-bending nonuniformities on GaAs(110) surfaces (see Fig. 30).

![Fig. 30 Time resolved photoemission with nanosecond resolution, used to study laser excited states of semiconductors at beamline X24C. Much of these data are acquired during single bunch operations, which decreases by over an order of magnitude the mismatch between laser and synchrotron pulse repetition rates. Clockwise from upper left: (i) Time decay of the laser-induced surface photovoltage (SPV) shift of the Si 2p core level. Analysis of such decay curves has characterized surface space-charge dynamics and has revealed extremely large surface recombination rates for a variety of Si(111) surface preparations. (ii) Elimination by the SPV of inhomogeneous broadening caused by band-bending nonuniformities on GaAs(110). The narrowing of spectra recorded during the 5 ns laser pulse aided interpretation of an unexpected surface photodissociation discovered in these experiments. (iii) Excited state photoemission. Until now, exclusively the domain of laser-pump-and-probe experiments, excited state photoemission was demonstrated for the first time using a synchrotron probe. The peak at 5.7 eV originates from electrons which transiently photo-populate a surface state pocket at \( \bar{X} \) in the GaAs(110) surface Brillouin zone. J. P. Long, S. S. Goldenberg, J. C. Rife, and M. N. Kabler (NRL).
The past year was successful for X-ray absorption spectroscopy (XAS) at the NSLS in many ways: with rapid growth in both the number and variety of experiments performed, with advances in data analysis, and with the demonstration of several fundamentally new experimental techniques.

The experimental XANES and EXAFS work carried out by PRT's and by general users is summarized in the abstracts of beamlines X9A, X11A, X10C, X18B, X19A, X23A2, and X23B found in the second volume of this Annual Report. The number of physical and life science programs making use of XAS techniques is ever increasing, and the beam time allocation process through the general user proposal system is now highly competitive.

One example of the importance of XAS in biological systems is the determination of the function of the E7 amino acid (histidine) in mammalian myoglobin. Researchers at X9 have reported the structure of the myoglobin oxygen-binding active site in which the E7 histidine is replaced by tyrosine. They conclude from EXAFS and optical absorption data that tyrosine binds directly to heme in its ferric form and does not allow any ligands to interact with the myoglobin heme.

Another example of the importance of the XAS technique is its application to environmental research. Soil contamination by toxic heavy metals is a major and growing national environmental issue. DuPont Engineering researchers at X11 have used fluorescence EXAFS to determine that the lead in a typical environmental material, waste pond sludge containing 0.23% Pb by weight, is predominantly in the form of lead sulfide. A likely source of lead sulfide is the action of sulfate-reducing bacteria under anaerobic conditions. Lead sulfide is expected to present minimum hazard to the groundwater, but care needs to be taken that it is not converted to a soluble form by any other activity undertaken at the site.

As a whole, the field of x-ray spectroscopy continues to mature as a result of the international effort to define standards for experimental practice and data analysis, and individual efforts aimed at developing accurate theories for simulating experimental spectra. The FEFF program, developed by John Rehr and coworkers at the University of Washington, is now in regular use as part of the data analysis procedure. The program computes theoretical EXAFS spectra from first principle for an arrangement of atoms with essentially a single adjustable parameter: an overall amplitude scaling factor arising from many-body effects which is uncertain to within about 20%. In addition to permitting studies to be performed on systems where no experimental standards are available, experience has shown that analysis using an FEFF calculated standard is more reliable than one using an experimental standard which is structurally or chemically too different from the unknown. Efforts are underway to extend FEFF to perform multiple scattering calculations and thereby accurately simulate spectra for atoms beyond the first coordination shell.

One of the new experimental techniques demonstrated at the NSLS during the past year overcomes one of the fundamental stumbling blocks for high resolution XANES spectroscopy: the broadening arising from the core hole lifetime. In standard XANES spectroscopy, the limited lifetime of the core hole produced in the photon absorption process results in an uncertainty in the energy of the final state and hence a smearing of the edge features in the absorption spectrum. This effect becomes more pronounced as the atomic number of the absorber increases. The new high-resolution XAS technique developed by K. Hamalainen, J. B. Hastings, D. P. Siddons, and L. E. Berman (NSLS) using X25 is based on high resolution analysis of the fluorescence radiation emitted when the core hole decays, as represented by the diagram in Fig. 31.

By counting only fluorescent photons which have energies within a narrow range, the energy of the initial core hole (and also, via energy conservation, the energy of the excited electron) is fixed. The longer lifetime of the secondary hole created in the fluorescence process results in a substantial reduction in
Fig. 31 Schematic representation of high-resolution X-ray fluorescence spectroscopy as compared to standard (low resolution) fluorescence measurements. In the high resolution fluorescence measurement, only a small energy slice of the emitted fluorescence radiation is accepted. By conservation of energy, this improves the energy resolution of the X-ray absorption measurement.

The remarkable improvement in the structural detail evident in edge regions is represented in Fig. 32, which compares normal and high-resolution XANES measurements for the Dysprosium L(III) edge.

The technique has also been used to perform spin-polarized measurements on anti-ferromagnetically coupled Mn samples.

Another experimental technique developed in the past year is diffraction anomalous fine structure spectroscopy (DAFS). In DAFS spectroscopy, developed by Charles Bouldin and coworkers at NIST and Larry Sorensen, J. J. Rehr and coworkers at the University of Washington (see abstracts for X23A2, and also independently X10C), the intensity modulations in a diffraction peak are measured as the incident energy is varied through and well past an absorption edge of one of the elements contributing to the structure factor. The experimental geometry, shown in Fig. 33, is similar to a conventional diffraction experiment, but with an energy-scanned incident beam.

Fig. 32 The energy resolution of XAS has for the most part been limited by the natural lifetime broadening of the core hole. If, however, the energy of the fluorescent photons produced in the decay of this core hole can be determined with a resolution narrower than the lifetime width, the core hole lifetime broadening can be overcome. To realize this, a high-resolution fluorescence spectrometer, based on a backscattering spherically-bent silicon crystal in a Johann geometry, was constructed and tested at X25. Shown above is the observed XANES spectrum of a Dy(NO₃)₂ sample at the Dy L(III) edge, for which the analyzer window was centered on the Dy L₂,₃ fluorescence line. A conventional transmission XANES spectrum of the same sample is shown for comparison. The enhanced resolution using the backscattering analyzer reveals fine structure which is totally invisible in the conventional spectrum. K. Hämäläinen, D. P. Siddons, J. B. Hastings, and L. E. Berman (NSLS); see Phys. Rev. Lett. 67, 2850 (1991).
Fig. 33 The experimental setups for EXAFS (transmitted, or forward scattered signal) and DAFS (0,26 diffracted signal) spectroscopies.

The observed intensity variations in a 111 reflection from a copper crystal are shown in Fig. 34 (a), superimposed upon the absorption spectrum.

The region after the edge is seen to contain oscillations analogous to EXAFS, and indeed the same Fourier transform magnitude (Fig. 34 (c)), is obtained after subtraction of a smooth background. But the oscillations are, in fact, 90° out of phase (Fig. 34 (b)) with the EXAFS oscillations since the absorption is linked to scattering via a Kramers-Kronig transform.

The significance of DAFS arises from the fact that it generalizes EXAFS from an energy scanning spectroscopy to one that uses both photon momentum and photon energy simultaneously. This double tunability retains the chemically selective character of EXAFS, while adding several important new capabilities. For instance, it is possible to use different diffraction peaks to study separately each phase of a multi-phase material. This has been demonstrated by a study of a multilayer system of In$_x$Ga$_{1-x}$As in which the structure of buried layers of different In composition (and hence, different lattice constants) was determined using DAFS measurements at three different photon momentum transfers, Q. In addition to macroscopic spatial selectivity, DAFS also extends the elemental selectivity of EXAFS to include site selectivity, i.e., by using DAFS spectra with different photon momentum transfers, it is possible to separately determine the short-range order about crystallographically inequivalent, but chemically equivalent atoms. This has been demonstrated by DAFS measurements of the two inequivalent sites of Cu atoms in the 123 high T$_c$ superconductors. The addition of site selectivity to EXAFS has long been sought, but has never been satisfactorily achieved until the use of DAFS. Work on X10C shows that DAFS measurements can be made on powder samples, which will lead to applicability of this method to a very wide range of materials.
Fig. 34 Superposition of the EXAFS and DAFS spectra from a copper crystal: a) Raw data; b) Isolated first shell oscillations illustrating π/2 phase shift; and c) Fourier transforms. J. O. Cross, H. Straigier, L. B. Sorensen (U. of Washington), C. E. Bouldin, and J. Woicik (NIST).
X-ray scattering and crystallography research at the NSLS is performed on two-thirds of the X-ray Ring experimental stations, and comprises about half of the x-ray experimental abstracts in this Annual Report. Glancing incidence studies of surfaces and interfaces, magnetic scattering studies of anti-ferromagnetic and heavy-Fermion systems, powder diffraction studies of new complex materials such as Fullerenes and high-T_c super-conductors, precession crystallography studies of protein structure, and small-angle scattering studies of polymers and other amorphous materials and liquids continued to predominate in FY91, while new scattering methods such as intensity fluctuation spectroscopy ("speckle" diffraction), diffraction anomalous fine structure (DAFS), and time resolved Laue-diffraction protein crystallography emerged.

In an experiment by M. Sutton et al. at X25, intensity fluctuation spectroscopy was carried out for the first time using hard x-rays. The method involves illuminating a sample with a coherent light beam. Different phase shifts are introduced into components of the beam scattered by different regions of the sample, resulting in a speckled scattered beam image. By studying how the image changes with time, the time evolution of the arrangement of the scattering regions can be determined. In the X25 experiment, the sample was a Cu_3Au crystal which had been quenched from the disordered to ordered phase. The scattered beam was the (100) Bragg peak, which is not allowed for the disordered phase, and the scattering regions were the ordered antiphase domains. Intensity fluctuations were observed in a portion of the Bragg peak image, which arose from the growth of the domains following the quench (see Fig. 35).

The Diffraction Anomalous Fine Structure (DAFS) technique is analogous to Extended X-ray Absorption Fine Structure (EXAFS), in that short-range

![Fig. 35](image)

**Fig. 35** When a coherent light beam illuminates a disordered sample, components of the beam scattered by different regions of the sample can have different phase shifts, introducing (in the far field) a speckled diffraction image that results from the constructive and destructive interference of the beam components. By observing how the image changes with time, it is possible to determine the time evolution of the arrangement of the scattering regions. Alternatively, the intensity variation of a single point of "speckle" in the image gives information on the time correlations of the sample inhomogeneities. This technique, called intensity fluctuation spectroscopy, was demonstrated for the first time with hard x-rays at X25. Shown above is the intensity of a single speckle in a Cu_3Au(100) Bragg peak using a coherent incident beam, following a rapid temperature quench from the disordered phase (for which the (100) peak is not allowed) to the ordered phase at time zero. The time evolution of the growth of ordered antiphase domains gives rise to the observed intensity fluctuations. Had an incoherent incident beam been used, a purely monotonic increase in intensity (without any fluctuations) would have resulted. M. Sutton, E. Dufresne (McGill U.), S. G. J. Mochrie (MIT), L. E. Berman (NSLS), G. Hald, and G. B. Stephenson (IBM).
structural information can be extracted by Fourier-transforming an anomalous-scattering dependent signal, recorded as a function of photon energy above an absorption edge. In the case of EXAFS, the signal of interest results from x-ray absorption, and is directly related to the imaginary part of the anomalous scattering amplitude. In the case of DAFS, the signal of interest is a Bragg peak intensity, which contains contributions from both the real and imaginary parts of the scattering amplitude. Experiments by Bouldin et al. at X23A2 and Pickering et al. at X10C illustrated how DAFS can be used to obtain site-selective short-range structural information that is hard to come by using EXAFS. (See also X-ray Absorption Spectroscopy in this section.)

Interest in Laue-diffraction crystallography has revived. In this technique, the continuum of x-ray wavelengths from the source, rather than a single wavelength, is made available for use. The sample selects unique wavelengths to diffract, based on its structure and orientation, and the pattern is recorded on film or an image plate. In principle, only a few short exposures are required to obtain enough information to determine the crystal structure, since all of the x-ray intensity radiated by the source is used, and each exposure can sample a large portion of reciprocal space. The technique is ideal for studies of dynamic processes in large unit cell samples, such as protein crystals, for which an enormous number of reflections are required to determine the structure. The short exposure times can also allow full data sets to be recorded before the sample suffers from radiation damage. Prototype Laue diffraction measurements of the response of a crystallized enzyme, trypsin, to a pH jump, performed by Singer et al. at X25 and X26C, revealed subtle structural changes. Typical exposure times varied from 25 msec at X25 to 800 msec at X26C.

Surface and interface reflectivity and diffraction studies have been especially popular at the NSLS. For example, Gibbs et al. at X22C have used high resolution surface x-ray diffraction to study details of the temperature-induced surface reconstructions of Pt(001) and Au(001) (see Fig. 36).

Complementary truncation rod reflectivity (Sandy et al., X20A)
and surface diffraction (Grübel et al., X22C) measurements of the high-temperature reconstruction of the Pt(111) surface point to a compressed incommensurate surface layer consisting of regions of ideal and faulted stacking (see Fig. 37).

Surface diffraction measurements of the structure of K monolayers on a Cu(100) surface by Meyerheim et al. at X16A revealed a continuous change in the incommensurability of the overlayer with time following the initial deposition, which may have been caused by desorption, clustering, or contamination. Diffraction measurements of the Ag/Si(111) buried interface by Hong et al. at X14A confirmed the preservation of the initial Si(111) 7X7 reconstruction following room temperature deposition of a thick Ag film; the reconstruction disappeared after annealing the interface. In experiments by Tidswell et al. at X16B, diffraction and reflectivity were used to learn about the structure and roughness of both the liquid-solid and liquid-vapor interfaces for a hydrocarbon film on a Si wafer. Electrochemical electrode surfaces were studied with x-ray diffraction and reflectivity by You et al. at X10B, Toney et al. and Wiesler et al. at X20A, Armstrong et al. at X20B, and Ocko et al. at X22B/X25 (see Fig. 38).

Real-time studies of film growth on surfaces have also been performed using x-ray diffraction: Au growth on Si was studied by Chiarello et al. at X6B and X22B, Pd$_2$Si growth on Si(111) was examined by Bennett et al. at X16A, and Xe growth on Ag(111) was studied by Wang et al. at X18A. Anomalous reflectivity methods were employed to learn about film structure by Bai et al. at X6B and Sanyal et al. at X22C. The x-ray standing wave method was used to study alkali overlayer registry on Si(111) by Lagomarsino et al. at X15A, high-$T_c$ film quality by Zegenhagen et al. at X15A, rare gas adsorption on a graphite single crystal by Swanson et al. at X24A, and the InP(110) surface reconstruction by Woicik et al. at X24A.

Thermal diffuse x-ray scattering continued to be employed for strain and precipitation studies in alloys and compounds, by Butler et al. and Larson et al. at X14A, and Xe growth on Ag(111) was studied by Wang et al. at X18A. Anomalous reflectivity methods were employed to learn about film structure by Bai et al. at X6B and Sanyal et al. at X22C. The x-ray Standing wave method was used to study alkali overlayer registry on Si(111) by Lagomarsino et al. at X15A, high-$T_c$ film quality by Zegenhagen et al. at X15A, rare gas adsorption on a graphite single crystal by Swanson et al. at X24A, and the InP(110) surface reconstruction by Woicik et al. at X24A.
and Mahadev et al. and Na et al. at X18A. A recent application was for short-range order and correlation determinations in liquid polymers, by Zhao et al. at X22B and X22C.

Magnetic x-ray scattering has been gaining more interest at the NSLS. Highlighting this field was the observation of critical magnetic scattering from the spiral antiferromagnet Ho in the vicinity of its Neél temperature, by Thurston et al. at X22C (see Fig. 39).

C\textsubscript{60} Buckminster-Fullerenes ("Bucky-balls") are of great interest at the moment, and some interesting powder diffraction studies have been performed at the NSLS. Heiney and co-workers studied orientational ordering phase transitions in C\textsubscript{60} using the high-resolution capabilities of X7A. The high resolution and good signal-to-noise allowed detailed intensity measurements to high orders, enabling the identification of two phases, one in which the C\textsubscript{60} molecules had random orientation within the lattice, and another below 250K whereby the molecules were ordered in sets such that they were rotated about local <111> axes by about 25° (see Fig. 40).

Another Bucky-ball experiment made use of the powder diffraction capability of X3A2. There, Stephens and his colleagues studied the structure of Fullerenes which were doped with alkali metals. They were able to show that in the K-doped C\textsubscript{60} (which is known to superconduct), the K ions fit into interstitial sites, where they constrain the orientation of the C\textsubscript{60} into the two possible states having eight hexagonal rings facing <111> directions. In contrast to the pure C\textsubscript{60} discussed above, rotational disorder was shown to persist at least down to 11K. Other dopants were also studied (see Fig. 41).

A recent example of "surface powder diffraction" was carried out by Heald and Jayanetti on X11A. They were able to follow the crystallization of a 2000 Å film of Ge covered with 1000 Å of Al. The presence of a metal layer is known to reduce substantially the crystallization temperature of amorphous Si and Ge. Glancing-angle powder diffraction, reflectivity, and EXAFS allowed this recrystallization process to be followed in a time-resolved manner, giving insight into the crystal nucleation and growth mechanisms (see Fig. 42).
Fig. 40 Molecular structure of Buckminster-fullerene, C\textsubscript{60}. High resolution powder diffraction experiments carried out in a University of Pennsylvania - Brookhaven collaboration at beamline X7A have revealed the existence of an orientational ordering transition in C\textsubscript{60} at 250K and an analogous transition in C\textsubscript{70}. P. A. Heiney, J. E. Fischer, A. R. McGhie, W. J. Romanow, A. N. Denenstein, J. P. McCauley Jr., A. B. Smith (U. Penn.), and D. E. Cox (BNL Physics Dept.).

Fig. 41 The crystal structure of the superconducting Buckminster-fullerene compound, K\textsubscript{2}C\textsubscript{60}. The raspberries are C\textsubscript{60}, and the smaller isolated spheres are potassium cations, with different colors (see cover) in different crystallographic sites. This structure was solved by Peter Stephens and coworkers at SUNY and UCLA, from powder diffraction data taken at beamline X3. P. W. Stephens, L. Mihaly (SUNY at Stony Brook), F. Diederich, K. Holczer, S.-M. Huang, R. B. Kaner, R. L. Whetten, J. B. Wiley (UCLA), and P. L. Lee (SUNY at Buffalo). See Nature, 351, 632 (1991).

Synchrotron radiation is often quoted as being especially beneficial when samples are small and contained in difficult environments. A good example of this type of application is the high-pressure, high-resolution experiments performed by Jephcoat and coworkers using X7A. The combination of a horizontally-focussing monochromator and a linear position-sensitive detector resulted in moderately high flux together with efficient detection. The study illustrated, among other things, the crucial importance of the use of a pressure-transmitting fluid (in this case helium) in the reduction of strain gradients in the sample, and hence the ability to perform meaningful high-resolution measurements (see Fig. 43).
Another often-quoted advantage of synchrotron radiation is its tunability, and the potential to use anomalous scattering as a way to record complex diffraction patterns. An interesting application of this idea was pursued by Warner et al. at X7A. Their aim was to try to distinguish the scattering within a molecule by atoms of the same element but with different valences. As is well-known, the absolute position of x-ray absorption edges is sensitive to the chemical state of the absorbing species. Thus, very close to the nominal edge, different valence ions will have different anomalous scattering factors. Warner and his colleagues were able to demonstrate that scattering factor differences of from one to two electrons between Fe$^{11}$ and Fe$^{111}$ in α-Fe$_2$PO$_5$ were observable.

As in other areas, small-angle x-ray scattering with synchrotron radiation has concentrated on two aspects, the ability to make time-resolved measurements and to obtain high resolution. Examples of time-resolved measurements are the experiments on phase separation and crystallization kinetics in polymers performed on X3A2 by Chu and his colleagues. In addition to small-angle scattering, wide-angle diffraction can be performed simultaneously on this instrument, which allows correlations of the behaviors in both microscopic and macroscopic scale lengths. Such studies can lead to better understanding of these industrially-important materials and processes. In a similar vein, the experiments by Shen and collaborators at X12B to determine the phase diagram of a microemulsion involving a surfactant, decane, and water using small-angle x-ray scattering showed its power to unravel a complex and untrackable problem typical of those found in industrial processes.

Fig. 42 X-ray diffraction data obtained at beamline X11A.B from a 2000 Å amorphous Ge thin film covered with 1000 Å of Al, while annealing at 162°C. On the left is the raw diffraction data at time zero and three annealing times. On the right is the intensity of the Ge(111) reflection plotted versus the square root of the annealing time. After an initial nucleation period the Ge diffraction intensity follows a $t^{1/2}$ time dependence. This indicates that after the reaction moves away from the interface it is diffusion limited, likely due to the need for the metal atoms to diffuse to the crystallization front. The enhanced diffusion caused by interaction of the semiconductor (Ge) and the metal (Al) substantially reduces the amorphous Ge crystallization temperature. S. M. Heald and J. K. D. Jayanetti (BNL Dept. of Applied Science).

Fig. 43 High resolution, high pressure power diffraction results for ferroelectric LiNbO$_3$ at 12 GPa from beamline X7A. The importance of a pressure medium is shown by the order of magnitude increase in line width obtained without solid helium as a medium. Use of a smaller collimator (0.020 mm pinhole) does not result in narrower peak profiles, indicating that pressure gradients are not the dominant factor in lineshape at this pressure. A. P. Jephcoat (Oxford U.), L. W. Finger (CIW), and D. E. Cox (BNL Physics Dept.).
Instruments such as those at X3A2 and X12B routinely achieve resolutions down to q-values of $10^{-2}$ Å$^{-1}$. There is a region below this which is inaccessible to such instruments. The device at X23A3 is designed to access the region $10^{-5}-10^{-1}$ Å$^{-1}$ using crystal collimators (the Bonse-Hart geometry) (see Fig. 44).

The studies of microporous foams by Olivier et al. is an example of an application of the instrument to learn about correlations in the length range normally considered the realm of visible light scattering. Of course, x-rays have the big advantage that they can address non-transparent samples, which makes the high resolution small-angle x-ray scattering technique much more generally applicable.

It is clear that the wealth of interest evident in x-ray scattering and crystallographic studies at the NSLS will ensure their continued predomination among the materials characterization tools in the future.

Fig. 44 Small Angle X-ray scattering (SAX) spectrum from beamline X23A3, plotted on a log-log scale, of a microporous foam. B. J. Olivier, D. W. Schaefer (Sandia Nat. Lab.), S. Krueger and G. Long (NIST).
M. Dudley and his group, in collaboration with D. Paine from Brown University and R. N. Sacks from United Technologies Research Center, have applied the technique of topographic imaging in grazing Bragg-Laue geometries, recently developed on X19C, to the analysis of defects as a function of depth in a 200nm thick In$_x$Ga$_{1-x}$As strained layer system, achieving a depth resolution better than 10nm. This level of resolution is unprecedented in topography.

On beamline X23A3 David Black and Harold Burdette studied the microstructure of natural and man-made diamond. The relationship between growth conditions and defect microstructure of man-made diamond was investigated. It was found that the quality of the grown crystals varies dramatically from the seed end to the top of the crystal. The quality of type Ia and IIa natural diamond is being studied to determine the best quality diamond for use as substrates for homoepitaxial growth of CVD diamond films. The relationship between the microstructure of these natural substrates and the quality of the CVD films is being studied.

H. H. Schloessin, R. D. Spal, and R. A. Secco have used the hard x-ray microscope developed at X23A3 to investigate pressure or percussion figures in single crystals of calcite. These figures, which characterize the cohesive strength of the crystal as dependent upon structure, are created by dynamic loading which was applied to the crystal with a newly developed indentor mounted to the microscope. A complete set of diffraction images was obtained for one crystal from the point of touch down of the indentor to fracture. These images showed that the pressure figure consists of a twinned lattice.

M. Dudley and his group on beamline X19C, in collaboration with Julia Phillips' group of AT&T Bell Labs, have made considerable progress in their project relating to the non-destructive characterization of crystals which are candidates for use as substrates for high $T_c$ superconductor epilayers. Twin operations in LaAlO$_3$ single crystals were solved. In addition the second order phase transition at 435°C was studied in situ (see Figs. 45 and 46).

M. Dudley and his group on beamline X19C, in collaboration with Bruce Foxman of Brandeis University and William Jones of Cambridge University, have made significant progress in the characterization of defects in $p$-terphenyl single crystals. Complete dislocation and twin analysis has been performed. Following this, the low temperature monoclinic to triclinic phase transition at 130°C was studied in situ.

M. Dudley and his group on beamline X19C, in collaboration with D. Hodul of Varian Research Center, made observations of dislocation motion in high carbon content Si induced by rapid thermal processing. Nucleation of dislocations at precipitates was observed, followed by combined glide and climb.

M. Dudley and his group on beamline X19C, in collaboration with the group of I. Baker at Dartmouth, have continued to make significant progress on their project concerning the examination of ice single and polycrystals. In situ deformation experiments have been performed on bicrystals which shed much light on deformation mechanisms in polycrystalline ice. In particular the nucleation of dislocations at grain boundaries was observed.

M. Dudley and his group on beamline X19C, in collaboration with David Larson of Grumman Aerospace Corporation, have made significant breakthroughs in their project concerning the characterization of growth defects in CdZnTe single crystals. A new method for the characterization of twin operations has been developed. Similarly, progress has been made in the characterization of MBE grown CdTe in collaboration with Bob Silberstein, Myung Lee and Don DiMarzio of Grumman.
M. Dudley and his group on beamline X19C, in collaboration with C. Fazi of U.S. Army Harry Diamond Laboratories, and D. Gordon-Smith of University of Warwick, continue to make significant progress in their studies of the distribution of breakdown damage in epitaxial silicon p-n junctions. This has enabled significant contributions to be made to the understanding of the fundamentals of the breakdown process for the cases of forward and reverse bias, as well as for combinations of these.

M. Dudley and his group on beamline X19C have made progress in a new project involving high temperature deformation studies on InSb. Dislocation configurations around high temperature indentations afford much information on the relative mobilities of screw and 60° dislocations in this material.

Figs. 45, 46 Synchrotron White Beam Reflection Topographs, $g=220$, $\lambda=0.98\AA$, recorded from a LaAlO$_3$ single crystal as it undergoes a second order, rhombohedral-to-cubic phase transition at 435°C. The temperature increases from 100°C to 435°C in Fig. 45 and decreases from 325°C to 35°C in Fig. 46. Note the (101) mirror twins in the room temperature rhombohedral phase (Fig. 45 (a)). These twin planes become mirror planes in the cubic phase so that the twins disappear as shown in Fig. 45 (d). As the temperature is subsequently lowered, these twins reappear as seen in Fig. 46 (a). Note that the twinning density is lower following a single cycle through the transition (Fig. 46 (d)).
Section II

Symposia, Workshops and Projects
NSLS USERS’ MEETING

Mark Rivers
U. of Chicago
UEC Chairman

The twelfth NSLS Annual Users’ Meeting was held on May 20–22, 1991. The first day was devoted to five workshops, which were attended by a total of 221 people. The workshop topics included:

Atomic and Molecular Science, chaired by Brant Johnson of Brookhaven National Laboratory. The workshop topics included photoionization studies of atomic ions and molecules, high resolution molecular core level photoelectron spectroscopy and studies of chemistry induced by core electron excitation.

Computational Tools in XAFS, chaired by Tim Morrison of the Illinois Institute of Technology. This workshop covered the use of \textit{ab initio} codes in modeling XANES and EXAFS data, standardization and criteria in EXAFS analysis, and ancillary tools in EXAFS analysis. The workshop concluded with a caucus on time-resolved EXAFS in the U.S.

Surface Structure, chaired by Doon Gibbs of Brookhaven National Laboratory. This workshop focused on x-ray surface scattering studies. Talks included topics such as the phase behavior of Au and Pt surfaces, phase transitions of surface adlayers, and organic monolayers on water.

The main Users’ Meeting began on Tuesday morning and was attended by 312 people. The invited keynote speaker, U.S. Congressman George Hochbrueckner, was unable to attend because of a Congressional session, but he sent a letter which was read by BNL Deputy Director Marty Blume. Hochbrueckner expressed his enthusiasm and support for such NSLS projects as x-ray lithography, coronary angiography, x-ray microprobe and microtomography and studies of catalysts using x-ray scattering and photoemission spectroscopy.

Denis McWhan, the chairman of the NSLS, outlined the achievement of the NSLS in the last year and plans for the future. A total of 1,950 users conducted experiments on the 29 beamlines on the VUV Ring and 56 beamlines on the X-ray Ring. The upgrades to the storage rings completed this year include the addition of a global feedback system to stabilize the vertical beam position in the X-ray Ring. Horizontal feedback will be accomplished in the coming year with the arrival of new pick-up electrode receivers. Upgrades in the accelerator control computer systems and the injection system are also planned or underway. McWhan outlined steps which have been taken to improve the working environment for NSLS users. These include improvements in electronic communications and a decision to create a comfortable user lounge and library. He announced that a decision had been made to increase from one to three the number of Proposal Study Panels which review General User Proposals for experiments on the X-ray Ring. The new panels will cover the fields of Scattering/Crystallography, Spectroscopy and Imaging/Other. McWhan also noted that the Insertion Device Beamlines are now reaching routine operations and are preparing to accept general user proposals. Users are encouraged to begin submitting proposals for experiments to be done on the U5 undulator, the U13 wiggler, the X1 undulator, the X13 miniumulator, the X17 superconducting wiggler and the X25 hybrid wiggler. McWhan also discussed the issue of user fees which are being studied by the U.S. Department of Energy. These would require all users to

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pay for beam time at facilities such as the NSLS. McWhan stated that “if user fees are instituted it could kill DOE user facilities such as the Light Source. We are working with other facilities to make sure our arguments are heard.” The topic of user fees was discussed further at the round table forum on Tuesday afternoon. Users were encouraged to make their feeling about this issue known in Washington. The NSLS Users’ Executive Committee has written a letter to Admiral Watkins explaining their opposition to the proposed fees.

The scientific talks at the meeting included two talks on X-ray lithography. Jim Murphy discussed the status of the Phase I X-ray Lithography Source at the NSLS. This warm compact ring has met all of its design specifications with a maximum stored current of 1200 mA at 200 MeV. Currents as high as 250 mA have been achieved when injecting at the lower energy of 77 MeV. The superconducting magnets for the Phase II cold ring are now being fabricated. Rick Freeman of AT&T Bell Labs reported on progress in x-ray projection lithography at the NSLS. Freeman considers projection lithography to be a promising technique for producing circuits with features around 100 nm, which he believes will be required sometime after the year 2000 when chip densities reach 1 Gbit. Projection lithography uses x-ray optics to produce a demagnified image of the mask on the photoresist. Freeman stated that while the technique is very promising it will require an enormous investment to become commercially viable.

D. Peter Siddons discussed recent results of Mössbauer spectroscopy using synchrotron radiation. By using a very high resolution monochromator, his group has, for the first time, been able to do Mössbauer spectroscopy using synchrotron radiation on samples which were not perfect crystals. The advantages of synchrotron radiation for Mössbauer studies include its polarization and high intensity, which should permit the study of small samples. It also permits one to study nuclei which do not have a radioactive parent. Using existing synchrotron sources, however, it is only possible to study samples which have a very high concentration of the isotope of interest (such as isotopically enriched foils). The hope is that by using third generation synchrotron facilities it will be possible to do Mössbauer studies of nuclei at natural abundances.

Other talks covered soft x-ray spectroscopy of metal enzymes, coronary angiography, observation of speckle with x-rays, nuclear physics at the LEGS beamline, scattering from bulk liquids and thin wetting layers, photoelectron holography and time resolved studies of heterostructure interfaces.

Four new members were elected to the Users’ Executive Committee. These are Paul Cowan (Argonne National Laboratory), C.T. Chen (AT&T Bell Labs), Gabrielle Long (NIST) and Larry Sorenson (Univ. of Washington). Neal Shinn was elected as Vice-Chairman, and will become Chairman at the Users’ Meeting in 1992.

The meeting banquet was held on Tuesday night in Berkner Hall. Special awards were made this year in honor of two recent retirees from the Department of Energy. Chalmers Frazer was thanked for his years of service both at BNL where he served as a senior physicist and Deputy Chairman of the Physics Department, and in the Department of Energy where he served as Chief of the Solid State Physics and Materials Chemistry Branch. Don Stevens was honored for his tremendous efforts in the Department of Energy which were critical in the development of both the current generation of synchrotron facilities, such as the NSLS, and also in starting the next generation, such as the Advanced Light Source at Berkeley and the Advance Photon Source at Argonne.

Other highlights of the meeting included a poster session, in which over 60 posters were presented, displaying research at the NSLS. A vendor display included 18 companies with products ranging from vacuum equipment to multilayer crystals to X-Windows terminals.
WORKSHOP ON SURFACE STRUCTURE

Doon Gibbs
BNL Physics Department

X-ray diffraction and reflectivity studies of solid and liquid surfaces and interfaces are a central component of the scientific program at the NSLS. The workshop on surface structure sampled the diversity of experiments now being performed on the floor of the X-ray Ring. Approximately seventy-five persons attended the one day meeting. The program follows:

I. Session Chair: Jacqueline Krim, Northeastern University

In Situ Surface X-ray Scattering of Electrochemically Deposited Metal Monolayer

Structure of Electrified Interfaces

M. Toney, IBM

B. Ocko, BNL

II. Session Chair: A.D. Navaco, Lafayette College

Phase Transitions of Surface Adlayers

High Temperature Studies of Metal Surfaces

Phase Behavior of Au and Pt Surfaces

K. Liang, Exxon

E. Conrad, Univ. of MO

G. Grübel, BNL

III. Session Chair: Lonny Berman, NSLS

Standing Wave Studies of X-ray Semiconducting Surfaces and Interfaces

2D Solid and Liquid Pb on Si(111)

Coherence and Phase Separation of Steps on Si(111)

Steps and Step Bunching After Homoepitaxy on Si(001) Surfaces

P. Cowan, NIST

E. Fontes, AT&T

K. Blum, MIT

P. Fuoss, AT&T

IV. Session Chair: Sunil K. Sinha, Exxon

Organic Monolayers on Water

Complete Wetting of a Rough Surface

2D Order Kinetics: Ising System in a Dilute Random Field

P. Dutta, Northwestern Univ.

I. Tidswell, Harvard

P. Eng, SUNY at Stony Brook

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A day-long workshop dealing with the area of electronic and chemical phenomena at surfaces was held in conjunction with the 1991 Annual Users Meeting. The goal of the workshop was to present, in a relatively informal manner, new results in this field obtained at the NSLS. The workshop consisted of a series of invited talks from members of the NSLS user community. The talks were targeted at a general surface physics and chemistry audience, and the schedule allowed plenty of time for questions and answers. This format worked very well. This meeting complemented a parallel session on surface atomic structure held at the same time.

The first technical presentation of the day was a lively talk given by Peter Johnson (BNL - Physics, U5), who spoke on spin resolved photoemission studies of adsorption on ferromagnetic surfaces. With the audience now wide awake, the next talk was by C. T. Chen (AT&T - Bell Labs, U4B) who presented a fascinating talk on the use of soft x-ray magnetic circular dichroism as a tool for studying magnetic phenomena at the surfaces of crystals and thin films. (This talk provoked a vigorous question period!). Moving further into the area of surface adsorbates, the third talk of the day was by Gwyn Williams (BNL - NSLS, U4IR) who discussed the use of synchrotron generated infrared radiation in the study of the coupling between adsorbate vibrational modes and the electronic structure of a metal surface. Following a short coffee break, Neal Shinn (Sandia National Lab, U3/U16) brought the proceedings firmly into the area of surface chemistry with a discussion of the chemical properties of epitaxial metal overlayers. Jory Yarmoff (University of California at Riverside, U8) gave the final talk before lunch. Continuing the chemistry theme, his was a thought provoking presentation concerning the halogen etching of semiconductors. Following lunch, the proceedings resumed with an almost philosophical discussion by Steve Kevan (University of Oregon, U4A) of time and energy scales at surfaces. The difficult role of following Steve fell to Paul Bruhwiler (University of Pennsylvania, U12) who revisited the enduring topic of core level spectra from adsorbates on simple metal surfaces. Jack Rowe (AT&T Bell Labs, U4A) continued the core level discussion by speaking next on the electronic structure of Si surfaces and interfaces. Following another break for caffeine, the penultimate talk was presented by Dave Heskett (University of Rhode Island, U12), who discussed the structure of Bi overlayers on III/V semiconductor surfaces. The technical section of the workshop ended with a talk by Eric Jensen (Brandeis University, U14/U7) on the results of the painfully low signal Auger-photoemission coincidence spectroscopy studies of solid surfaces. The workshop concluded with an open discussion on the future of the UV Ring, led by Neal Shinn, Gwyn Williams, and Kevin Smith. Overall, this workshop was very successful, was well attended, and was a measure of the vitality of the surface science community at the NSLS.
WORKSHOP ON IMAGING

Harald Ade
SUNY at Stony Brook
Chair

Shawn Williams
SUNY at Stony Brook
Co-Chair

The increasing size, diversity and success of the X-ray imaging community at the NSLS revealed the necessity of bringing together experts from many fields with the common thread of imaging, to discuss the hardware and software techniques used to get the most information out of their samples. One measure of the success enjoyed by members of this community might be represented by the two R&D 100 awards won by imaging devices at the NSLS during the last few years. In addition to hardware and its applications, one of the workshop’s goals was to help facilitate the transition towards more sophisticated and easier image analysis and presentation.

David Sayre opened the workshop, to provide a conceptually cohesive framework, with a general talk delineating principles and concepts of imaging. Subsequent talks were arranged in groups according to the primary concept and conceptual complexity of the various techniques. Microprobe devices, such as scanning photoemission (H. Ade, SUNY at Stony Brook) and transmission microscopes (S. Williams, SUNY at Stony Brook), X-ray fluorescence microprobes (K. Jones, BNL), as well as electron beam microscopes (M. Foster, BNL) were presented in talks before the lunch break. While electron microscopes obviously do not use the NSLS, its presentation was included in the workshop to provide insight into image processing and display techniques used by the electron microscopy community, with the intent to stimulate similar use of these techniques by the x-ray imaging community. This included, for example, a special way to color code concentration ratios of several components in a sample, which can then be presented in a single 2-D image.

During the lunch break, commercial vendors displayed hardcopy devices, and a small poster session enabled participants to display work not covered by talks. Projection imaging and related techniques, such as angiography (D. Chapman, NSLS), topography (M. Dudley, SUNY at Stony Brook) and microtomography (J. Dunsmuir, EXXON) were presented in the first afternoon session. J. Dunsmuir in particular had nice examples of data and image presentations, which included an impressive video tape with a "flight-simulator" representation of 3-D tomographs. The final session belonged to reciprocal space sampling techniques. I. McNulty (SUNY at Stony Brook) presented the first submicron images achieved with Fourier Transform Holography. R. Sweet concluded the talks by comparing concepts in crystallography and imaging, thus complementing D. Sayre’s opening remarks.

Since the organizers and selectively polled colleagues perceived a lack of sufficiently fast, hardcopy devices/facilities at BNL, the workshop concluded with a roundtable discussion of this topic. This discussion resulted in a formal letter of interest by the NSLS imaging community suggesting that BNL procure a high-end hardcopy device. It was favorably received and with the input of all potential users, investigations for the most suitable device are under way.

A 1991 R&D 100 Award was given for the Scanning Photoemission Microscope (SPEM) developed by researchers from SUNY at Stony Brook and BNL. The collaborators were: Erik Johnson, NSLS (rear); Dieter Kern, IBM (left); Erik Anderson, LBL; Janos Kirz, SUNY at Stony Brook; Steven Hulbert, NSLS; and Harald Ade, SUNY at Stony Brook.
UV FEL MACHINE REVIEWS

Ilan Ben-Zvi
NSLS

Sam Krinsky
NSLS
Deputy Chairman

The National Synchrotron Light Source is preparing a proposal for the construction of a linac based Free-Electron Laser (FEL) (see “Proposed UV-FEL User Facility at the NSLS” in Section IV). An advisory panel has been asked to review the FEL design and guide the NSLS team in the associated scientific and technological issues. The panel members are:

J. J. Bisognano - CEBAF
J. S. Goldstein - LANL
C. W. Leemann - CEBAF
J. M. J. Madey - Duke
R. Miller - SLAC
E. T. Scharlemann - LLL
H. A. Schwettman - Stanford
A. M. Sessler - LBL (Chair)
R. Sheffield - LANL
J. Wurtele - MIT

The advisory panel held two meetings in 1991. The first meeting took place on April 11–12. Presentations by the BNL staff outlined the design of a UV FEL to provide a high peak power, tunable, coherent source from the visible down to 100 nm, with the possibility for operations at 50 nm and below, with further development. “In order to provide multiple radiation sources, switching of the electron beam to different wiggles at kilohertz rates is proposed based upon transverse separation due to energy modulation and dispersion. Moreover, the output of a single FEL is divided between several users using a rotating mirror.”


The panel examined the key design choices of the machine and has confirmed the NSLS team’s choices. In their report they said, “The choice of a superconducting linac as the driver of the source offers unique opportunities in terms of electron beam quality, and flexibility of the pulse time structure and repetition rate...we strongly support the choice of a superconducting RF linac as the driver for a user facility UV FEL source.” The panel also supported the choice of operating temperature and frequency of the linac. Other issues that were examined were the choice of an FEL amplifier over an FEL oscillator and the proposed use of frequency multiplication in the FEL.

In general, the panel felt that “...the design has innovative features that should be pursued to clarify their full potential.” The advisory panel made a list of recommendations covering issues of the superconducting linac, beam optics, emittance preservation, FEL physics, wiggler design and more. It was stated that “...there are many items which require further work prior to a Proposal. The Panel is interested in following the development of these ideas and has agreed to meet again at the NSLS on October 21–22, 1991.”

In the October meeting the program started again with presentations by the NSLS staff; welcome and charge: S. Krinsky, project overview: I. Ben-Zvi, FEL design: L. H. Yu, Scientific case: E. Johnson, electron gun: K. Batchelor, electron transport: S. Kramer and X. Zhang, linac: H. Halama, lasers: A. Fisher, diagnostics: J. Rogers, wiggler: G. Ingold, wiggler errors: A. Friedman. The reaction of the panel was again positive: “The panel was very favorably impressed with the progress that has been made since our meeting in April. In particular we are pleased to note that significant progress has been made on the scientific case for the proposed facility.”

The panel made a series of comments on the draft CDR, recommending a cost estimate to be developed, further design of the recirculating arcs, reduction of the sensitivity of the FEL to parameters, increased injection energy, capability of higher average linac beam current, sizing magnets for higher gradients in the linac, magnetic beam bunching, study of beam optics of the superconducting cavities, wake field policy, study of the beam matching to both undulators, treatment facility for cavities, beam stability, beam diagnostics, control systems and a commissioning plan.
The panel focused its deliberations upon pre-construction R&D. After much deliberation the panel was of the opinion that there are approximately eight items that should be addressed before embarking on the construction project. Most of these items are being addressed in the R&D program of the Accelerator Test Facility (ATF) (see “Accelerator Test Facility” in Section IV). The pre-construction R&D items are:

1. High duty factor RF gun.
2. Matching of gun into ATF linac and subsequent acceleration.
3. Superconducting cavities control.
4. Visible FEL experiment on the ATF.
5. Harmonic generation experiment on the ATF.
6. Construction and measurement of prototype undulator and diagnostics.
7. Seed laser demonstration.
The Department of Energy High School Honors Research Program, now in its fifth year at Brookhaven National Laboratory, was host to 60 high school students this year. The two week program, held between July 24 and August 6, was designed to provide a top high school student from each state access to several of the premier research machines at the Laboratory, as well as to expose them to the day-to-day “business” of scientific research. In addition to the 50 states, a student from the District of Columbia, Puerto Rico, the Department of Defense Dependent Schools, and seven foreign countries (Canada, England, France, Italy, Japan, Mexico, and Germany) attended this year’s program.

The first two full days of the program were devoted to familiarizing students with the NSLS and the BNL environment. They heard lectures on safety (T. Dickinson), properties of synchrotron radiation (S. Hulbert), applications of synchrotron radiation (R. Klaffky), and relativity (J. Rogers). Sessions were also held to discuss physics in general and the specific experiments the students would be doing at the NSLS and the Department of Applied Science.

Sessions were also held to meet and work with their team members, as well as to apply their new knowledge. The students worked in ten groups of six students each, to calculate the theoretical photon flux output of the NSLS at two different photon energies. In addition, the students toured both the NSLS and the Laboratory site during the initial two days. After a day’s visit to New York City, the next three days were devoted to running their experiments. Thanks to the combined efforts of the scientific staff and the Office of Educational Programs, there was a total of 6 experiments available for the students to do. A total of 26 three-hour shifts at the NSLS, and 4 three-hour shifts at the Department of Applied Science enabled each of the 10 teams of students to perform 3 different experiments. The six 1991 experiments are described below.

**Experiment XXX: Optical Diffraction (D. Chapman/N. Lazarz).** This experiment used a He-Ne laser and simple objects to introduce principles of diffraction from periodic structures and computer controlled data acquisition. The students measured the wavelength of the He-Ne laser light with a machinist’s ruler and using this data, determined the spacing of a Ronchi ruling.

**Experiment U4-IR: Fourier Transform Infrared Spectroscopy (G. Williams).** The students studied the operation of the FTIR on U4-IR and obtained spectra of glass, black polyethylene film, potassium bromide, potassium iodide and barium fluoride. The students then determined the “spring constant” for the ionic compounds and observed how well the measured data fit a simplified theory of atomic vibration.

**Experiment U-14: Monochromator Efficiency and the Photoelectric Effect (C.-C. Kao).** The students determined the monochromator transmission efficiency between 100 and 300 eV. This was done by measuring the photoelectric current generated by illuminating a metal with a known photoelectron yield with light from the NSLS. The calculated flux from this measurement (monochromator output) was compared to the theoretic photon flux (monochromator input).

**Experiment X-26: Trace Element Analysis by X-Ray Fluorescence (K. Jones).** Students bombarded a known sample with “white” x-rays and observed the fluorescence peaks corresponding to the different trace elements in the sample. They then applied their new skills by trying to identify the “perpetrator” at a “crime scene” by performing a trace element analysis on a hair sample from the “crime scene.” They compared this data to the elemental analysis of each team member's hair to identify the unknown “perpetrator.”

**Experiment X-19: Oxygen Absorption in Hemoglobin (L. Furenlid).** Students determined the change in the iron K-electron binding energy as a function of oxygen loading and used this relationship to predict the K-shift for partial oxygen loadings.
FP-ST: Shock Tube Studies of Reaction Kinetics (B. Klemm/J. Sutherland). Students used the Flash Photolysis - Shock Tube apparatus in the Department of Applied Science to determine the rate constant for the reaction between 1-butene and atomic oxygen at a pressure of about 600 torr and a temperature of approximately 1200 K. The operation and capabilities of the apparatus were also thoroughly investigated.

During the three days that the experiments were being done, the students who were not at the NSLS or the Department of Applied Science taking data, wrote the Experiment Reports for the completed experiments. Ten members of the scientific staff (mentors), then reviewed these first drafts, which were returned to the teams with comments. The mentors were: J. Smalley, DAS; B. Johnson, DAS; M. Bhat, DNE; A. Davenport, DAS; A. Sedlacek, Chemistry; M. Rivers, DAS; S. Feldberg, DAS; R. Beuhler, Chemistry; C. Thorn, Physics; R. Thomas, OEP. After their first drafts were completed, the students toured five of the BNL departments (Chemistry, Applied Science, Medical Physics, and the HFBR), to expose them to the comprehensive research programs undertaken at the laboratory. In addition, during the week, the students heard two special lectures (DNA Damage and Repair Mechanisms – R. Setlow and Global Climate Change – B. Manowitz).

During the entire time that the students were at BNL, there were six program advisors available 24 hours a day to assist the students in all aspects of the scientific program as well as to provide supervision. The six program advisors were: J. Callaway, E. Islip H. S.; L. Celenza, Central Islip H. S.; L. Haman, Dayton Avenue School; C. Hudson, Central Islip H. S.; N. Coggins Lynch, E. Islip H. S.; and W. Lynch, E. Islip H. S.)

After a weekend spent visiting Port Jefferson or a water amusement park and then the beach at Watch Hill, the students formally presented their papers to their peers, their mentors, the program advisors and the scientific staff. On their last evening at BNL, a formal awards ceremony was held in Berkner Hall, with each student receiving a laser-engraved plaque commemorating their participation in the program. Information regarding the program and copies of the student reports or a videotape of their formal presentations can be obtained by contacting Karl Swyler or Don Metz in the Office of Educational Programs (516) 282-7171.
The average VUV ring performance statistics for FY 1991 are given below:

Average fill current
- 837 mA.
Average charge rate
- 38 mA/min.
Average lifetime at 500 mA
- 200 min.
Total user integrated current
- 2480 A-hours (103.3 A-days)

Although the charge rate and fill current are down from last year's, the total integrated current delivered to the users increased slightly. With the major effort being expended on improving the injection system, the charge rate increased to 70 mA/min. at year's end with a slope that should exceed 90 mA/min. in the future.

Despite these problems, the integrated current exceeded last year's record value. This was accomplished with a 10% reduction in beam time available to the users. A major reason for this drop in beam time was the unfortunate vacuum accident which vented the ring to 6 torr in October 1990. The subsequent repair, conditioning, and studies required to return the ring to normal operating conditions took away from the available beam time. The vacuum history showed recovery continuing through April 1991. The increased demand from the users for special low current and bunch number operation also contributed to the reduced integrated current. This makes the integrated current, as a figure of merit, not completely representative of the productivity for an active research machine.

The major improvement on the machine has been the commissioning of the 4th harmonic rf cavity. Although this cavity is presently operated without a power source, the bunch lengthening achieved with this cavity has resulted in a factor of two increases in beam lifetime at the higher currents.

Since the bunch lengthening is current dependent, the effect on the integrated current over a fill period is about 30%. Powering this cavity to achieve optimum lengthening at all currents will be pursued in the future, when the resources are available to handle the phase locking of this system to the beam signal.

During this next fiscal year the major improvement will be the commissioning of new global feedback systems that will provide stabilization of the beam centroid in both the horizontal and vertical planes. Like the existing vertical global feedback system, the new system will damp beam oscillations in both vertical and horizontal planes, up to a frequency of 60 Hz, by factors of two to five. The new system will also provide higher dynamic range from the new rf receivers which will also be installed. In addition, with the doubling of the number of beam input channels, the new system will also damp beam oscillations that have correlations of their spatial sources that are different than the betatron tune number.

The global feedback systems will be able to handle the dynamic changes in the closed orbit. However, it doesn't address the reproducibility of the orbit from cold (zero current) fills or after shutdowns. Considerable effort will be made to track and eliminate the sources of these errors. The improved modeling of the ring which will result from these studies will also make it possible to consider user access to tuning the undulator gaps or photon energy. The new control system will provide
the ability for user accessible macros that will not only vary the gap but also provide the steering and focusing adjustments of the electron beam that will make these changes transparent to the other users.

Studies are also planned on better understanding and improvement of the injection process. If significant improvement can be made on injection efficiency and beam stability during the injection, then it may be possible to start to consider a possible top-off mode of injection. In addition to the obvious gain in integrated current, the top-off mode could stabilize the power level and density on mirrors and optical elements, in theory contributing to improved performance.

Once these efforts of improved performance of orbit stability, orbit reproducibility, and injection are achieved, there will be a need for user input to set priorities for future upgrades. Some of the potential improvements are:

1) - higher energy operation up to 800 MeV
2) - higher brightness beams
3) - greater flexibility in undulator tuning

However, the greatest potential may rest with the users, in proposing innovative photon beams and beamlines which will continue to keep the VUV Ring doing leading edge research well into the next century.

Fig. 1 VUV Ring time usage based on total time (not scheduled time) for fiscal year 1991.

Fig. 2 The VUV Ring integrated beam current available to users per month for fiscal year 1991.
Fig. 3 The injection current averaged over all fills of the VUV Ring during each month of 1991.

Fig. 4 The VUV Ring injection charge rate averaged over all fills during each month of FY 1991.
Fig. 5 The VUV Ring vacuum pressure at 500 mA beam current averaged over each month during FY 1991.

Fig. 6 The VUV Ring exponential beam lifetime at 500 mA beam current averaged over each month during FY 1991. The increased lifetime due to the harmonic cavity is evident beginning in December 1990.
Over the course of FY 1991 there were 247 different experiments performed, representing a 27% increase over FY 1990. The institutional breakdown of scientists participating on these experiments was 42% from universities, 21% from industry, 23% from BNL, 6% from other DOE labs, 4% from government labs, and 4% from abroad.

Major progress occurred in the U13UA high resolution/high flux soft x-ray spectroscopy branch line with the performance of the first gas phase experiments. On the U5U spin-resolved photoemission beamline a new TGM/SGM monochromator was aligned and an MBE facility was connected to the experimental chamber. In addition, there were numerous beamline upgrades, as discussed below.

On the U2 lithography beamline, beam uniformity was improved by replacing a collimating mirror with a flat mirror and by installing a more uniform beryllium window. A new M₀ mirror with improved figure was installed on the U3A ERG beamline to achieve a better focus, and a more accurately calibrated grating was installed. A preparation chamber was installed on top of the U4A experimental chamber to remove the need for a sample transfer mechanism. Also, a program was undertaken to improve the analyzer resolution by upgrading the detector. On the U4IR line a midrange IR (0.5-8μm) interferometer and microscope were commissioned; pump-probe experimental capability was added using Nd-YAG light transported from U10 through a fiber optical cable. An additional beryllium and polyimide window were added to U6 to allow lithographic exposures at atmospheric pressure. A variable line-spaced high flux grating monochromator operating from 50-220 eV with a resolving power of 500 was installed on U10A. The toroidal focusing and scanning mirror was repolished and recoated with nickel. On U10B a rotatable experimental chamber was installed for photodesorption studies at varying incident angle. On U11 design work for a second order sorting and beam steering chamber was completed and a new monochromator controller installed. A time-of-flight gas phase cluster experimental chamber was put into operation on U12B and alignment of U12A continued. On U14B simultaneous logging of vertical beam position, temperature of aluminum water system (which cools the storage ring chamber), and X and Y displacements of PUE 20 was implemented. The U15 beamline added the capability of detecting near IR to near UV luminescence from a large solid angle surrounding the emission region of decay products from core excited free molecules. A sample cryostat for fluorescence EXAFS between room temperature and 40K was also put into operation. The optical components on U16B were plasma cleaned using water vapor.

There were a number of changes on the UV floor. A new 40 ton air conditioning unit was installed on the roof, a stainless steel toxic gas exhaust system was installed for U11, U14, and U15, and the pump exhaust system was rerouted to remove it from beamline cable trays. A platform was built on the crane support beam to provide access for repairing components near the roof. An upgrade of beamline electrical distribution systems, an NSLS funded project, began. A new emergency generator was installed and wired into electrical distribution panels. Another major project was the installation of a new VUV roof.
## VUV Storage Ring Parameters as of January 1992

<table>
<thead>
<tr>
<th>Parameters</th>
<th>VUV Storage Ring</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal Operating Energy</td>
<td>0.745 GeV</td>
</tr>
<tr>
<td>Maximum Operating Current (multibunch operation)</td>
<td>1.0 amp ((1.1 \times 10^{12} \text{ e}^{-}))</td>
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<tr>
<td>Circumference</td>
<td>51.0 meters</td>
</tr>
<tr>
<td>Number of Beam Ports on Dipoles</td>
<td>17</td>
</tr>
<tr>
<td>Number of Insertion Devices</td>
<td>2</td>
</tr>
<tr>
<td>Maximum Length of Insertion Devices</td>
<td>(-2.5 \text{ meters})</td>
</tr>
<tr>
<td>(\lambda_c(E_c))</td>
<td>25.3 A ((486 \text{ eV}))</td>
</tr>
<tr>
<td>(B(p))</td>
<td>1.28 Tesla ((1.91 \text{ meters}))</td>
</tr>
<tr>
<td>Electron Orbital Period</td>
<td>170.2 nanoseconds</td>
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<tr>
<td>Damping Times</td>
<td>(\tau_x \approx \tau_y \approx 17 \text{ msec}; \tau_e \approx 9 \text{ msec})</td>
</tr>
<tr>
<td>Touschek lifetime dependent on current per bunch and vertical emittance</td>
<td>200 min @ 200 mA</td>
</tr>
<tr>
<td>Lattice Structure (Chasman-Green)</td>
<td>Separated Function, Quad, Doublets</td>
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</tr>
<tr>
<td>Magnet Complement</td>
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</tr>
<tr>
<td></td>
<td>24 Quadrupole ((0.3 \text{ meters each}))</td>
</tr>
<tr>
<td></td>
<td>12 Sextupole ((0.2 \text{ meters each}))</td>
</tr>
<tr>
<td>Nominal Tunes (v_x, v_y)</td>
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</tr>
<tr>
<td>Momentum Compaction</td>
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<td>R.F. Frequency</td>
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<td>Radiated Power</td>
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<tr>
<td>R.F. Peak Voltage (typical)</td>
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<td>Design R.F. Power</td>
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<tr>
<td>(v_S) (Synchrotron Tune)</td>
<td>0.002</td>
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<tr>
<td>Natural Energy Spread ((\sigma_x/E))**</td>
<td>(4.5 \times 10^{-4}) ((I &lt; 20 \text{ mA}))</td>
</tr>
<tr>
<td>Natural Bunch Length ((2\sigma))**</td>
<td>7.6 cm ((I &lt; 20 \text{ mA}))</td>
</tr>
<tr>
<td>Number of RF Buckets</td>
<td>9</td>
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<tr>
<td>Typical Bunch Mode</td>
<td>7</td>
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<tr>
<td>Horizontal Damped Emittance ((\varepsilon_x))</td>
<td>(1.5 \times 10^{-7} \text{ meter-radian})</td>
</tr>
<tr>
<td>Vertical Damped Emittance ((\varepsilon_y))</td>
<td>(\geq 2.8 \times 10^{-10} \text{ meter-radian (adjust.)})</td>
</tr>
<tr>
<td>Power per Horizontal milliradian, 1A</td>
<td>2.3 Watts</td>
</tr>
<tr>
<td>Source Size: (\sigma_x, \sigma_y)</td>
<td>0.5 mm, &gt; 0.06 mm</td>
</tr>
</tbody>
</table>

* Lifetime variable up to \(-350 \text{ minutes depending on use of bunch lengthening 211 MHz RF cavity.}*

**Current and RF voltage dependent.

**Source of Data:** NSLS Parameters, January 1983, compiled by A. van Steenbergen; updated values provided by Anne-Marie Fauchet and Norman Fewell (NSLS).
Commissioning of the high power insertion devices has continued during this period. Full power operation of X17 and X25 is now routine. The active interlock system for X21 is now complete, and this beamline is expected to come online shortly. Local feedback systems for X1, X5, X17, and X25 have been developed and are fully operational.

Beam stability has been greatly improved by the installation of a real time harmonic global feedback system. This system, which operates in both planes, is similar to that developed for the VUV Ring, for which the designers won a R&D 100 award. The installation of this feedback system, which has to function without adversely effecting the local feedback systems, is a remarkable achievement by the Diagnostics Group.

Installation of the RF receivers used by the feedback systems continues. At the present time, 26 units out of a full complement of 48 have been installed and are operational. When completed this system will allow real time orbit measurements. The Computer Group has developed a system that displays the X-ray orbit, using the present complement of receivers, at a display rate of approximately 5 orbits/sec.

An investigation into methods to reduce beam motion caused by operation of the HFBR helium compressor was carried out by a private consulting company and BNL staff. It was found that the magnitude of the motion was dependent on the water saturation levels of the ground between the HFBR and the NSLS. It was also found that the compressor frequency (approximately 12 Hz) coincided with the vibrational resonant frequency of the quadrupole/support girder combinations. Some magnet movement was reduced by tightening support bolts and regrouting some floor attachments. Further reduction was done by moving the resonant frequency of the quad/girder combinations. This was achieved by supporting the large amount of lead shielding on newly constructed supports, instead of on the quadrupole support girders. These changes reduced the beam movement sufficiently, such that the compressor could run without noticeable movement by the X-ray users, and since the vibration frequency lies within the range of the newly installed global feedback system, the future operation of the HFBR compressor should not be a problem.

Some of the problems encountered during the year include the vacuum failure of a RF cavity power coupling window, and the failure of a high voltage transformer. This transformer weighs approximately 2,000 lbs. and is located inside the RF penthouse on top of the roof, which posed quite a logistics problem. In addition to downtime due to normal equipment failures, this year we were plagued by a series of computer problems that led to approximately 20% of our unscheduled downtime. A concerted effort by the Computer Group finally alleviated the problem and the computer system is now working adequately and work on upgrading the system is progressing. The installation of a large air conditioning unit and ductwork in the power supply area has made a marked improvement in the temperature of that area.
Previously, inadequate cooling had led to extremely high ambient and equipment operating temperatures which could lead to equipment malfunction or failure.

On the whole this was a good running year for the X-ray Ring, and with most of our commissioning bugs behind us, we look forward to improved performance in the coming year.

![Pie chart showing X-ray Ring time usage for FY91]

Fig. 1 X-ray Ring time usage based on total time (not scheduled time) for fiscal year 1991.

![Bar chart showing X-ray Ring amp hours by month for FY91]

Fig. 2 The X-ray Ring integrated beam current available to users per month for fiscal year 1991.
Fig. 3 The injection current averaged over all fills of the X-ray Ring during each month of 1991.

Fig. 4 The X-ray Ring exponential beam lifetime at 150 mA beam current averaged over each month during fiscal year 1991.
X-RAY BEAMLINE OPERATIONS

Roger Klaffky
NSLS
X-ray Operations Manager

During FY 1991 routine operations began on four more X-ray beamlines, bringing the total number of operational beamlines to 55. These included X1B, X6B, X13B, and X17B2 (Angiography). Construction and/or commissioning took place on X4A, X6A, X21, X28A, X28B and X28C. A count of NSLS Safety Approval Forms indicate that there were 872 different experiments performed, of duration several days to several months, representing a 14% increase over FY 1990. The breakdown of scientists participating on these experiments was 46% from universities, 21% from industry, 16% from BNL, 7% from other DOE laboratories, 6% from government labs, and 4% from abroad.

There were a number of improvements in beamline capabilities this year. Installation of a water-cooled safety shutter in the X1 front end allows the X1 local orbit feedback system to remain on when the safety shutter is closed. The X1A1 soft X-ray microscope line has obtained 50-75μm resolution luminescence images from fluorescence dyes such as are used to tag sites of specific biochemical activity in fluorescence microscopy. The X1A2 SPEM achieved better than 0.2μm resolution ESCA for which it received an R&D 100 award. In its early operational phase, the X1B soft X-ray spectroscopy beamline demonstrated that it has unmatched intensity and resolution. A set of large d-spacing multilayer crystals were purchased for the X2B tomography monochromator to increase intensity. Low temperature (12°K) capability was added on the X3B1 EXAFS beamline. Various additions were made to the X14A diffraction line: a beam position monitor, an aperture with vertical motion for characterizing the monochromator sagittal focussing, and a diffractometer table with tilt and rotation capabilities. Utilization of storage phosphor plates was also implemented. The X5 LEGS beamline completed its first full year of operation. A higher power, shorter wavelength (300μm) laser was installed to increase the γ-ray energy from 300 to 340 MeV. Liquid helium III and helium IV targets were put into operation as well as two new detectors – an array of 48 scintillator bars for neutrons and a large sodium iodide detector for gammas and high energy charged particles (pions, protons). On the X7A energy dispersive diffraction beamline, a water-cooled Ge(111) channel-cut monochromator was installed which doubles the intensity below the Ge K edge (11.1 KeV). An NSLS-designed high-stability horizontal focussing device with an assymetric-cut Si(220) crystal was installed for experiments using a high pressure chamber or for experiments using less than 200μm of horizontal beam. The focussing has led to an order of magnitude increase in flux on small samples. On the X7B crystallography beamline there were a number of additions: a linear 2 cm CCD detector; software for surface diffraction measurements, vertical scan capability for the sample table, and capability to scan the second monochromator crystal χ angle. A new CCD camera was installed on X8C for protein crystallography experiments and the X8A beamline was fully characterized. A sagittally-focussing Si(111) monochromator was put into operation on X9A and a beryllium window installed at the end of X9B to replace a 0.003” aluminum window.
arrangement which was prone to developing leaks after exposure to white beam. A mirror feedback system was implemented on X10C to improve beam stability which is vital for measurements on submillimeter crystals using a small focussed beam. An EXAFS microprobe consisting of a mirror/pinhole arrangement and a raster scanned sample was used with (25μm) × (25μm) resolution elements. Sagittal focussing was introduced on the X11A beamline, leading to a two-fold intensity increase at 11 keV and a four-fold increase at 6 keV.

The entire data collection system on X12B was revamped to improve reliability and a 2D detector put into routine operation. On X14A a low temperature Displex unit was installed with a hemispherical beryllium dome for sampling full reciprocal space volume. The X15A monochromator housing was upgraded to allow operation with a helium atmosphere. A customized fluorescence detector was installed on X15B for EXAFS measurements.

On the X16A five circle surface X-ray diffractometer, a faster, smoother gearless servomotor was installed to increase the angular range to 45°, and a position sensitive detector was put into operation. Work continued on the conversion of X16C to a monochromatic beamline. The 17B1 (Materials Science) and X17B2 (Medical Research) lines ran in a full-time operational mode. The X17 superconducting wiggler cryogenic system was upgraded with the purchase of a new cryogenic compressor and helium transfer lines, rebuilding of the helium liq-

uefier and the old compressor for use as a spare, and an upgrade to automatic valve controllers. (X17C)

On X18A the data collection system was converted to a PC-based GPIB interface system, with software control of motors, counter, multi-channel analyzer PC board and temperature controller. A low temperature UHV surface chamber mounted on the X18A Huber diffractometer successfully operated in the spring. The X19A external monochromator drive system was installed, leading to marked improvement in performance and reliability. A window valve system was designed which will provide for selection of a standard beryllium window or a high throughput diamond window for lower energies. Design work was carried out on the white beamline X19C for the addition of a bent cylindrical mirror, a monochromator and a liquid surface spectrometer. On the X20 beamline turbopumps were replaced by ion pumps to reduce noise, the computer systems were upgraded, and the X-ray beam vertical offset in the X20C monochromator was decreased to 1.5 inches to reach higher energies. Beaml ine safety interlocks were completed on X21 and a monochromator vacuum tank installed in preparation for tests of the monochromator cooling scheme. On X22B a new liquid metal cell was installed and steps were taken to improve the beamline capabilities so as to change or scan energy to 10 keV in the horizontal diffraction mode. A new 3.5°C Displex refrigerator was installed on X22C. On X23A2 the Si(220) monochromator crystal was replaced with a Si(111) crystal to improve resolution and harmonic rejection. Also, an active feedback table was installed to isolate the monochromator from vibration sources. The computer system was upgraded to a full online EXAFS system similar to that on X19A. A vertical beam position monitor was installed on X24A, and the monochromator differential pumping and crystal mount were improved for rapid crystal changes. Sagittal focussing by the second Ge(220) monochromator crystal was implemented, reducing the amount of focussing required from the downstream toroidal mirror thereby extending the energy range. The beamline optics on the X25 wiggler were fully commissioned and a full experimental program commenced. On X26A a monochromator designed for two channel-cut Si(111) crystals was put into operation using a single Si(111) crystal for energy scans between four and fifteen KeV. Construction of the X28C diagnostic line for X-ray orbit position monitors was completed prior to taking first beam.

There were a number of changes on the X-ray floor. A mezzanine and elevated walkway were constructed at X5 to maintain an access route into the X-ray Ring while allowing the construction of the X4C and X6A hutch. A darkroom was constructed next to X19C. The area vacated by the NSLS Vacuum Group close to X24 was converted to a set-up space for staging of experiments on the X-ray and UV Rings. A new vent line was added outside the building for purging the LN$_2$ transfer line prior to filling dewars.
To improve communications between the NSLS and users an additional channel (CH.2) was added to the TV monitors for expanded messages on machine problems, a user pager system was put into operation to announce unexpected beam dumps and to indicate the return of beam after such dumps, and electronic mailing of the weekly user meeting minutes began. A Safety Approval Form data base was also implemented in the control room.

### X-Ray Storage Ring Parameters as of January 1992

<table>
<thead>
<tr>
<th>Parameters</th>
<th>X-Ray Storage Ring</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal Operating Energy</td>
<td>0.75 - 2.5 GeV</td>
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<td>Maximum Operating Current</td>
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<td>Circumference</td>
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<td>Number of Beam Ports of Dipoles</td>
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<td>Number of Insertion Devices</td>
<td>5</td>
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<tr>
<td>Maximum Length of Insertion Devices</td>
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<td>( \lambda_c(E_c) ) at 1.22 T (B)</td>
<td>2.48 A (5 keV)</td>
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<tr>
<td>( \lambda_c(E_c) ) at 5.0 T (W)</td>
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<td>( B_r(\rho) )</td>
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<td></td>
<td>16 Quadrupole (0.80 meters each)</td>
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<td>32 Sextupole (0.20 meters each)</td>
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<td>Natural Bunch Length ( (2\sigma) )</td>
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<tr>
<td>Number of RF Buckets</td>
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<td>Typical Bunch Mode</td>
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<td>Horizontal Damped Emittance ( (\varepsilon_x) )</td>
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<td>Vertical Damped Emittance ( (\varepsilon_y) )</td>
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<td>Power per Horizontal milliradian, 0.25A</td>
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<td>Typical Arc Source Size: ( \sigma_h, \sigma_v )</td>
<td>(-0.35 ) mm, (-0.15 ) mm</td>
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</table>

**Source of Data:** NSLS Parameters, January 1983, compiled by A. van Steenbergen; updated values provided by Anne-Marie Fauchet and Norman Fewell (NSLS).
The NSLS has, for a number of years, had an aggressive policy towards providing the Users with as good an environment for doing research as possible. Of course, fiscal, personnel, and space constraints have forced some limitations and compromises. However, this past fiscal year has seen some major upgrades in the quality of life come to fruition. Perhaps the most obvious to any visitor to the NSLS is the User Administration Office (UA) which has been moved to a very convenient, central location. The location, coupled with an expansion of the space available to the Users for registration, has created a central information and reception area of which we are very proud. It is not just cosmetics which greet the User. Recently, the User database has been implemented in the UA Office which, for the first time, allows Users to register by direct input into computer terminals. The duplication of entries is a thing of the past, with all necessary forms now being printed from the computer. The database registration allows the NSLS to meet all the new DOE demands on foreign national registration and facility utilization statistics in an efficient manner.

In order to move the UA Office to its proper location, another major improvement in life style at the NSLS took place. The old library has been moved to a new location and converted into the beautiful Chasman-Green Memorial Library/User Lounge. It is located where the NSLS stock room used to be. The stockroom has been moved to a nearby, convenient location and continues to provide the excellent 24 hour/day service for which it has gained a lot of praise. The library/lounge is an excellent place to use to get away from the hustle and bustle of the experimental floor, to have a small meeting or discussion of new results, or to explore the most recent literature.

This year has seen a very welcome increase in the scientific staff dedicated to User support on NSLS beamlines. Dr. Lars Furenlid has taken charge of the X19A spectroscopy beamline and has worked closely with the existing PRT to upgrade the quality and quantity of beamtime available to users. Recently, the NSLS was pleased to bring on board Dr. Mei-Ling Shek to run the U14A General User photoemission spectroscopy line. She brings years of experience in the field to the program of upgrades and expansion of the U14A facility. The NSLS now has at least one scientist associated with each NSLS operated beamline.

Safety is now a part of the culture of the NSLS and the highly successful upgrade in the quality of the NSLS User Shop is a prime example. Mr. Gerry Van Derlaske has joined the Experimental Program Support Section as User Shop Manager and is available during all normal working hours to assist anyone in need of the shop, to answer questions, to keep our full complement of tools in proper working condition, and to help train those users needing shop experience. A written document of basic rules and safety orientation has been prepared and is being used as the minimum basis for qualification for access to the shop. Mr. Van Derlaske is in charge of the training and the documentation of qualified users. The safe, productive environment now
found in Mr. Van Derlaske's Shop has resulted in improved safety and many positive comments from Users. The NSLS continues to put significant resources into the shop, knowing that quick turnaround of those many necessary "widgets" at any hour of the day or night is sometimes critical to success of an experiment. A new set of Shop User Rules has been established and already has resulted in a safer, cleaner Shop.

Gerry Van Derlaske, User Shop Manager

Effective interaction of the burgeoning NSLS User population with the NSLS Safety Personnel is critical to continuing our very high level of safety. In order to bring about closer communication between the Users and Safety Personnel most of the NSLS Safety Staff have now been concentrated in one central area adjacent to the experimental floor. Andrew Ackerman and Clem Auguste of the NSLS have joined John Alois, Chris Weilandics, and Rudy Zantopp from the BNL S&EP Division in offices near the NSLS Floor Manager. Also located in the same area are Randy Church, Supervisor of the Machine Operators and Operations Coordinators, and Steve Kemp, Maintenance Coordinator and Lead Operations Coordinator. Contiguous space has now been made available to the Coordinators so that they can continue to carry out technical projects when not on shift. Thus, Users can now go to one area of the NSLS to attend to any safety related issues or obtain assistance from the Floor Manager. The Floor Manager is now Mr. Richard Rothe who has replaced Mr. Mike Kelly.

This year saw the commissioning of the new communications channel on the NSLS video system. Channel 2 has been a very successful means of getting information to the Users during times of trouble with the machines, scheduling, computers, etc. Since it is independent of the main system control computers, it has proven many times to be the only effective means of communication from the Control Room to the experimenters.

As most Users know by now, the new card reader access is in place. After many months of problems it seems to be working reasonably well. It has been beneficial to the NSLS in access control for contractors and security of the building. The minor problems which still exist should be ironed out very soon.

The new gas cylinder area and liquid nitrogen fill systems have been completed and provide a much safer, weather protected environment. The NSLS has targeted funds for expansion of the existing area for storage of liquid helium and nitrogen dewars, hazardous gases, and additional gas cylinders.

Life is still not all rosy at the NSLS, but initiatives are underway to solve many of the existing problems. Throughout this year, like most, there have been problems with communication between the NSLS and the BNL Plant Engineering Division. This has led to disruptions on the experimental floor. In order to solve
this problem, the NSLS Management has been working with Plant Engineering on the means of notification of pending work and lines of responsibility. We are hopeful that the system we are now trying will lead to far fewer instances of work being started without proper notification of the affected persons.

Space continues to be a serious problem for the NSLS Community, both Users and Staff. As Denis McWhan discussed in his introduction to this report, two new initiatives for expansion of the present building is underway to add very badly needed space to the experimental floors. It is perhaps good to take a look at the space which the NSLS has made, and will make, available to the Users. The accompanying layout of the NSLS Experimental Floors shows the space available for experimental programs as shaded areas. In addition, the User Shop in the basement of Building 535 and the office and lab space in Building 510 should be included. It is obvious that we are making every effort possible to provide working and living space to the User Community.
Section IV
NSLS Projects
Richard Heese  
NSLS  
SXLS Project Head

The Phase I SXLS Ring has been running successfully for the last year, and has yielded a wealth of accelerator physics information on the performance of very compact synchrotrons. The ring's nominal design current of 500 mA at 200 MeV is consistently being exceeded during studies runs. The peak current that has been stored is 1,200 mA, far more than any other compact synchrotron in the world. The machine has proved to be extremely reliable, and the control system, a central database type system using a graphics oriented control interface with the operator, has proven to be very capable and simple to learn and use. The major impediments to totally smooth operations are ion trapping effects in the circulating beam and beam instabilities. Efforts to understand and control these effects are ongoing.

A linear accelerator, which will serve as an injector for the superconducting storage ring, is being manufactured. This machine is a 200 MeV high-current, short-pulse electron accelerator and will be able to fill the superconducting ring in about 15 seconds.

The superconducting dipole is continuing to be the subject of intensive study. It is a warm bore, iron-free magnet. The NSLS engineering staff has designed a novel support structure to allow extraction of synchrotron radiation from nine ports in each dipole while preventing distortion of the coils due to 200 tons of electromagnetic forces, while keeping the coils at cryogenic temperature.

Due to the strong curvature of the magnet coil structure, large values of higher harmonics, in addition to the required quadrupole and sextupole terms, are generated. This seriously reduces the dynamic aperture of the machine. Therefore, in addition to the basic coils, higher order correction coils are incorporated in the magnet along with trim coils for the quadrupole and sextupole components and vertical steering. Particle tracking with sophisticated codes, developed both at NSLS and elsewhere, confirm the effectiveness of these coils.

The detailed engineering design of the superconducting dipole magnet by General Dynamics was finally completed. Some scheduling delays have occurred due to the complexities of the superconducting dipole. The first magnet is expected to be delivered in January 1993, and the second magnet in May 1993.

The Technology Transfer Program continued in 1991: five people from Grumman Aerospace Corporation were on site, working with us as part of this project, along with the team at General Dynamics involved in the design and construction of the superconducting dipole.

The scientific aspects of the project are proceeding very well. The continuing cost escalation and schedule delays of the superconducting dipole, however, are a cause for concern. Other options to manufacturing the magnets with cost containment and reasonable schedules are being examined.
### SXLS Storage Ring Parameters as of January 1992

<table>
<thead>
<tr>
<th>Parameters</th>
<th>SXLS Phase I</th>
<th>SXLS Phase II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Normal Operating Energy</td>
<td>0.2 GeV</td>
<td>0.696 GeV</td>
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<td>Dipole Magnet Type</td>
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<td>Superconducting</td>
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<td>Maximum Operating Current</td>
<td>0.50 amp (8.8×10^10 e^-)</td>
<td>0.5 amp (8.8×10^10 e^-)</td>
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<tr>
<td>(measured)</td>
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<td>(design)</td>
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<tr>
<td>Circumference</td>
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<tr>
<td>Number of Beam Ports on Dipoles</td>
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<td>Number of Insertion Devices</td>
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<td>0</td>
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<td>λ_c(E_c)</td>
<td>424 A (29.3 eV)</td>
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<tr>
<td>B(ρ)</td>
<td>1.1 Tesla (0.6037 meters)</td>
<td>3.87 Tesla (0.6037 meters)</td>
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<td>Field Index, n</td>
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<td>0.1759</td>
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<tr>
<td>Electron Orbital Period</td>
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<td>28.3 nanoseconds</td>
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<tr>
<td>Damping Partition Numbers</td>
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<td>J_x = 0.53, J_y = 1.0, J_ξ = 2.47</td>
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<td>Damping Times</td>
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<td>τ_x = 0.216 msec; τ_y = 1.15 msec; τ_ξ = 0.46 msec</td>
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<td>Number of Superperiods</td>
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<td>SF Sextupole (0.05 meters)</td>
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<td>QF Quadrupole (0.155 meters)</td>
<td>QF Quadrupole (0.155 meters)</td>
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<td>D1 Drift (0.8965 meters)</td>
<td>D1 Drift (0.8965 meters)</td>
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<td></td>
<td>B Dipole (1.896 meters)</td>
<td>Dipole (1.896 meters)</td>
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<td></td>
<td>D1 Drift (0.8965 meters)</td>
<td>D1 Drift (0.8965 meters)</td>
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<td>SF Sextupole (0.05 meters)</td>
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<td>Radiated power per horizontal milliradian</td>
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<td>2.0×10^{-4}</td>
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<td>Natural Bunch Length (2σ) **</td>
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<td>Vertical Emittance (ξ_y)</td>
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<td>Source Size: σ_h, σ_v</td>
<td>&gt;0.34 mm, &gt;0.2 mm</td>
<td>0.75 mm, 0.75 mm</td>
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</table>

**Current and RF voltage dependent.

Source of Data: Jim Murphy (NSLS).
ACCELERATOR TEST FACILITY

Ilan Ben-Zvi
NSLS
ATF Project Head

Free-Electron Lasers (FEL) are considered by many to be the fourth generation synchrotron light sources. The NSLS is carrying out an extensive R&D program on short wavelength FEL, leading towards a proposal for a UV FEL User Facility (see "Proposed UV-FEL User Facility at the NSLS", in this Section). The Accelerator Test Facility (ATF) is a linac-laser complex, capable of producing high brightness electron beams and high power laser pulses synchronized with the electrons. The ATF is the test bed for developing and testing the components needed for a successful UV-FEL. At the same time, the ATF is serving an advanced accelerator R&D program such as laser acceleration of electron beams and the interaction of intense laser pulses with high brightness electron beams.

The ATF is operated as a users' facility for accelerator physicists. The high brightness electron beam of the ATF, produced by a laser-photocathode RF gun, is already available for experiments. Two experiments already share this ultra high brightness 4.5 MeV, 100 ampere beam. One is a Smith-Purcell radiation experiment by the BNL Advanced Accelerator Group in the Physics Department, the other is a Cerenkov FEL experiment by a Dartmouth-Oxford team.

This gun generates its high current - low emittance beam by irradiating a high quantum efficiency metal cathode in the presence of very high intensity RF electric fields. The generation and careful manipulation of such beams are an area of intense activity in accelerator physics, being the key to short wavelength FELs. The performance of the gun in the past year has been outstanding. The ATF team has measured nano-Coulomb beam bunches about 10 picoseconds long (producing a peak current of about 100 amperes) with a normalized rms emittance of $4\pi$ mm mrad.

The other key components of the BNL ATF are a 50 MeV (up to 100 MeV in the future) electron linac, a 10 ps 10 mJ Nd:YAG laser, a 10 GW CO$_2$ laser system producing 100 ps pulses (10 ps soon). The lasers are synchronized to within one picosecond to the electron beam pulses. During 1991, the 50 MeV beamline leading from the linac to the experiment hall was constructed. Fifty MeV beams will be tested in the linac building in spring 1992 and the 50 MeV experimental program will be started in the fall of 1992.

ATF experiments with a special interest for the NSLS short wavelength FEL programs are the RF gun development program, the visible wavelength FEL oscillator experiment, and the high-gain harmonic-generation experiment. The FEL experiments also include the development of novel superconducting undulators providing a high field and very low magnetic field error.

The RF gun program produces the now famous "BNL gun" model, a one and a half cell p mode S band gun, operating at a peak cathode electric field of 100 MV/m. However our program has gone beyond that. An extremely fruitful collaboration has been established with Grumman Aerospace Corporation in which a potentially higher brightness, but most importantly very high duty factor gun has been developed and is now in an advanced production. To carry out this program Grumman has invested a significant amount of their internal R&D funding. The gun will be tested at the ATF.

The visible FEL oscillator will employ a superconducting micro-undulator (68 period, 0.88 cm period length and 0.5 Tesla peak field at a gap of 0.44 cm). Prototypes of this undulator were built (up to 7 periods long) and performed extremely well in superconducting tests. The field errors of the undulator were very low as constructed, making this perhaps the first undulator that will require no trimming after manufacture. The FEL experiment makes use of the short period of the undulator and of the low emittance beam generated by the RF photocathode gun to operate at 500 nm with a 50 MeV electron beam. Emittance scales with energy so that at 250 MeV a wavelength of 100 nm would be practical. This experiment is done in collaboration with MIT and Rocketdyne.

The high-gain, harmonic-generation experiment is another
facet of the technology-transfer collaboration with Grumman, which is providing the undulator for the experiment. This undulator is a derivative of the superconducting micro-undulator of the visible FEL oscillator. It uses the same principle of a continuous yoke but has parabolic pole faces for two axis focussing and four distinct regions: a prebunching undulator, a dispersive section, an exponential growth section and a tapered section. This experiment emulates the FEL physics of the User Facility UV FEL. The principle of high gain harmonic generation will be tested and characterized in these experiments, as well as the superconducting undulator, the undulator diagnostics, error and alignment studies, and tapering control.

Other light source techniques are being pursued at the ATF. A final example is the coherent synchrotron emission experiment, in which the short, high charge ATF pulse will generate coherent and intense mm wave radiation in a bending magnet. This radiation will also be used as a pulse length diagnostic for the ATF.

References


The NSLS is designing an accelerator based UV/VUV radiation source\(^1\,^2\) that will provide picosecond and sub-picosecond pulses of coherent ultraviolet radiation for wavelengths from 300 to 75 nm. Pulse width will be variable from about 7 ps to under 200 ps, with repetition rates as high as 10 kHz, single pulse energies >1mJ and therefore peak pulse power >200 MW and average beam power >10W in a 10\(^{-4}\) bandwidth. The facility will be capable of “pump-probe” experiments utilizing the FEL radiation with 1) synchronized auxiliary lasers, 2) a second, independently tunable FEL beam, or 3) broad spectrum, high-intensity x-rays from the adjacent National Synchrotron Light Source. The UV-FEL will make possible new avenues of inquiry in time resolved studies of diverse fields including chemical, surface and solid state physics, biology, and material science.

The UV-FEL beam is generated by a high duty factor laser photocathode RF gun, accelerated by a recirculating superconducting linear accelerator then separated into two pulse streams feeding two undulators. A drawing of the facility layout is shown below. The radiation is generated by “conventional” tunable Ti:sapphire lasers, multiplied by conventional techniques to the visible or near UV. This radiation is then injected into the bunching section of the undulator, which is resonant at the injected radiation’s frequency. The energy modulated electron beam is bunched magnetically and fed into the next undulator section, now resonant at a VUV harmonic of the seed radiation. The radiation is now amplified, first in an exponential section, then in a tapered undulator section and transferred to the users.

The wavelength is changed by tuning the seed laser (and varying the energy of the electron beam by the energy modulator cavities to maintain the FEL resonance condition). Thus fast tuning of as much as 30% is possible at each undulator. The two undulators will be staggered in wavelength, to provide a tuning range of 60% without a change in the linac’s energy. The energy modulating
cavities come in slightly detuned pairs, so that alternate pulses in each of the undulators are independently tunable. Thus, up to four independent experiments may operate at one time, each with independent control of the wavelength and pulse duration.

To generate short wavelength coherent radiation one must generate, transport, and accelerate an extremely low emittance electron beam without diluting it. The electron beam of the UV-FEL will be generated by a laserphotocathode RF gun of the type that has been developed at the Accelerator Test Facility. To provide the high repetition rate that is needed for the User Facility, a high duty factor RF gun has been designed in collaboration with Grumman Aerospace Corporation. This gun will operate at a repetition rate of 5 kHz (macropulses) and deliver two pico-second pulses of between one and three nanoCoulombs per pulse at a variable multi-nanosecond separation in each macropulse. Thus the total repetition rate will be 10 kHz. Extensive simulations of the gun’s performance lead us to anticipate a normalized rms emittance of about $4\pi$ mm mrad, a value that is similar to what has been measured at the ATF.

At the energies required for a UV-FEL, the best combination of peak current, stability, energy spread and time structure is available from a superconducting linac at 500 MHz. With such a machine it is possible to run either short, sub-picosecond bunches or long, a few tens of picosecond bunches with little degradation of energy spread due to wake fields or fundamental mode curvature. At 500 MHz, it is possible to operate at 4.5K, simplifying the cryogenic system. Superconducting linacs are available commercially from a number of manufacturers. To reduce the cost of the linac a recirculation scheme with about three passes through the linac will be used. This arrangement has additional advantages, such as the possibility of extracting bunches at various energies destined for a number of FELs operating at various wavelengths, as well as the option to do bunch compression at an intermediate energy.

A 750 MHz superconducting cavity modulates the energy of the accelerator beam. This choice of frequency allows us to accelerate one micro-pulse and decelerate the other, as long as their pulse separation is a multiple of 4 ns. These micropulses are then separated by converting the energy difference into a trajectory modification that sends each micropulse into another undulator. Thus the radiation pulses generated in each undulator can be produced with a controlled small delay, from coincidence to many nanoseconds apart. This feature makes it possible to do FEL on FEL pump-probe experiments.

Unlike FEL oscillators requiring cavities, our FEL amplifier approach eliminates the resonator mirrors which are a difficult aspect of oscillator FEL designs. The laser amplifier scheme proposed here will provide high peak power VUV radiation with the mode structure, bandwidth and frequency stability of an input seed laser.

Wavelength tuning will be done through energy modulation rather than through undulator gap change. (Naturally the seed lasers will tune in synchronism.) This final energy modulation will be done by special purpose accelerating cavities placed just before each of the two undulators, thus necessitating no transport element adjustments. This method offers the following advantages:
1. Simpler undulator design;
2. Undulator operation at its highest $K$;
3. Much faster wavelength tuning, limited in principle only by the seed laser tuning speed.

The undulator design will be based on a superconducting undulator that has been developed at the ATF for a visible FEL oscillator experiment. This undulator has a high axial magnetic field, high precision and low cost per unit length.

The electron bunches in each undulator come at a repetition rate of 5 kHz. The energy modulation for wavelength tuning will be done by two cavities in series, one at the exact linac frequency and the other detuned by 2.5 kHz. The beating of the two frequencies will then create a differential energy modulation, every other pulse in the undulator having an independent energy setting. By switching between two seed lasers, one for each 2.5 kHz stream of electrons, we create two independently tunable FELs operating in the same undulator. The UV radiation pulses will be separated by rotating mirrors and sent to user's stations. Therefore the UV-FEL User Facility will serve four concurrent
users, independently tuning their wavelength and pulse length.

The FEL output in 10^{-4} bandwidth is 1 mJ per pulse, resulting in an average power of 5 watts per undulator in two undulators. The availability of radiation with these characteristics would open up new opportunities in photochemistry, biology, material science and physics.

References


Stability of the electron orbit is essential for the utilization of the NSLS low emittance storage rings as high brightness radiation sources. For an individual user, the required orbit stability can be achieved by a local bump feedback system. However, to install local bump feedback systems for every beamline is both costly and impractical. The coupling between different local bumps may introduce instability, and there may not be enough space for the large number of trim coils required. Therefore, our approach to achieving the necessary orbit stability has been the development and installation of global orbit feedback systems based upon harmonic analysis of the orbit movements and the correction magnetic fields. A harmonic feedback system corrects the Fourier components of the orbit nearest to the betatron tune. The Fourier analysis is done by a simple linear analog network. The input voltages are proportional to the orbit displacements at the detectors, and in real time the output voltages are proportional to the Fourier harmonic coefficients. The feedback does not force the displacement to zero at the detectors, but forces the coefficients of a few harmonics nearest the betatron tune to vanish.

The first development of the harmonic feedback system was a prototype system to correct the vertical orbit on the VUV Ring. This system uses four position monitors and four correctors, and has been successfully used in operations since 1989. Based upon the experience with the VUV Ring, feedback systems have been developed and installed during the last year on the X-ray Ring. Compared to the VUV Ring system, the new X-ray Ring systems eliminate three harmonic components of the orbit distortion instead of only one. In the low frequency circuits of the new systems, MDAC's (Multiplying Digital to Analog Converters) were used to replace the potentiometers in the Fourier analysis networks. This makes the system more accurate and easier to install than the VUV Ring system. The vertical feedback has been successfully in operation on the X-ray Ring since February 1991 and the horizontal system since September 1991, providing a dramatic improvement in orbit stability of more than a factor of five at all source locations (see Figures 1 and 2). During installation a method was developed to decouple several local feedback systems and operate them simultaneously with the global system. The global system not only greatly reduces the orbit movement but it also significantly reduces the required feedback correction strengths of the local feedback systems.

A horizontal global feedback system is scheduled to be installed in the VUV Ring by Spring 1992, followed by the installation of a new vertical system.

Fig. 1. Long-term and short term vertical beam position movement at PUE 31 on the X-ray Ring when the global feedback was turned on and off every 30 min. during an 8 hr. machine study period.
A major effort has been underway to improve the reliability and performance of the computer system. This effort has been focused on the design and installation of a distributed computing system. Various control systems have been examined with the aim of incorporating a complete control system into our facility. Work aimed at improving the existing control system has also continued. In particular, communication to the orbit measurement system was changed from a serial link to ethernet which resulted in a significant decrease in the time required to measure orbit data. The move to workstations has continued with the additions of new application software for the analysis and display of orbit and vacuum data.

A number of new systems were made operational this past year including the X13 undulator, X21 active interlock and X25 local feedback systems. The computer system for the X17 superconducting wiggler was upgraded to provide independent ramping and improved error diagnostic capability. In addition the global orbit feedback system was made operational on the X-ray Ring.

Effort has gone into improvements which are aimed at providing increased operational reliability of the NSLS facility. Work in this area includes a beamline status system which will provide the operations staff with the information to monitor the beamline front ends, vacuum, and water flow status. This information will help the operations staff determine and prevent the causes of beam dumps. Other work in this area included the conversion of some older multibus microcomputers to VME based systems. These systems included the booster and the single bunch micros.

References


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**NSLS COMPUTER SYSTEM**

*Ed Desmond*

**NSLS**

Fig. 2. Global improvement of orbit stability in the X-ray Ring upon closing the feedback loop. Solid curve represents orbit variation with feedback off and dashed curve with feedback on.
Beamline U3A

Los Alamos National Laboratory beamline U3A was converted from a white light line to a high throughput, low resolution line. U3A was formerly used with only a mirror and filter to provide a time resolved calibration of a GaAs(Fe) x-ray calorimeter. We were able to track the change in bunch length with time after a VUV Ring fill, but our detector response calibration was unsatisfactory without beam monochromatization. In 1991 we completed a new monochromator, added a new detector calibration end station, added a water cooled filter holder, and completed modifications to the shielding required by the beamline review committee. All hardware additions were completed and installed on the beamline. The monochromator was internally aligned. The converted U3A is ready for its commissioning period. It has vacuum isolation from the ring through an efficient differential pumping system. This permits the monochromator to operate at $1 \times 10^{-6}$ torr with motors, encoders, and moving parts inside the chamber. Monochromator motions and readouts are controlled by a computer. The exit is fixed while the energy is tuned over a range from IR to 2 keV by choosing among a selection of four pairs of dispersing elements, including crystals, multilayers, gratings, and mirrors. Resolution from the crystals and gratings can be high at the expense of flux, or vice versa. Most applications planned for U3A will use the high flux, low resolution mode. It is primarily a radiometric line, but will be made available to any qualified user who finds it suited to his scientific needs. R. L. Blake, B. Davis, R. G. Hockaday, and D. Holmberg (LANL).

The above figure shows a view of the U3A beamline during re-assembly. The large chamber at right-center houses the new monochromator. Barely discernable to its right is some beamline shielding around the differential pumping station. Two of three calibration chambers are located left (downstream) of the monochromator. The converted beam line is now in the commissioning phase.
Beamline U4IR

The high level of activity at the U41R Infrared beamline on the VUV storage ring is clear from the cluster of instruments surrounding the exit port. These include three Nicolet interferometers covering the wavelength region from 1 micron to 1 mm. In the foreground is the IR microscope used for high pressure diamond anvil cell studies. To the right is the ultrahigh vacuum surface science chamber used for studying vibrations at metal surfaces. In the center rear, incorporated into the spectrometer, is the chamber used for the high Tc and pump-probe studies. For these latter experiments a laser light from U10 is brought via a fiber optic cable. The synchrotron beam can be quickly switched between any of the above instruments. C. Hirschmugl and G. Williams (NSLS).

Beamline U16B

Modification and Installation of Cornell End Station

The current measurement chamber was significantly improved by the addition of a second chamber designed specifically for the insertion, modification and characterization of samples prior to transfer into the main measurement chamber. Operation of the modified new end-station in the final stages was evaluated on the beamline using a silicon single crystal target. Due to problems associated with stored beam operating characteristics and instrumental problems, the characterization studies of CoSi2 films and overlayers had to be rescheduled.

The modified end-station is expected to significantly enhance the output and quality of future in-situ film and catalyst studies on the beamline. T. N. Rhodin, R. P. Merrill, S. Woronick, and P. Ciszek (Cornell U.).

Design of High Sensitivity Soft X-ray Fluorescence Detector

A high sensitivity x-ray fluorescence solid state detector, obtained from Canberra Instruments, was experimentally evaluated for the quantitative detection of very small quantities of low-Z elements (nitrogen, carbon and oxygen) normally present in powders and films using near edge adsorption spectroscopy. An instrument of modified design was designed and will be installed based on this work. It is expected that the application of this new instrument will substantially enhance measurement of NEXAFS and EXAFS spectra on nonconducting samples. T. N. Rhodin, R. P. Merrill, and S. Woronick (Cornell U.).
Beamline X5

The target and detector stand for LEGS experiments on hydrogen and helium. The liquid target is enclosed in the transparent cylinder at the center of the assembly. Above the target is an array of forty-eight plastic scintillator bars to detect and identify neutrons and protons. Below the target are twenty-four plastic and CaF$_2$ scintillator phoswiches for detecting protons and pions. The entire apparatus, including target cryogenic systems, is mounted on rails so that it can easily be moved into and out of the gamma-ray beam, to allow other experiments to proceed while this apparatus is being modified and tested. It has been in use for experiments during the past year.
Beamline X8A

Los Alamos National Laboratory beamline X8A became operational in 1991. This general purpose line was developed to serve a variety of user-provided end stations, particularly for radiometric calibration of experimental systems to be used elsewhere and for scientific experiments in atomic physics, x-ray spectroscopy, and electron spectroscopy. A double focusing mirror and two crystal monochromators provide an end station beam about 2mm in size with very low harmonic content and stray light. Full operation to design specs has been achieved. Si(111) crystals provide high resolution from 2 to 6 keV with a flux up to $1 \times 10^{12}$ photons/sec at 5 keV. Beryl crystals give medium resolution from 0.9 to 2.5 keV. W/Si multilayers yield low resolution from 0.26 to 2 keV. Several successful calibrations were performed, including one that resolved a long standing uncertainty in a high temperature plasma diagnostic experiment.

The figure above is a view looking upstream from the end station area on X8A. A user brings his vacuum-compatible end station and attaches to a beamline valve and differential pumping system. A user vacuum from $1 \times 10^{-5}$ to $1 \times 10^{-11}$ torr can be accommodated. Shown here is a calibration end station. The experiment to be calibrated is centered in the picture with a three-axis manipulator just to its left; this permits beam profile scanning, imaging calibration of a separate detector, and other functions. Just to the right of the calibration package is the absolute reference detector: here it is a gas ion chamber. A differential pumping station upstream of the end station is a vital permanent part of this beamline, which often has end stations with poor vacuum and some detector gases that diffuse through thin windows. A lift table synchronized to the monochromator is also required for most experiments. R. L. Blake, R. G. Hockaday, and M. Sagurton (LANL).
Since 1960, the advances in crystallography studies of proteins have made great progress and have permitted the interpretation of enzyme structures at atomic resolution. This enables a discussion of the reaction mechanisms in terms of positions of atoms and atomic groups. Recently, X-Ray Absorption Spectroscopy (EXAFS) has become a valuable technique in determining the distances of metal centers of proteins in solution state. EXAFS, in combination with any transient technique like stopped flow, flash photolysis, and temperature jump methods, potentially provides information about dynamic structures of proteins during the course of reactions.

A schematic diagram of the stopped flow unit. It has a mixing time of 5–7msec and is capable of operating between −10°C to +40°C. A number of oxygen sensitive reactions could be studied when the samples were filled into syringes under inert atmospheric conditions.

Stopped flow apparatus during an EXAFS experiment on X9A. A large area photomultiplier tube (PMT) with ZnS phosphor was used to collect the x-ray fluorescence signal. Also shown is an R928 Hamamatsu PMT for reflectance optical monitoring of a sample during EXAFS data collection.
The optical signal at 390nm for a pyridinium chlorochromate ion. The reaction produces $\text{Cr}^{3+}$ from $\text{Cr}^{6+}$ ion during the course of $\text{CH}_3\text{OH}$-$\text{HCHO}$ oxidation.

The x-ray fluorescence signal for a 20mM chromate ion sample monitored at 5.996 keV. A substantial change in the near edge structure makes it ideal to calibrate the EXAFS-stopped flow instrument.

EXAFS spectra collected before and after the reaction from a large number of kinetic profiles shown above.
Beamline X10A

Couette Shear Cell designed for in-situ x-ray scattering of macromolecular fluids under flow. The purpose of this research program is to explore, via in-situ x-ray scattering techniques, new non-equilibrium steady-state structures of complex fluids that are produced under shear flow, due to the continuous input and dissipation of energy. Such experiments are among the few quantitative microscopic studies, probing length scales ≤ 10 Å and as large as ~μm of fluid materials under flow, and will lead to our understanding of phases and phase transitions away from equilibrium, and more generally of non-equilibrium statistical mechanical phenomena. For details see: C. R. Safinya, E. B. Sirotta, and R. Plano, "The Nematic to Smectic-A Phase Transition under Shear Flow: A Non-Equilibrium Synchrotron X-ray Study", Phys. Rev. Lett. 66, 1986 (1991).
Exxon's X10C beamline is designed specifically for x-ray absorption spectroscopy and anomalous x-ray diffraction. The beamline consists of horizontal and vertical entrance slits, UHV double crystal monochromator with AC feedback and a bent-cylindrical focusing mirror.

Many experimental techniques on focused beamlines require increased positional stability. These experiments are not limited by photon flux but by statistical and non-statistical errors in electron beam fluctuations, optic vibrations, monochromator tracking errors and angular deviations resulting from monochromator crystal heating.

We have developed and successfully tested a mirror feedback system on beamline X10C which is designed to dynamically minimize vertical beam motion at the sample position and compensate for these errors. The system functions by monitoring the beam position near the sample location with a position sensitive ion chamber coupled through a feedback loop to a piezoelectric transducer controlling the mirror tilt angle. The technique will reduce ±0.5 mm beam deviation to ±0.5 microns from DC to 10 Hz. This system has enhanced the quality of fluorescence and transmission XAFS data for very small samples, extremely dilute samples and inhomogeneous samples.

The figures above demonstrate the improvement in quality of XAFS data by using a mirror feedback system for a 0.3 mm by 1.0 mm single crystal sample. In this case, the vertical focus is of the same order as the sample size and small beam motions during the energy scan severely distort the Fourier transform. The experiment was performed in the fluorescence mode with a 13 element Ge-detector.

We are also developing a new electronics for an AC feedback system which locks on the peak of the Darwin rocking curve and dynamically compensates for monochromator crystal heating. The feedback electronics uses an amplifier stage with preset dead bands, and an automatic gain control (AGC) stage to modify the loop-gain through a transfer function which significantly extends dynamic range, reduces noise and enhances sensitivity. Mike Sansone (Exxon).
Beamline X11A

Focusing Crystal

Photograph of slotted triangular crystal employed for sagittal focusing at beamline X11A at the NSLS. The crystal was cut from a 0.76mm Si wafer into a triangle of base-width 66.5mm and height 85mm. Grooves were machined into the crystal face using a 0.05mm-wide diamond saw, such that the thickness of the weak links was 0.1mm. The face of the crystal has 74 grooves and thus 73 reflecting surfaces of width 0.75mm. The crystal is surrounded by an aluminum bracket to protect the crystal during handling. S. M. Heald (BNL Dept. of Appl. Sci.) and G. M. Lamble (NSLS/North Carolina State U.).

Focusing Crystal Images

Photograph of the images observed in the X11A hutch: (a) from the focusing crystal when flat, (b) the focused beam with a pre-monochromator slit height of 1mm, and (c) the focused beam with a pre-monochromator slit height of 0.5mm. The focused beam in case (c) is about 1.5mm. The intensities achieved by focusing over normal flat-crystal running, with a 1cm hutch slit-width, vary between a factor of four and two over the present operating energy range of 6 to 11 keV. S. M. Heald (BNL Dept. of Appl. Sci.) and G. M. Lamble (NSLS/North Carolina State U.).
A 44 year old male sustained an acute myocardial infarction on Feb. 19, 1991. On Feb. 25, cardiac catheterization revealed an 80% stenosis of the Right Coronary Artery (RCA) - see left hand CINE image. Balloon angioplasty was performed to re-open the RCA - see middle CINE image. Transvenous coronary angiography was performed on Aug. 22, 1991 at the Synchrotron Medical Research Facility (SMERF) showing that the RCA was still patent - see right hand SMERF image, Left Anterior Oblique 40° view. Beam parameters: X17 superconducting wiggler field was 3.0 Tesla; ring current was 206mA.
The polychromatic beamline X-17C is equipped with an energy disperseive x-ray diffraction (EDXD) system for probing ultra-small samples in high-pressure diamond anvil cells. The incident beam over an energy range of 5 to 100 keV is controlled by a double slit system made of tungsten and tantalum that is capable of producing uniform beam sizes of 5 to 10 μm impinging on samples in a diamond anvil cell. The alignment of the incident beam, sample, and diffracted beam are achieved by an automated Θ-20 system with a positional accuracy of 1 μm and angular accuracy of 0.001°. X-ray diffraction of polycrystalline samples at multimegabar pressure range and at temperatures of 50 - 3000 K have been performed. J. Z. Hu et al. (Carnegie Inst. of Washington).

A cryogenic system for single-crystal EDXD at high pressures. The sample, which is a single-crystal helium, is grown from its fluid phase at 11 GPa at room temperature in a "membrane" diamond cell. The pressure is generated by a membrane press using gaseous helium as the hydraulic fluid. The x-rotational circle, which contains the diamond cell at its center, is cooled in a liquid-helium cryostat, which in turn is located on the Θ-20 sample stage. The pressure at low temperatures can be measured simultaneously with a ruby fluorescence system which includes a He-Cd laser as the excitation radiation source, a microscope for steering the laser to a ruby chip at high pressures and for collecting the fluorescence, and a polychromatic spectrometer for measurement of the pressure-shift of the ruby R-line. P-V-T equation of state, crystallographic structure, and phase diagram of helium have been determined in this system. R. LeToullec et al. (U. of Paris) and J. Z. Hu et al. (Carnegie Inst. of Washington).
Beamline X18A

The figure shows a UHV chamber at the MATRIX beamline X18A which has recently been completed for structural analysis of adsorbed films and single-crystal surfaces. It is particularly well suited for investigations of physisorbed and other weakly bound films due to its combined in-situ LEED and low-temperature capabilities. The chamber is transportable and mounts on a standard Huber x-ray diffractometer. The utility of these features for studies of the structure and phase transitions of physisorbed films has been demonstrated in some preliminary investigations of the Xe/Ag(111) system. J. R. Dennison (Utah State U.), S.-K. Wang, P. Dai, H. Taub, T. Angot (U. Missouri-Columbia), and S. N. Ehrlich (Purdue U.).

Beamline X23A3

A new small-angle x-ray scattering instrument, optimized for high throughput at the NSLS, high angular and wavelength resolution, large sample cross-sectional area, accurate energy tuning, excellent signal-to-noise ratio and harmonic rejection has been commissioned on X23A3. The instrument has been used for anomalous scattering measurements near the Cr K-edge, for measurements of pore-size distributions in ceramics, and for the study of disordered structures, as well as for a number of other materials systems.
Cryogenic X-Ray Optics

The X25 wiggler white beam, when doubly-focused, has a power density exceeding 150 W/mm$^2$ at the focal point. This is a representative density which optics at next-generation synchrotron source undulator beam lines will have to withstand. Crystal monochromators cannot diffract x-rays efficiently, nor preserve the high brightness of the source, unless surface strains are kept within a few arcseconds. This is impossible to achieve for all materials at room temperature in the presence of the X25 doubly-focused beam. At cryogenic temperatures, however, thermal strains can be reduced substantially, since the thermal expansion coefficient becomes very small and the thermal conductivity increases in many materials. In a joint ESRF/NSLS experiment at X25, the diffraction efficiency of a silicon crystal was measured as a function of temperature, using the doubly-focused incident beam. Plotted above is the slope error across the crystal surface, determined from x-ray rocking curves, compared with the ratio of the thermal expansion coefficient to thermal conductivity. As expected, the observed slope error is close to zero at 100 K, resulting in perfect diffraction efficiency.
Section VI
Informational Guides
## INFORMATIONAL GUIDE TO THE NSLS VUV BEAM LINES

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O = Operational (beam line is actively used in research).
M = Commissioning (beam line is built but is being run for the sole purpose of detecting flaws in the configuration).
C = Construction (beam line is being assembled).
P = Planned (beam line design is completed but construction has not yet begun).
N = Conceptual (pre-design stage).

ARUPS = Angle-Resolved Ultraviolet Photoemission Spectroscopy.
XPS = X-ray Photoemission Spectroscopy.
EXAFS = Extended X-ray Absorption Fine Structure.
SEXAFS = Surface EXAFS.
ERG = Extended Range Grasshopper.
CD = Circular Diachroism.
C-T = Czerny-Turner.
IR = Infrared Interferometer.
LEGS = Laser Electron Gamma Source.
MOD = Magnetic CD.
PGM = Plane Grating Monochromator.
TGM = Toroidal Grating Monochromator.
NIM = Normal Incidence Monochromator.
SGM = Spherical Grating Monochromator.
S-N = Seya-Namioka.
SXES = Soft X-ray Emission Spectrometer.
TOK = Transverse Optical Krystron.
UPS = Ultraviolet Photoemission Spectroscopy.
NEXAFS = Near Edge X-ray Absorption Fine Structure.
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Appendices
List of Abstracts: VUV Beamlines
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Characterization of Fluorocarbon Polymer Films and Adsorbed Protein by Ultra-Soft X-ray Absorption Spectroscopy
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<td>R. Bartlett, P. Walsh, J. Samson, and Z. He</td>
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<td>Partial Photoionization Cross Sections of Ne Across the K-Edge</td>
<td>R.J. Bartlett, P.J. Walsh, and J. He</td>
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<td>L.M. Hirsch and T.J. Shankland</td>
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<td>Photoemission Study of Electronic Structure Near EF in (\text{YBa}_2\text{Cu}_3\text{O}_6) for 6.3 &lt; x &lt; 6.9</td>
<td>R. Liu, B.W. Veal, A.P. Paulikas, J.W. Downey, A.J. Arko, and J.J. Joyce</td>
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<td>Calibration of a Soft X-ray Film Spectrograph</td>
<td>T.J. Tanaka, N.D. Shinn, R.E. Ripple, A. Widman, and M.A. Palmer</td>
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<td>ARPES Study of (\text{YBa}_2\text{Cu}_4\text{O}_8) and (\text{YBa}_2\text{Cu}_3\text{O}_6)</td>
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**Beamline X7B**


**Beamline X9A**


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Beamline X12 A & B


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Beamline X14


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Beamline X15A


Beamline X16A


Beamline X17B1


Beamline X17B2


Beamline X18A


Beamline X18B


**Beamline X19A**


**Beamline X19C**


Beamline X20


Beamline X22B


Beamline X22C


Beamline X23A2


Beamline X23A3


Beamline X23B


Beamline X24A


**Beamline X24C**


**Beamline X25**


**Beamline X26A,C**


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