National Synchrotron Light Source ◆ Brookhaven National Laboratory

March 2000

Newsletter

High-Resolution X-Ray Fluorescence Spectroscopy at X-25

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The x-ray spectrum resulting from atomic fluorescence transitions has been the subject of study for more than a century. Chemical effects have been known for a long time[1] but substantial work has focused on identifying lines from different elements rather than studying their chemical dependence. The strongest transitions such Kα are used routinely as an analytical tool for elemental analysis and particle characterization as well as detection channel for x-ray absorption spectroscopy (XAS) of dilute systems[2]. During the last 15 years, with the advent of more intense x-ray sources and efficient high resolution analyzer devices, x-ray fluorescence spectroscopy has received more and more attention as a valuable tool for quantitative chemical and structural analysis. The new techniques enable the study of extremely dilute systems such as transition metals in proteins and weak fluorescence lines.

In the following, we give a brief overview of some of our work performed on Beamline X-25 over the past two years. We will concentrate on the K fluorescence of Mn compounds and try to illustrate with a few examples how each part of the spectrum is affected by chemical and/or structural changes of the local Mn environment. We will not discuss resonant techniques or XAS detected with high resolution, which by themselves present an interesting field of research. Except for one example, the initial state is a Mn *1s* core hole excited well above the Mn K edge at 6.54 keV.

The work has become possible by the use of a newly designed multi-crystal X-ray spectrometer, based on several spherically curved perfect crystals in Rowland geometry[3]. At Bragg angles close to back scattering a solid angle of up to 0.5% of the total 4π sr is captured with sub eV energy resolution. The good energy resolution is crucially dependent on the back scattering condition, and the typical range for one reflection order is ~100 eV. For the analysis of Mn Kα fluorescence, we used the Ge (3,3,3) reflection ($\theta_B \sim 75^o$) for all of the Mn Kβ work we used the Si(4,4,0) reflection ($\theta_B = 79^o$-87^o).

Figure 1 shows an overview of the Mn K fluorescence of Mn(II)O. At the lowest energy is the spin orbit split $K\alpha_1$/$K\alpha_2$ doublet (*2p* →*1s*) and 600 eV higher the eight times weaker Kβ main region (*3p* →*1s*), split into $K\beta_{1,3}$ and Kβ' through *3p - 3d* exchange interaction. At even higher energies, much weaker transitions from valence levels are apparent after magnifying the spectrum by a factor 500. The $K\beta_{2,5}$ region results from transition with ligand *2p* as well as metal *4p* and *3d* character. The Kβ" peak corresponds to 'interatomic' or 'crossover' transitions from ligand *2s* levels. At excitation energies sufficient to simultaneously create Mn *1s* and *2p* holes, spectral features can be observed even above the Fermi level indicated by the dashed line. These lines, referred to as KLβ or Kβ''', are *3p* → *1s* transitions with a *2p* spectator hole. Their energies can be esti-

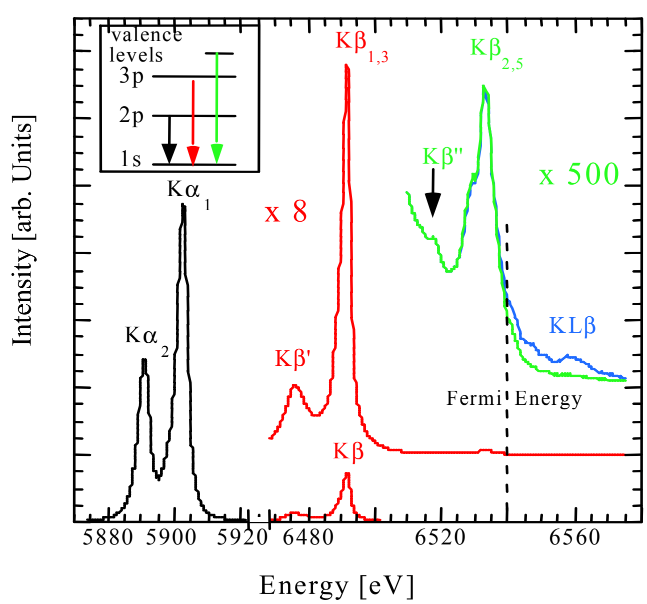


Figure 1: *Mn K fluorescence spectrum of Mn(II)O*

mated using the Z+1 model with the *2p* hole adding one effective nuclear charge to the potential that acts on the *3p* electrons. For Mn, this results in a shift of +60 eV corresponding to the energy difference between Fe and Mn L edges (*2p* binding energy). How are different parts of the spectrum affected by changes in the local Mn environment?

Figure 2 shows effects of Mn valency on different peak energies. Both $K\alpha_1$ and $K\beta_{1,3}$ shift to lower energies with increasing oxidation state, whereas the $K\beta_{2,5}$ main peaks shift in the opposite direction. The origin for the shifts of $K\alpha_1$ and $K\beta_{1,3}$ lies in the respective exchange interaction between the *2p* and *3p* hole with the unpaired *3d* electrons. The *2p-3d* Slater integrals are by a factor 2 to 3 smaller than those for *3p-3d*. Consequently, $K\alpha_1$ shifts less than $K\beta_{1,3}$. The strongest shifts are observed in $K\beta_{2,5}$ and, similar to the K absorption edge, the shifts are to higher energy with higher oxidation state reflecting an increase of the *1s* binding energy when *3d* electrons are removed. Studies of $K\beta_{1,3}$ lines have been used as an independent probe to characterize the oxidation states of the Mn cluster in photosystem II, the protein complex responsible for photosynthetic reduction of water and oxygen release [4-6].

Figure 3 shows the summary of a study of the Kβ'' region resulting from 'interatomic' ligand (N,O,F) *2s* to Mn *1s* transitions[7]. The energy of Kβ'' is very distinct for each of these ligands of neighboring Z. Furthermore, as shown on in the inset for a variety of oxygen ligated Mn compounds, the intensity of Kβ'' decreases exponentially with the ligand distance. We estimate that the method can be used to determine distances to ~0.1Å accuracy if the num-

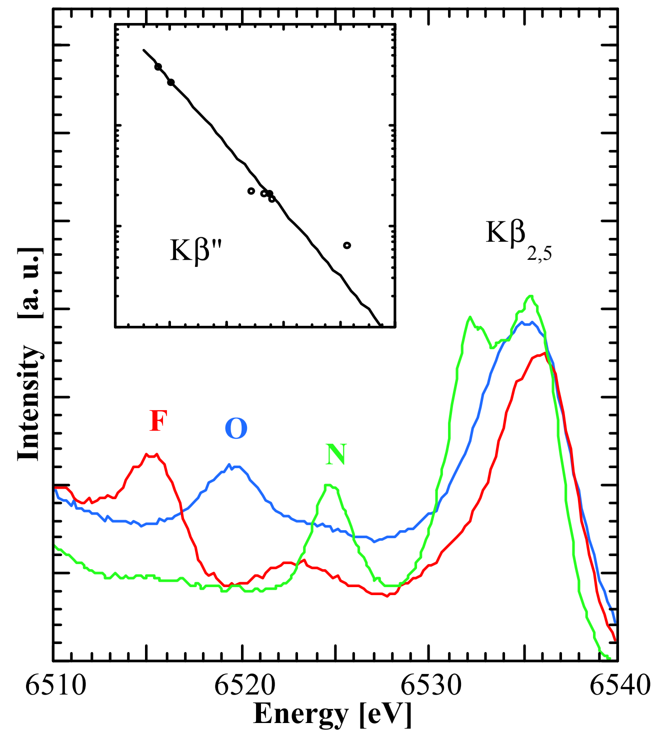


Figure 3: *Kβ'' and* $K\beta_{2,5}$ *regions for compounds with different ligands:* MnF_4 *(cross),* MnO_2 *(circle),* $[Mn(N)(CN_5)]^{3-}$ *(triangle). Inset: Dependence of integrated Kβ''' intensity normalized to the number of ligands as a function of ligand distance.*

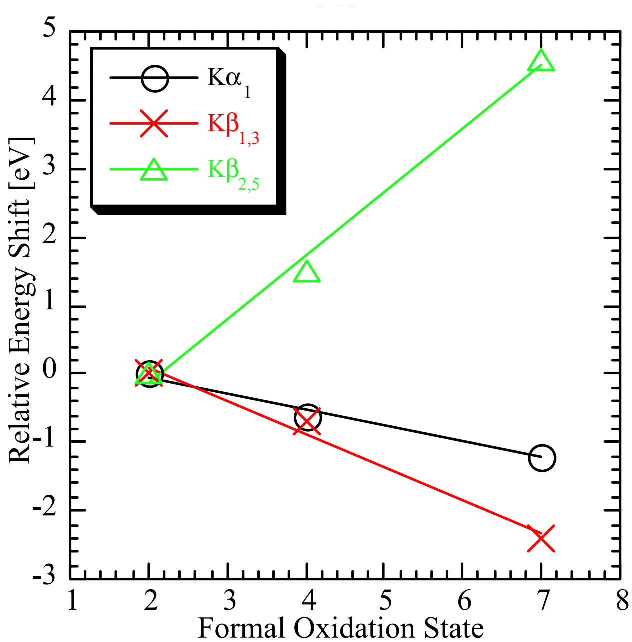


Figure 2: *Peak positions of K fluorescence features for Mn(II)O, Mn(IV)*O_2 *and KMn(VII)*O_4

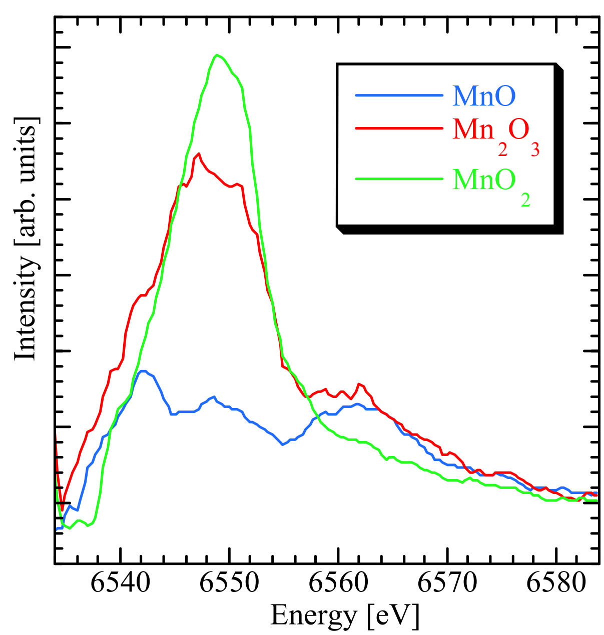


Figure 4: *KLβ satellites for different Mn oxides. The tail of the* $K\beta_{2,5}$ *spectrum taken below the threshold of double ionization is subtracted*

ber of ligands is known. $K\beta''$ unambiguously discriminates between ligands such as O versus N, demonstrating its potential as a unique alternative for systems which defy structural analysis using other techniques. Examples of possible future applications in biology are studies of the binding of O and Cl to the Mn cluster of photosystem II and the nature of the Fe-Ni bridging ligand in hydrogenase enzymes.

Fluorescence features above the Fermi level known as $KL\beta$ or $K\beta'''$ satellites are the result of multi electron (*1s* and *2p*) excitations with the *2p* hole acting as spectator during the core hole decay. We have recently reproduced studies by Deutsch *et al.*, which show that the evolution of $KL\beta$ as a function of excitation energy does not reveal any sharp resonances indicating a predominant shake-off mechanism as opposed to a shake-up transition of the *2p* electron into a bound state[8]. Furthermore we studied $KL\beta$ for a series of Mn compounds and observed a pronounced dependence of line shape and intensity on the chemical environment (**Figure 4**). Future work will show to what extent $KL\beta$ satellites can be utilized in the characterization of chemically relevant systems.

In conclusion, every part of the K fluorescence spectrum of Mn is sensitive to its chemical environment, and as a trend we observe that weaker lines are more strongly affected. 'Interatomic' transitions have a unique sensitivity to the ligand type and distance, whereas core to core transitions such as $K\alpha$ and $K\beta_{1,3}/K\beta'$ are mostly effected by the number of unpaired *3d* electrons and hence the oxidation or spin state. $K\beta_{2,5}$ and $KL\beta$ show strong spectral changes with both valency and ligand type, and efforts in better theoretical understanding of these features are underway. Currently, we can record the whole $K\beta$ spectrum of concentrated compounds with very good statistics in one hour. High quality spectra of extremely dilute systems such as photosystem II (containing 10 ppm Mn) have been obtained up to the $K\beta_{1,3}/K\beta'$ region and the basic features of $K\beta_{2,5}$ and $K\beta''$ were observed. Using multilayers and/or more intense synchrotron sources in the near future, we should be able to study weak transitions also in these dilute systems. ■

References

[1] A. Meisel, G. Leonhardt, R. Szargan, "X-ray Spectra and Chemical Binding," [translated and edited by E. Kallne *et al*.] Berlin; New York: Springer-Verlag. Springer series in Chemical Physics; v. 37 (1989).

[2] J. Jaklevic, J.A. Kirby, M.P. Klein, A.S. Robertson, G.S. Brown, and P. Eisenbeger, "Fluorescence Detection of EXAFS: Sensitivity for Dilute Species and Thin Films. Solid State Communications, Vol. 23, 679-682, (1977).

[3] U. Bergmann, and S.P. Cramer, "A High-Resolution Large-Acceptance Analyzer for X-ray Fluorescence and Raman Spectroscopy," *SPIE Proc.,* Vol. 3448, 198-209, (1998).

[4] U. Bergmann, M.M. Grush, C.R. Horne, P. DeMarois, J.E. PennerHahn, C.F.Yocum, D.W. Wright, C.E. Dube, W.H. Armstrong, G. Christou, H.J. Eppley, and S.P. Cramer, "Characterization of the Mn Oxidation States in Photosys tem II by Kb X-ray Fluorescence Spectroscopy," *J. Phys. Chem. B*, Vol. 102 (N42), 8350-8352, (1998).

[5] R.M. Cinco, C. Fernandez, J. Messinger, J.H. Robblee, H. Visser, K.L. McFarlane, U. Bergmann, P. Glatzel, S.P. Cramer, K. Sauer, V.K. Yachandra, and M.P. Klein, "Refined Model of the Oxidation States and Structures of the Mn/Ca/Cl Cluster of the Oxygen Evolving Complex of Photosystem II," published in 'Photosynthesis: Mecha nisms and Effects', (Garab, G. Editor): Kluwer Academic Publishers, 1273-1278, (1998).

[6] J. Messinger, J.H. Robblee, C. Fernandez, R.M. Cinco, H. Visser, U. Bergmann, P. Glatzel, S.P. Cramer, K.A. Campbell, J.M. Peloquin, R.D. Britt, K. Sauer, V.K. Yachandra, and M.P. Klein, "Oxidation States and Struc ture of the Manganese Cluster in the S_0 State of the Oxygen Evolving Complex," published in 'Photosynthe sis: Mechanisms and Effects', (Garab, G. Editor): Kluwer Academic Publishers, 1279-1282, (1998).

[7] U. Bergmann, C.R. Horne, T.J. Collins, J.M. Workman, S.P. Cramer, "Chemical Dependence of Interatomic X-Ray Transition Energies and Intensities - A Study of Mn $K\beta''$ and $K\beta_{2,5}$ Spectra. *Chem. Phys. Lett.,* Vol. 302, 119-124, (1999).

[8] M. Deutsch, O. Gang, K. Hämäläinen, C.C.Kao, "Onset and Near Threshold Evolution of the Cu X-Ray Satellites," *Phys. Rev. Lett.,* **76**, 2424, (1996).

Rapid Data 2000 Course
"Rapid Synchrotron Macromolecular Data Collection,"
offered at BNL April 9-14, 2000

The Biology Department and the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory announce their new course for Rapid Data Collection and Structure Solving in Macromolecular Crystallography, to be held at the NSLS from 9-14 April 2000.

Students may apply to take data on THEIR OWN SPECIMENS. Students have come from a wide range of levels — from graduate students to full professors.

Please see the course web site at:
http://www.x12c.nsls.bnl.gov/rr_course_2k/course_announce.html

Application materials can be found at:
http://www.x12c.nsls.bnl.gov/rr_course_2k/applic.html

And a report of last-years course can be found at:
http://www.x12c.nsls.bnl.gov/rr_course/

Chairman's Introduction

Michael Hart
NSLS Chairman

Just before the start of the 20th century, our vision of the electromagnetic spectrum was expanded from the visible and near infrared to cover the range from radio waves to x-rays. Another half century passed before lasers and synchrotron radiation appeared and became, over many years of technical development, the sources of choice for a wide range of applications. By the end of the century, lasers had been developed over wide wavelength ranges from the ultra violet to radio wavelengths with enormous power, and exquisite control of wavelength, time structure, polarization and coherence while applications of synchrotron radiation had been extended from hard x-rays to the far infrared. *Dedicated* storage ring based facilities had become the norm, energies had reached 8 GeV and diffraction limited performance had been demonstrated over most of the spectral range. Control of polarization and time structure continues to advance. There is no sign of any leveling off in the growth of the user community or in the range and scale of the applications in all areas of science and technology.

The last NSLS Newsletter of the 20th century reported an important milestone in the research necessary to develop the next generation light source. A collaboration of scientists from BNL and ANL lead by Li-Hua Yu from the NSLS reported the successful demonstration of High Gain Harmonic Generation (HGHG) in a Free Electron Laser (FEL) at the FEL-99 conference in Hamburg Germany in August. This important result shows dramatically the potential benefits of a laser seeded FEL as contrasted with the self amplified spontaneous emission (SASE) approach pursued at other laboratories. The December 1999 issue of Synchrotron Radiation News carried invited articles about new concepts in storage rings under the headline "Future Light Sources," showing no signs of saturation or shortage of ideas for the future.

The **table** shows a few of the x-ray synchrotron radiation facilities, above 2 GeV in energy, operating now [O] or planned for the 21st century.

Storage Ring	E [GeV]	C [m]	ε [nm-rad]	I [ma]
Elettra [O], Italy	2	259	7	300
SLS Switzerland	2.4	288	4.4	400
Soleil France	2.5	337	3	500
ANKA Germany	2.5	110	39	400
NSLS [O], USA	2.584	170	45	350
NSLS, USA	2.8	170	53	250
CLS Canada	2.9	170	18	500
Diamond UK	3	397	3-8	300
Boomerang Australia	3	164	16	300
Spear 3 USA	3	234	18	200/500

Clearly, a very large expansion is foreseen world wide and, equally, clearly there has been a strong convergence of views relating to the design optimization. The shift to lower energy storage rings is enabled by the availability of new technology and design concepts and driven by cost considerations.

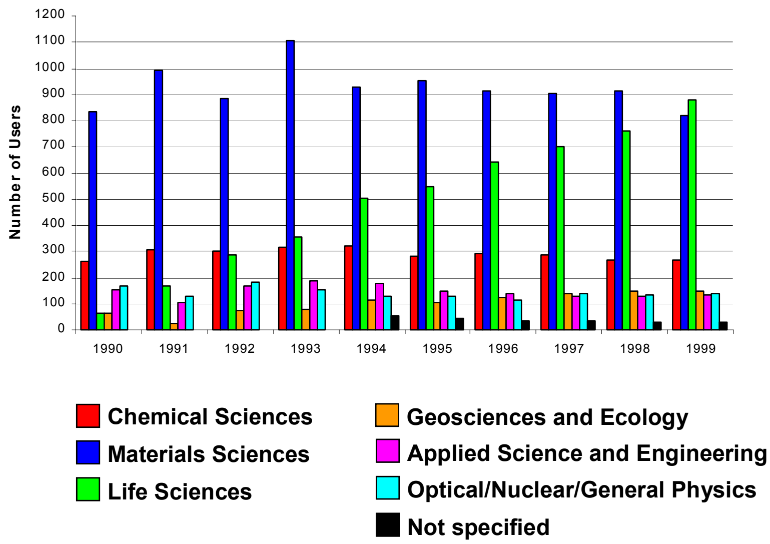
It is difficult to estimate the growth in the user community because users travel worldwide between facilities but journal surveys show a steady increase in the range and number of publications with no sign of saturation. A major review (by the Department of Energy Basic Energy Sciences Advisory Committee, Chaired by Dr. R. Birgeneau) of the achievements of the last decade or two in the United States and the prognosis for the future concluded:

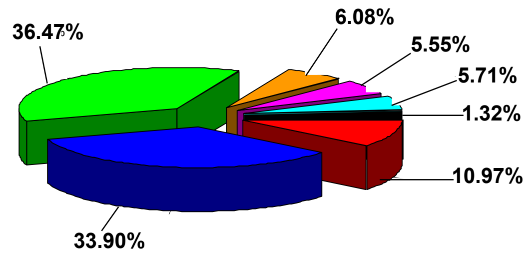
"The most straightforward and most important conclusion of this study is that over the past 20 years in the United States synchrotron radiation research has evolved from an esoteric endeavor practiced by a small number of scientists primarily from the fields of solid state physics and surface science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

An indication of present and future participation can be seen in the user statistics for the NSLS. In the final year of the century, Structural Biologists became the largest user group (**Figure 1**) but they still account for only about 10% of the station beamtime. At the turn of the century, the total number of individual users of the NSLS (since its inception) was 8464 and nearly 10% of them came in 1999 for the very first time. As **Figure 2** shows, although the number of users per year has been roughly constant at about 2400 for almost a decade, the user community is not stagnant but is continually renewed. More than a third are under 31 and more than two-thirds are younger than 41 years in age. Here are clear indicators that synchrotron radiation light sources in general and the NSLS in particular are thriving and will do so well into the new century. ■

NSLS Users by Field of Research

(1990 – 1999)





Type of Science	1990	1991	1992	1993	1994	1995	1996	1997	1998	1999
Chemical Sciences	264	304	303	318	321	281	292	285	268	265
Materials Sciences	837	993	882	1104	929	952	916	902	914	819
Life Sciences	62	167	286	354	503	548	642	701	761	881
Geosciences and Ecology	62	26	75	80	113	106	123	136	147	147
Applied Science and Engineering	155	106	167	186	177	147	138	126	127	134
Optical/Nuclear/General Physics	170	129	183	151	129	128	116	136	131	138
Not Specified	0	0	1	0	56	44	34	34	32	32
Total	1550	1725	1897	2193	2228	2206	2261	2320	2380	2416

Figure 1: *NSLS Users by field of research (1990-1999)*

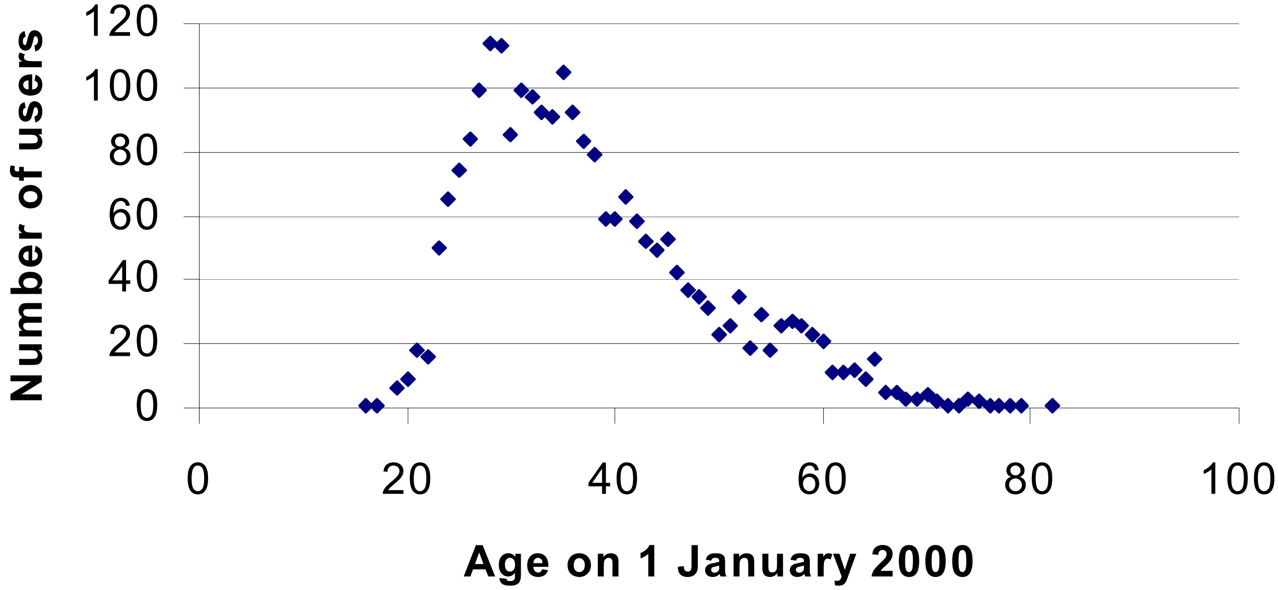


Figure 2*: NSLS Users by age*

A Users' Perspective

Barbara Illman, Users' Executive Committee Chair
USDA FS Forest Products Lab, University of Wisconsin

Did anyone have any doubt? The year 2000 opened at NSLS without Y2K problems. The new millenium opened with other problems for the NSLS. At the November Town Meeting, Director Michael Hart reported that the $4 million from NIH keeps the NSLS budget "status quo," but if it is taken away in two years, the impact from the deficit will be severe. NIH has indicated that the money will not be given as planned. I think the users and NSLS administration need to continue to work together to ensure the healthy future of this facility. Actions of the UEC members include writing letters to Congress, visiting NIH and contacting BNL management. I think the NSLS users need to express support for the opportunity they have to conduct research at this world-class facility.

Users have a unique opportunity at the Annual User's meeting this year to be heard by DOE, BNL and NSLS. The Scientific Advisory Committee (SAC) that makes recommendations about NSLS has agreed to meet with users May 23 in Berkner Hall at 3-3:15 p.m. till 4:30 p.m. Some agenda items are:

1. Status and outcomes of tenure reviews, what has SAC learned from the process?
2. Evaluation of proposal submissions and roles of PSPs.
3. Scientific vision for facility as seen by SAC and changing role for NSLS.
4. Critical infrastructure issues facing us.
5. Relationship of NSLS to BNL as a national laboratory.

The 2000 NSLS Annual Users' Meeting and Workshops will be held May 22-24, 2000. Highlights of the meeting include a one-day meeting, reception, poster session and poster contest, instrumentation and equipment exhibit and two days of workshops. One-day workshops will focus on Very Bright Infrared Sources and Applications, New Approaches to Solving Protein Crystal Structures, Environmental and Geological Sciences, XAFS of Dilute Systems, Chemical Applications of Synchrotron Radiation, and Collaboratoria. I encourage you to view the agenda at **http://nslsweb.nsls.bnl.gov/nsls/users/meeting/2000/request-pkt.htm**. I want to personally thank the organizers who have worked hard to put together the 2000 Meeting. They are Mark Chance, Meeting Chairman; Chris Jacobsen, Program Chair; Simon Bare, Workshop Chair; Peter Stephens, Poster Session; Linda Feierabend, NSLS Meeting Coordinator; Nancye Wright, NSLS Equipment Exhibitor; Mary Anne Corwin, NSLS User Administrator.

All users can take part in this community by contacting any of the UEC members listed below or sending comments and questions to me at billman@facstaff.wisc.edu

Members of the 1999-2000 NSLS Users' Executive Committee

Top row: Mary Anne Corwin (NSLS User Administrator), Lisa Kelly (UEC Secretary, Univ. of Maryland), Carol Hirschmugl (Univ. of Wisconsin), Steve Ehrlich (NSLS), Lisa Miller (NSLS), Ken Evans-Luderodt (Lucent Technologies), John Hill (BNL Physics), Jean Jordan-Sweet (IBM Research Div.), Dale Sayers (North Carolina State Univ.). Bottom row: Chris Jacobsen (SUNY Stony Brook), Erik Johnson (NSLS), Mark Chance (UEC Vice Chair, AECOM), Barbara Illman (UEC Chair, USDA/Univ. of Wisconsin), Michael Dudley (SUNY Stony Brook) and John Parise (UEC Past Chair, SUNY Strony Brook). Missing from the photo: Malcolm Capel (BNL/ Biology), George Cody (Carnegie Inst. of Wash.), Mark Lucas (Ohio Univ.), Larry Carr (NSLS) and Robert Bartynski (Rutgers Univ).

This will be my last column as UEC chair. I want to thank Michael Hart and the User Administration Office for their cooperation and help. They have worked with the UEC on every user issue we have had and have worked to maintain the excellence of the NSLS - the most user-friendly and cost effective of the nation's sources. The organization of town meetings, the annual user meetings, the Activity Report, day-to-day contact with users and a host of other affairs would be considerably more difficult were it not for Mary Anne Corwin, Linda Feierabend, Nancye Wright, Eileen Pinkston, and Lydia Rogers. The UEC relies heavily on them.

My best wishes to you all. See you 'round the rings.'

ATF Sets New Standard on Relativistic Thomson Scattering

Igor Pogorelsky
NSLS-ATF

Introduction

The concept of an x-ray and gamma-ray laser synchrotron source (LSS) based on Thomson scattering (or inverse Compton scattering) between laser photons and relativistic electrons is an example of a symbiotic relationship between accelerators and high-power lasers that may lead to novel femtosecond light source facilities. Enticed by these prospects, the BNL Accelerator Test Facility (ATF) started proof-of-principle study of the LSS based on a combination of the photocathode rf linac and picosecond CO_2 laser (BNL Report #62447, 1995). In 1998, this study evolved into the Japan-US collaboration on development of the polarized gamma source for Japan Linear Collider. The first collaborative Thomson scattering experiment conducted at the BNL ATF in September 1999 set a new record in the x-ray yield, intensity and brightness for the relativistic Thomson sources.

The success of the ATF experiment is based on a systematic approach to optimization of the relativistic Thomson scattering process for the maximum photon yield. Two prime factors contribute towards this goal. First, the ATF LSS benefits from the fact that the CO_2 laser beam (λ=10 µm) carries 10 times more photons than a solid state laser (λ=1 µm) of the same power. Second, in designing a high-yield LSS, we choose the most efficient counter-propagation interaction geometry.

The reported experiment done with a gigawatt CO_2 laser is the first step towards the future femtosecond x-ray LSS. The advanced 1000 times more powerful LSS will be demonstrated before the end of 2000 upon completion of the ATF laser upgrade to the terawatt level.

BNL ATF Laser Synchrotron Source experiment

A principle diagram of the ATF CO_2 LSS experiment is shown in **Figure 1**. The electron beam produced by the photocathode rf gun and accelerated to 60 MeV (γ=120) in the ATF rf linac is quadrupole focused in the middle of the interaction cell. Steering coils allow transverse adjustment of the e-beam position. Electron beam parameters in the interaction point are: bunch charge 0.5 nC, energy spread 0.15%, normalized emittance ε_n=2 mm mrad, bunch duration 3.5 ps, focus spot size σ_b=32 µm. Recollimated after the interaction cell, the electron beam is deflected by the dipole magnet that separates it from the backscattered Thomson x-rays.

The 0.6 GW, 180 ps pulses generated by the ATF CO_2 laser are sent in a head-on collision with the e-beam and focused at the interaction point. In order to focus the CO_2 laser beam tightly, the short focal length optical element (parabolic copper mirror with the focal length of F=15 cm) is placed on the way of the e-beam. Naturally, the mirror has a hole (5 mm in diameter) drilled along the e-beam axis. This hole transmits both the electrons and the backscattered x-rays. To avoid the laser energy loss and the material ablation at the hole edge, the initially Gaussian laser beam profile is telescoped into the "donut" shaped beam using a pair of ZnSe axicon lenses. Stepper motors provide two-axis tilt of the parabolic mirror for precision positioning of the laser focus on the e-beam. The spent laser beam is extracted from the vacuum cell and recollimated onto the optical diagnostic using a similar parabolic mirror with a hole.

The e-beam spot size in the interaction region was measured by the wire scan (**Figure 2a**). Then, the laser spot size can be determined measuring Thomson x-ray signal by transverse scanning of the electron beam across the interaction point (**Figure 2b**). The results of these scans indicate that the laser focus closely matches the electron beam size with σ_L=σ_b=32 µm.

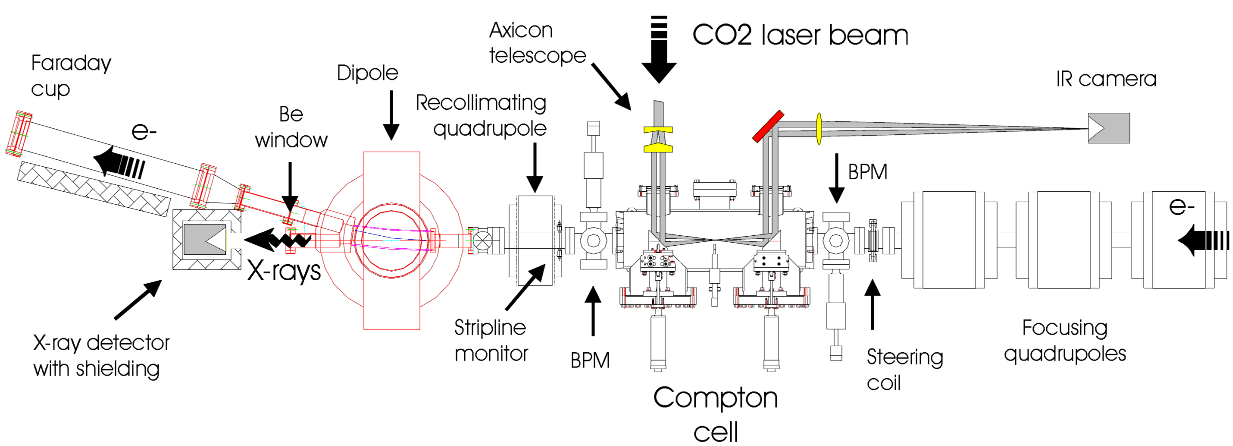


Figure 1: *Diagram of the ATF Thomson scattering experiment*

In the counter-propagating configuration, the temporal shape of the electron bunch defines the x-ray pulse. The bunch shape was measured by adjusting the phase of the linac rf field to produce a linear energy chirp to the electron bunch arriving to the monochromator positioned after the linac. A collimating slit in the dispersive region of the monochromator filters out a narrow slice of the bunch that is measured by a Faraday cup. The result of these measurements is 3.5 ps FWHM as shown in **Figure 2c**.

A variable optical delay permits timing adjustment between the electron bunch and CO_2 laser pulse. Incidentally, this provides a tool to measure the CO_2 laser pulse profile. **Figure 2c** shows the observed dependence of the Thomson signal upon the time delay. Because the Thomson signal is linear proportional to the laser intensity, the obtained plot characterizes the time structure of the laser pulse. Based on these observations, the laser pulse is 180 ps FWHM. The 600 MW peak power is obtained by time integrating the plot in **Figure 2c** and normalizing it to the 200 mJ energy in the pulse.

The Thomson x-rays diverging within a cone with the opening angle of $\theta=1/\gamma$=8 mrad are detected with the 20 mm aperture Si diode placed 1.5 m downstream from the interaction point. On the way to the detector, the x-ray beam passes a 250 μm thick Be window and propagates 20 cm in the air.

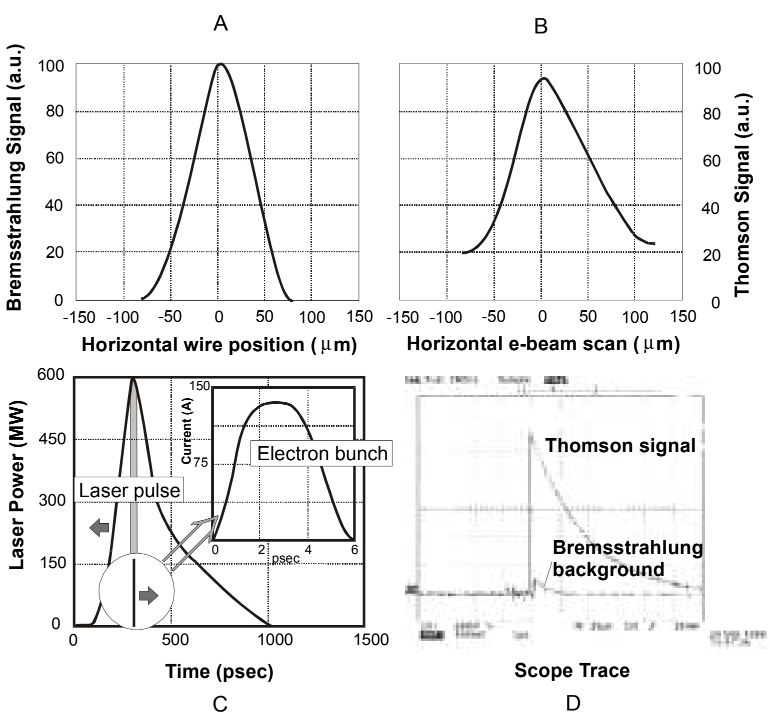


***Figure 2:** Experimental results from the ATF Thomson scattering experiment. A – transverse wire scan of the e-beam focus shows σ_b=32 μm; B – laser/electron cross-correlation indicates closely matched focal spots $\sigma_L=\sigma_b$=32 μm; C – 3.5 ps electron bunch counter-propagating with the 180 ps laser pulse radiates 3.5 ps x-ray pulse, shaded is a portion of the laser pulse that is utilized for the x-ray production (fits into the laser waist length); D – Thomson x-rays are detected at a high signal-to-background discrimination.*

For the reported experiment conditions, the maximum scattered photon energy that results from the 0.113 eV CO_2 laser photon upshift by the 60 MeV electrons was 6.5 keV (1.8 Å). The detected minimum photon energy was 5 keV. This has been verified by placing spectrally selective Ti and Ni foil filters in front of the Si detector. The minimum energy threshold is due to the combined effect of the angle acceptance of the Si detector and x-ray absorption in the Be window and the air.

At the matched focusing and exact alignment and synchronization of the laser and electron beams, the researchers were able to observe strong Thomson x-ray signal on the Si detector, much above the background level (due to the 60 MeV electron bremsstrahlung). A typical S/N ratio was up to 100 (see **Figure 2d**). The maximum measured signal was 2.2 V.

Calibration of the Si detector done in the NSLS x-ray beamline shows that 2.2 V of the detected signal corresponds to 2.9×10^6 photons/pulse. A correction on the spectral transmission of the beryllium and the air gives 6.4×10^6 photons in the 5-6.5 keV spectral range before the Be window. 27% of the total photon number in the Thomson spectrum falls into this spectral window. Thus, 2.4×10^7 photons/pulse were produced via Thomson scattering at the laser–electron interaction. Dividing these numbers by the electron bunch length, one can obtain 8×10^{17} photons/second flux on the detector or 7×10^{18} photons/second flux generated at the interaction region. The spectral bandwidth is estimated at 1%.

Next, the measured photon flux was compared with the theoretically expected value for the conditions of the BNL ATF LSS experiment. The expected number of Thomson scattered photons in the $\theta=1/\gamma$ cone calculated by the Monte-Carlo code CAIN is 2.8×10^7 photon/pulse. This number closely matches the result derived from the x-ray detector signal and confirms the highest photon yield ever demonstrated via laser Thomson scattering on relativistic electron beams.

Near future with terawatt CO_2 laser

The reported experiment is the first step in the development of the high-brightness LSS at the BNL ATF. The obtained agreement between the theory and the experiment allows straightforward extrapolation towards the next stage of the Thomson scattering experiment that will utilize the same interaction cell and the electron beam but the thousand times more powerful 1 TW CO_2 laser of the 30 ps pulse duration. Upon completion of the ongoing CO_2 laser upgrade and already demonstrated electron bunch compression to 300 fs, the ATF LSS will produce the x-ray flux of 10^{22} photon/sec. The diagram shown in **Figure 3** illustrates a unique strategic position of the ATF LSS among other existing and prospective laser driven x-ray sources.

The next stage of the ATF LSS experiment will also open an opportunity for in-depth study of Thomson harmonics generated on relativistic electron beams. With a 1 TW CO_2 laser beam focused into the s_L=32 mm spot the normalized laser strength of a=1 will be attained. Then the nonlinear Thomson scattering effect comes to scene. Apart from basic scientific interest, the expected nonlinear Thomson scattering may be used for expansion of the x-ray spectrum to benefit potential applications. ■

List of principle participants: I. Ben-Zvi, I. Pogorelsky, K. Kusche, P. Siddons, J. Skaritka, V. Yakimenko, X.J. Wang, (Brookhaven National Laboratory, USA), T. Hirose (Tokyo Metropolitan University, Japan), S. Kashiwagi, M. Washio (Waseda University, Japan), A. Tsunemi (Sumitomo Heavy Industries, Ltd., Japan), T. Omori, J. Urakawa (KEK, Japan), D. Cline, Y. Liu, P. He (UCLA, USA).

The result of the Accelerator Test Facility Thomson scattering experiment has been reported at the symposium "New Visions in Laser – Beam Interactions," Tokyo Metropolitan University, held on October 11-17, 1999, and will be published in Nuclear Instrum. & Methods in Sci. Res. A.

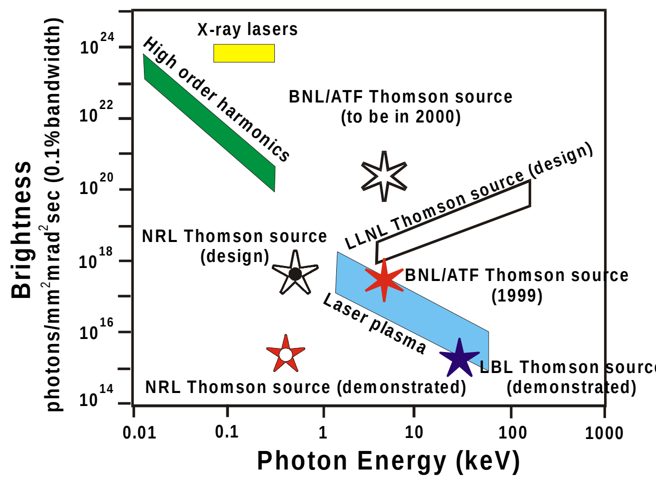


Figure 3:** Peak brightness of existing and projected laser-driven x-ray sources of the picosecond and femtosecond pulse **length.

Dave Cox Symposium

Steven Shapiro
BNL Physics

A symposium and banquet were held on Dec. 17, 1999 to honor Dave Cox as he retires after 37 years of research at BNL. The symposium was attended by about 50 participants who came from the US and abroad to pay tribute to Dave for his many contributions to the crystallographic community and to Brookhaven. The symposium started with a talk by Chalmers Frazer, a retired BNL and DOE employee. He reviewed the early years of Dave's career doing neutron diffraction at Westinghouse first, and then at Brookhaven where he performed experiments at the graphite research reactor and at the newly built High Flux Beam Reactor. Larry Finger, recently retired from the Carnegie Institute, then gave a history of Dave's successful efforts to construct a high-resolution x-ray powder diffraction beamline at the NSLS, which was the first of its kind using synchrotron radiation. After a brief recess, the afternoon continued with talks by some of Dave's recent collaborators who described highlights of Dave's scientific accomplishments with the X-7A beamline. Tony Cheetham of University of California at Santa Barbara spoke about their work, using both neutrons and x-rays, to understand the structure property relationship of the technologically important zeolites. The symposium closed with Art Sleight, Oregon State University, describing some of Dave's latest significant work on studying the mixed valency in superconducting and magnetic oxides. The evening concluded with cocktails and dinner at Berkner hall where gifts and tokens of appreciation were presented to Dave and his wife, Martine. A number of Dave's friends and colleagues used the convivial atmosphere to expound upon Dave's many extracurricular activities!

Dave's contributions to science will endure. It is worthwhile to note that Dave ranks Number 332 in the list of "1000 Most Cited Physicist, 1981-1997" with nearly 4000 citations to his work and an average of 45 citations per article. It is very fitting he has recently been honored with the Brookhaven Science and Technology Award for his prolific contributions to Brookhaven science. His intelligence, wisdom and judgment will be missed. We all wish him a long and healthy retirement. ■

Dave and his wife, Martine, enjoy some of the extemporaneous remarks by his 'friends' after the dinner at his symposium.

Safety & Compliance

Bob Casey
Associate Chair for ESH

ISM Validation

This spring is a time of considerable activity in the ESH arena at NSLS and BNL. NSLS users should be aware of what is going on and be prepared for potential involvement. The biggest event is the Integrated Safety Management (ISM) audit that is scheduled for the first two weeks of May 2000. This assessment will be conducted by the DOE and will examine the ESH programs at NSLS and the rest of BNL to determine if the processes and principles of ISM have been fully addressed.

I am sure that all users from the DOE Community are aware of ISM since it is a program commitment that extends throughout DOE. ISM is intended to ensure that the research program is conducted safely through the systematic integration of ESH into the ongoing work. For our users, the principal manifestation of ISM comes from our experimental safety review program and its associated controls and training. Before any research is approved for beam time, the Safety Approval Form (SAF) must be completed and submitted by the users. The principal reviewer of the SAF at NSLS is Andrew Ackerman, but on occasion Tom Dickinson or John Aloi may be the reviewer. Once in a while for very unusual work, the NSLS ESH Committee will review a SAF.

This review is very important in ensuring that the research can be safely performed at the NSLS. Procedures may be recommended and engineered or other administrative safeguards may be required. Most of the research at NSLS can be conducted readily without complicated safeguards. Following approval of the SAF, and authorization of the work to begin, it is extremely important that the users conduct the work consistent with the program described in the SAF and with any controls established during the review. Finally, we ask the users for feedback on the safety review process to determine if it is operating effectively from their perspective.

There is nothing complicated about ISM -- our programs have incorporated its processes for a long time. The biggest change in recent years is the need for increasing rigor and formality in the conduct of the program. During the two-week period in May, the DOE auditors will review in depth the NSLS Experimental Safety Review process described above -- it is likely that the team will want to interview users as well. We will keep you posted.

ESH Training

We are planning a number of changes during the first half of the year in the NSLS ESH Orientation required for all users. We are shifting from the required reading and ESH video to a program delivered entirely by computer. This change should greatly improve the effectiveness and efficiency of the training program and shorten the time required to complete the training.

It is important that users comply with all training requirements which permit access to the experimental floor (ESH Orientation, BLOSA) and which provide safe utilization of equipment (e.g., laser, overhead cranes, etc.). No exceptions are permitted.

There is an important distinction in training requirements that must be observed for short-term users and those who are at the NSLS for long periods of time. A user is considered short-term if he/she is at the NSLS for 60 days or less during the year. The NSLS ESH Orientation is designed for the short-term user and incorporates elements of required BNL training for radiation safety, Environmental Management, Emergency Response, and Stop Work. Long term users are expected to receive the regular BNL training in each of these topics.

Environmental Management

Another area of considerable visibility at the NSLS and BNL is the incorporation of ISO 14001 criteria for environmental management into the ESH program. Self-certification of compliance with the ISO 14001 criteria is a major BNL goal for this fiscal year. Compliance with these criteria will not require major changes in the current program, but it does result in a systematic examination at the NSLS to ensure that all environmental aspects of our work are understood and that training and procedures as needed are in place to prevent those impacts.

The most important environmental aspect of the research program conducted at NSLS is the generation of hazardous wastes. Although the quantities of waste are typically small, the Laboratory is committed to strict compliance with federal or local requirements established for these wastes. BLOSA training should emphasize these requirements, and all beamlines that generate a hazardous waste must have personnel who have received the BNL hazardous waste training program. ■

Weekly Users' Meetings

X-Ray Users: Wednesdays,
11:30 a.m., Seminar Room

VUV Users: Thursdays,
11:30 a.m., Conference Room A

To subscribe to the X-Ray Users and/or VUV Users List Server and receive the minutes each week: Send an e-mail to listserv@bnl.gov. Leave the subject blank and place the following text in the body of the message: **subscribe nsls-vuv-minutes-l@bnl.gov, Your Name or subscribe nsls-xray-minutes-l@bnl.gov, Your Name** (that's "-L", not the number 1)

User Administration

Mary Anne Corwin
NSLS User Administrator

Safety Approval Form System

In January, an updated version of the Safety Approval Form (SAF) went into production. New global searches allow researchers to locate existing forms more easily and to sort records in any number of ways. Beamline staff have been granted "read-only" access to view forms submitted so they can ensure that the researchers have submitted sufficient information concerning the experiments to be performed on their beamlines and that experimenters have appropriate training. Researchers also now have the capability of creating a new form based on an existing one.

Once the SAF has been approved, the submitter will receive an email indicating the approval date and any NSLS comments concerning the experiment.

Useful SAF navigating tips:

1. *Red hyperlinked row numbers*. Searches in the SAF system result, generally, in more than one record. For example, there may be more than one experimenter on a SAF. For each record, you will see a red hyperlinked row number to the left of the record. Selecting this number will allow you to access the record for the experimenter you wish to view.

2. *List View button*. Most forms connected with the SAF have a List View button, which allows you to view a list of records for a particular form. For example, if you wish to view a list of all materials previously submitted for the SAF, press "List View." All materials will be listed, row by row. Here again, you will see red hyperlinked numbers to allow you to access information about each material.

3. *Requery*. Most webforms are attached to databases, as is the SAF. When attempting to retrieve data from the database, you may find it necessary on occasion to "refresh" or "requery" the database. You may otherwise, receive an error. With the SAF system, this occurs when you have accessed one record, updated information, and attempt to access a second record on the same form (experimenters, for example). To avoid the error, simply press the "requery" button before retrieving the next record in the list.

R E M I N D E R S . . .

All Foreign Nationals

All foreign nationals must file Form IA-473. Approvals are valid for two years. Go to the NSLS home page, then User Administration for a link to Forms.

Foreign nationals who have never registered or who require training or retraining must arrive at NSLS User Administration on weekdays (no weekends or holidays) prior to 3:00 p.m. so we may verify the submission of Form IA-473. Any foreign national arriving at any other time must have a valid, active appointment with the NSLS and a valid BNL photo identification card or will be requested to leave the NSLS to return on the next normal workday. See NSLS Policies and Procedures Section 2.3.6 at URL: **http://www.nsls.bnl.gov/PandP/pnp-ch2.htm**

Users Must Pre-Register Before Each Visit

All NSLS users must pre-register with NSLS User Administration prior to each visit so that BNL Gate Security is notified of your arrival date. To access the online preregistration form, go to URL: **http://nslsweb.nsls.bnl.gov/nsls/dbforms/user-regis.asp** ■

Q & A on ISM

Question #1: Who is responsible for ensuring that the research programs are conducted safely and in compliance with ESH requirements?

Answer: *Safety is a line responsibility at the NSLS. As such, the NSLS Chair is ultimately responsible to ensure safe operation. That responsibility flows from the Chair through the line to the individual users and workers on the experimental floor. For each experiment, the principal investigator or beamline spokesperson is responsible to ensure that the research is conducted in compliance with the requirements established during the Experimental Safety Review. ESH staff is available to provide support and to provide oversight of the program.*

Question #2: What process is used to ensure that research is conducted safely at NSLS?

Answer: *The Safety Approval Form and its review and authorization process is designed to ensure that the research and its hazards are described and identified, and that controls are established to minimize any associated risk.*

Question #3: Who authorizes an experiment to receive beam at the NSLS?

Answer: *The Experimental Review Coordinator has been authorized by the NSLS Chair to approve an experiment to receive beam. Following approval by the ERC, the NSLS Operations Coordinator can enable a beam if all required conditions are in place.*

Question #4: What is the most significant environmental aspect of the research at the NSLS?

Answer: *Generation of hazardous waste*

X-Ray Ring Status

Rich Biscardi
X-Ray Ring Manager

Boris Podobedov has recently joined the NSLS staff in the position of the X-ray Ring Studies Manager. James Safranek previously held this position. Boris just received his Ph.D. from Stanford University where he did his thesis research on beam dynamics at the SLAC damping rings. Boris' main project at the NSLS will be upgrading the X-ray ring digital feedback system. In addition, he is now responsible for scheduling studies time for the X-ray ring. Boris can be reached at his office, Room 2-137, by phone at 344-3701, or by e-mail, borisp@bnl.gov.

Progress has been made toward reducing the emittance at 2.8 GeV. The defocusing sextupole power supply arrived at the NSLS after a functional inspection at the manufacturer's facility. The operational location was prepared, and it was put in place immediately upon arrival. The mechanical group has completed the work necessary to connect the magnet coils to the power supply. This system will be commissioned during studies periods during the winter. When commissioning is complete, all standard operations will be at 2.8 GeV.

For higher current operation, the rf and mechanical groups have been preparing a second all-copper cavity. Upon its arrival at BNL in early fall 1999, the cavity was configured for various acceptance and power testing. The cavity was driven to greater than 70 kilowatts for approximately 8 hrs and at 50 kW for several days to condition the inner surfaces. The cavity was installed during the December shutdown. As a result, standard operations will now be 275 mA at 2.8 GeV and 400 mA at 2.584 GeV.

The installation of the new X-17 wiggler had been scheduled for the December '99 shutdown, but was postponed due to a problem uncovered during acceptance tests. The final stages of the acceptance tests called for powering the magnets to each of the three operating mode conditions, and then performing integrated magnetic measurements during ramping. During this period, the new wiggler power supplies purchased from Danfysik and the computer control interface were also to be tested. After correcting some problems associated with integrating the power supplies and control system, the wiggler failed with the magnets powered to about 90% of full field. Diagnosis of the problem revealed an internal high resistance on the main power lead. It is suspected that the joint between the superconducting coil and the main lead has failed, but the full nature of the problem will not be known until the cryostat is disassembled.

The wiggler has been shipped to Oxford. Return of the wiggler to the NSLS is expected in early spring of 2000. At that point, additional acceptance tests will be performed to validate the integrity of the repairs as well as to test the control system, establish magnet power ramping rates and to quantify integrated magnetic field errors during power up and steady state. We expect that all testing can be completed and the wiggler installed in the X-17 straight section during the next major winter shutdown. ■

VUV Ring Status

Stephen Kramer
VUV Ring Manager

The ring came back from the winter shutdown on schedule and with most of the work completed. The upgraded radiation shielding has now been completed around the entire ring. There are still a few holes in the shielding that will be completed during future maintenance periods, but all of the vertical walls have been enhanced in their radiation absorption power. This improvement in the shielding will allow studies to begin on the safety issues related to implementing Top-Off Injection into the ring. A Task Force has been setup to handle upgrading the Safety Analysis Document for the ring to see if injection can take place into the VUV ring with the safety shutters open, a necessary condition for successful Top-Off Injecting into the ring.

New water-cooled mirrors were installed in the U5 (beam profile monitor) and the U3B (diagnostic) beamlines. The U5 beamline used to show an apparent motion of the focused beam spot of about 1.3 mm over the operating current range. The new mirror shows less than 100 microns peak to peak motion with 30 microns of this motion coming from real motion of the electron beam with beam current. The U3B mirror will be part of the diagnostic beamline that will study energy changes of the electron beam in the ring. Some of these changes occur from instabilities of the beam due to self-induced voltages that change the energy distribution of the beam.

Other work performed on the ring during this shutdown included:

1) leak checking the injection region and sealing a small leak found at an ion pump flange,
2) replacing a bad ADC board in the orbit micro,
3) replacing a stepper motor in the scraper and control boards in the TOK undulator gap control,
4) replacing unions in the dipole bus water cooling circuit, and
5) replacing the front end vacuum valve on the U12 beamline. ■

Novel Combination of Fluorescence Microscopy with Infrared Micro-spectroscopy

Lisa Miller (BNL-NSLS) and Tom Tague (Spectra Tech, Inc.)

Infrared micro-spectroscopy (IRMS) is a widely used and valuable technique for identifying the chemical makeup of small particles. By taking advantage of the high brightness of a synchrotron infrared source, particles that are too tiny to be analyzed with a conventional thermal (globar) source can now be examined in detail.[1,2] With the advent of the synchrotron infrared microscope, new applications of IRMS are just beginning to be realized.[3-5] Frequently, sample visualization is benefited by the use of fluorescence illumination. Fluorescent compounds absorb light and then emit light at wavelengths longer than the absorbed wavelength. Primary (natural) fluorescence is known to occur in plant cell walls, wool, as well as many pharmaceutical products. Secondary fluorescence is the use of fluorescent dyes or fluorochromes to illuminate samples which do not exhibit native fluorescence. The dyes typically are bound to the compounds of interest. Fluorescence is particularly useful for visualizing samples against a dark background and can have a detectivity of 50 molecules per square micron. By the 1950's, the use of secondary fluorescence had made fluorescence microscopy a common analytical tool.

To date, combining fluorescence microscopy and IRMS has required analyses with two separate microscopes. This procedure required the user to somehow mark the fluorescent regions of interest (often with a photograph) before transferring the sample to the infrared microscope. The optical design (finite tube length) of earlier infrared microscopes did not allow the ready insertion of accessory optics, such as optical components for fluorescence microscopy. For the first time, an infrared microscope has been modified such that fluorescence sample visualization and infrared micro-spectroscopic analysis can be performed simultaneously. This novel infrared microscope, located at Beamline U10B, has applicability to many fields of investigation, such as biophysics, cell biology, medicine, forensics, microelectronics, and geology.

Biomedical studies

It is becoming increasing clear that IRMS is a powerful tool for determining the chemical composition of biological and biomedical samples. This is largely due to the dramatic improvement in spatial resolution attainable using a synchrotron infrared source. The high brightness of the synchrotron source permits high quality data collection at the diffraction limit, e.g. 3-25 μm spatial resolution in the mid-infrared region (4000 – 400 cm^{-1}). In contrast, fluorescence illumination has been a commonly used technique in biology for many years. Immunofluorescence, where fluorochrome labels are attached to specific antibodies, has become a widespread and powerful technique in cell biology for visualizing targeted antigens.

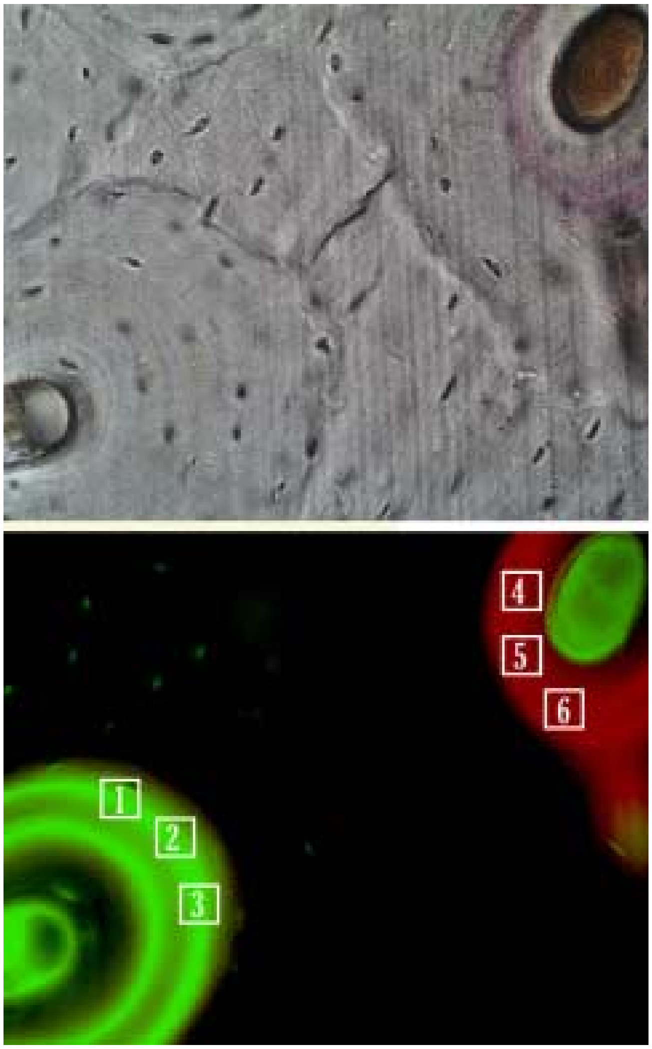
One important use of fluorochrome labels has been in the study of osteoporosis. Throughout a lifetime, bone is remodeled, i.e., old bone is eroded away and new bone is deposited. Although it is clear that osteoporosis is associated with a reduction in bone mass and a fragile skeleton, it is not understood whether the chemical composition of bone remodeled after the onset of osteoporosis is different from normal bone. Changes in the bone chemistry associated with osteoporosis might reveal important information pertaining to the cause of osteoporosis and how it might be treated.

Female monkeys *(Macaca fascicularis)* that have had their ovaries removed often develop osteoporosis, similar to postmenopausal women. In studies by Jerome and coworkers, a colony of these monkeys were administered fluorochrome labels at one and two years after ovariectomy (Ovx) or Sham ovariectomy (Intact).[6] These labels were taken up into newly remodeled bone (**Figure 1**). Green (calcein fluorochrome) and orange (alizarin complexone fluorochrome) fluorescence represent new bone deposited one and two years after surgery, respectively. By combining fluorescence microscopy and IRMS, the chemical composition of newly remodeled bone from Intact versus Ovx monkeys has been compared.[7,8] **Figure 1** shows infrared spectra of bone remodeled 1 year (spectra 1-3) and 2 years (spectra 4-6) after ovariectomy. Infrared absorption bands present at 500-650 and 900-1200 cm^{-1} ($-PO_4^{-3}$) and 850-880 cm^{-1} ($-CO_3^{-2}$) indicate the presence of mineral in the bone. Peaks at 1660 and 1550 cm^{-1} reveal Amide I and Amide II absorption bands, respectively, indicating the presence of protein (primarily collagen) in the bone. There are no infrared absorption bands present in the spectrum resulting from the fluorescent dyes. Secondary fluorescing dyes are typically utilized at concentrations dilute enough to be undetectable in the infrared region.

Comparison of the fluorochrome-labeled bone in Intact versus Ovx monkeys indicates that bone from monkeys with osteoporosis can be characterized as having abnormal collagen structure and reduced rates of mineralization. Coupled with factors such as trabecular architecture and bone shape and size, these ultrastructural factors may play a contributing role in the increased bone fragility in osteoporosis.

Microelectronics

The microelectronics industry continues to be focused on miniaturization of integrated components on silicon wafers. For the integration of microcomponents to be effective, the wafer must be clean to the parts per billion level.[9] After component integration takes place, individual components can suffer damage when conducting surface contaminants are present. Typically, quality control is per-



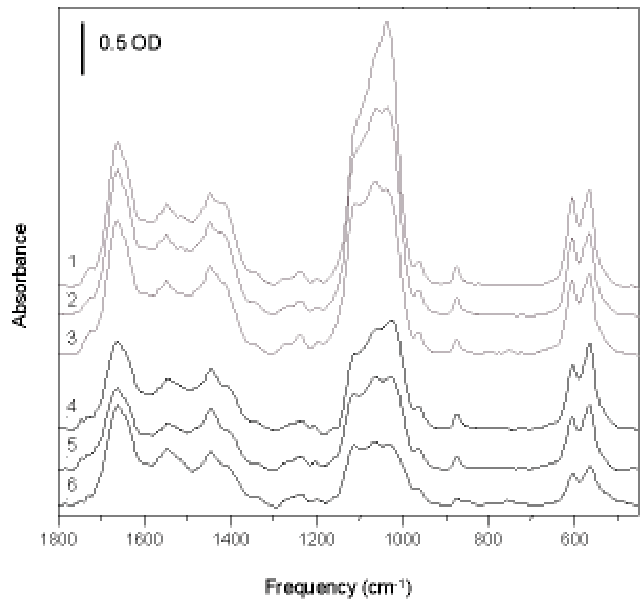


Figure 1: *Optical images of a region of cortical bone from an Ovx monkey tibia under (top left) normal transmission illumination and (bottom left) ultraviolet fluorescence illumination. The bone sample is embedded in poly- methyl methacrylate and microtomed to a 5 μm thickness. (Bottom right) Individual IR spectra were collected in the calcein-labeled region of bone (spectra 1-3) and alizarin complexone-labeled region of bone (spectra 4-6). For each of these spectra, 128 scans were collected in transmission mode at 4 cm^{-1} resolution using a 6 x 6 μm square aperture and a Cu-doped Ge detector.*

formed at many steps in the manufacturing process to ensure that the product is free of contaminants. When contaminants are found, it is vital that chemical characterization of the particles takes place to determine the source of contamination.

Currently, there are no satisfactory techniques for performing quality control on semiconductor wafers and circuit boards. Typically, an optical microscope is used to scan for contaminant particles. This can be a tedious process and is particularly difficult when investigating integrated circuits. Under normal illumination, it is difficult to discern the presence of dust and other small contaminant particles within an integrated circuit (**Figure 2**). In contrast, fluorescence illumination with ultra-violet light cloaks the non-fluorescing components, making it easy to identify contaminant particles. Once identified, an aperture is set to mask a specific particle and IRMS is used to characterize the chemical makeup of that contaminant.[10,11] Infrared analysis of one of the contaminant particles in **Figure 2** reveals the presence of low molecular weight polystyrene (**Figure 2a**). **Figure 2b** shows a reference spectrum of polystyrene (molecular weight 800) and 2c shows the result of mathematically subtracting the polystyrene spectrum from the raw spectrum. Finally, **Figure 2d** shows a reference spectrum of an alkyd urea resin, which closely matches the subtraction result found in **Figure 2c**. Thus, the identification of the these contaminants allows for precise identification of their origins in the manufacturing process.

Beamline U10B

Beginning in May 2000, Beamline U10B will be available for General Users. The beamline will be equipped with a Nicolet Magna 860 step-scan FTIR and Spectra Tech Continuμm infrared microscope (**Figure 3**). Beamsplitters and detectors are available for the mid- and far-infrared regions. For fluorescence microscopy, the excitation source is a mercury arc lamp, where wavelengths are selected by utilizing Olympus fluorescence cubes. In addition to fluorescence imaging capabilities, sample viewing can also be enhanced with visible light polarizers and differential interference contrast (DIC).

The Continuμm infrared microscope is the first commercial system that has the capability of simultaneous sample viewing and infrared data collection. This feature is especially important for the high-resolution applications that require a synchrotron source, where accurate sample masking is critical. Moreover, this feature saves time for the user by eliminating the need to switch from “view mode” to “infrared mode” for data collection. An automated mapping stage is also a time-saving feature of this system. Software control of the sample position, aperture settings, and mapping simplify the user interface. This ability to rapidly visualize and chemically characterize minute samples provides a new and important capability for a wide range of applications. For more information on Beamline U10B, please contact Lisa Miller at lmiller@bnl.gov. For color images of **Figures 1-3**, refer to the NSLS Newsletter online at www.nsls.bnl.gov. ■

Figure 3: *Gywn Williams (NSLS) at the helm of the new Spectra Tech Continuμm infrared microscope with fluorescence microscopy capabilities.*

References

[1] G.L. Carr, J.A. Reffner, and G.P. Williams, *Rev. Sci. Instr.,* **66**, 1490-1492, (1995).

[2] J.A. Reffner, P.A. Martoglio, and G.P. Williams, *Rev. Sci. Instr.*, **66**, 1298, (1995).

[3] N. Jamin, P. Dumas, J. Moncuit, W.H. Fridman, J.L. Teillaud, G.L. Carr, and G.P. Williams, *Proc Natl Acad Sci USA*, **95**, 4837-40, (1998).

[4] L.M. Miller, C.S. Carlson, G.L. Carr, and M.R. Chance, *Cell. and Mol. Biol.,* **44**, 117-127, (1998).

[5] D.L. Wetzel, S.M. LeVine, *Science,* **285**, 1224-1225, (1999).

[6] C.P. Jerome, R.A. Power, I.O. Obasanjo, T.C. Register, M. Guidry, C.S. Carlson, and D.S. Weaver, *Bone,* 20, 355-364, (1999).

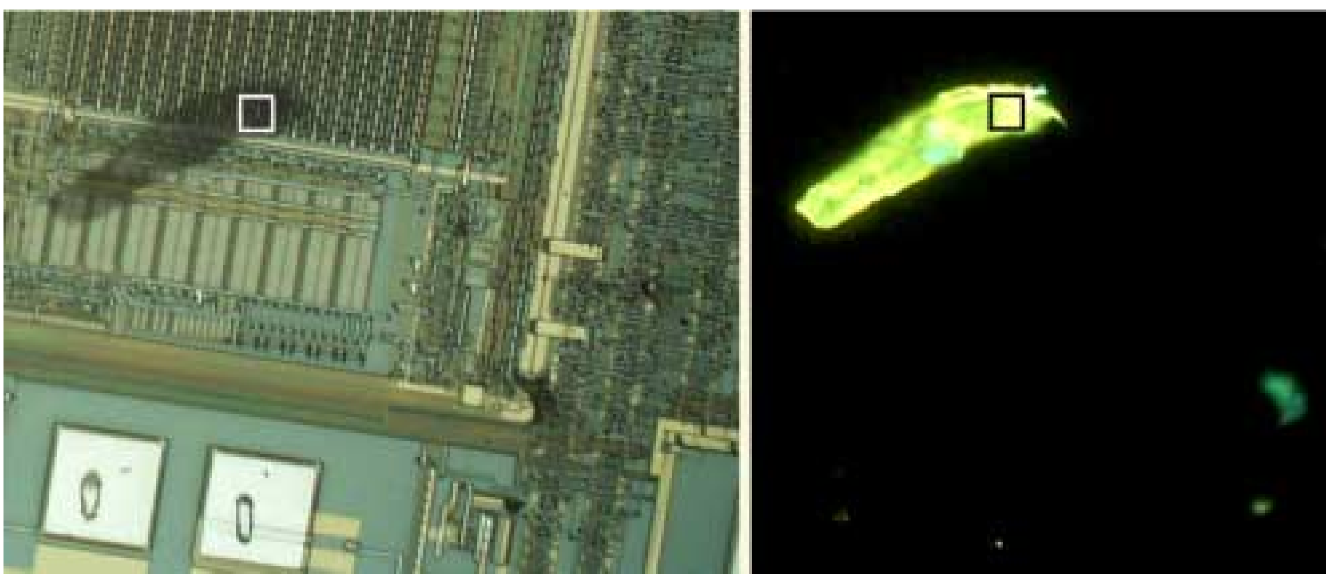
[7] L.M. Miller, R. Huang, M.R. Chance, and C.S. Carlson, *Synch. Rad. News,* **12**, 21-27, (1999).

[8] L.M. Miller, J. Tibrewala, and C.S. Carlson, *Cell. and Mol. Biol.* (2000), *in press*.

[9] E. Fogarassy, A. Slauoi, C. Fuchs, and J.L. Regolini, *Appl. Phys. Lett.,* **51**, 337, (1987).

[10] O.M.R. Chyan, J. Wu, and J.J. Chen, *Appl. Spec.,* **51**, 1905-1909, (1997).

[11] O.M.R. Chyan, J.J. Chen, F. Xu, and J. Wu, *Anal. Chem.* **69**, 2434-2437, (1997).



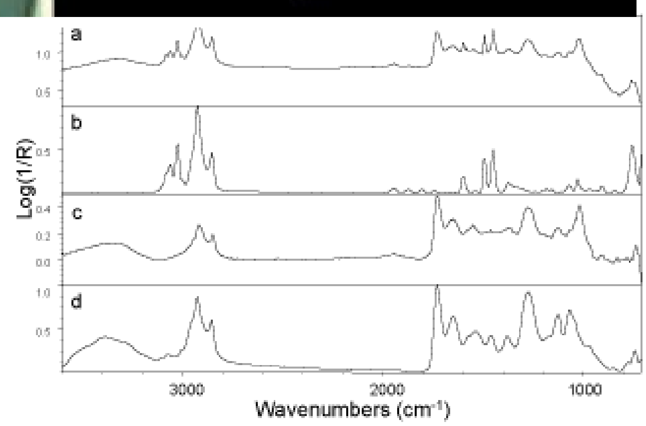


Figure 2: *Optical images of an integrated wafer under (top left) normal reflection illumination, and (top right) ultraviolet fluorescence illumination. (Bottom right) Infrared spectra collected from* ***(a)*** *the highlighted particle on the integrated wafer, and* ***(b)*** *an 800 MW polystyrene from the Hummel Polymer Reference Library (Nicolet Instrument Corp.).* ***(c)*** *Infrared spectrum resulting from the subtraction of the polystyrene reference spectrum from the raw spectrum.* ***(d)*** *Infrared spectrum of an alkyd urea resin, also from the Hummel Polymer Library. For each of the IR spectrum of the contaminant, 128 scans were collected in reflection mode at 4 cm^{-1} resolution using a 10 x10 mm square aperture and an MCT-A detector.*

FOCUS ON . . NSLS Operations Coordinators

Richard Heese

The NSLS Operations Coordinators are an integral part of the Operations Group. On the X-Ray and VUV experimental floors, they are the major interface between more than 2,000 annual users and the NSLS "organization." The NSLS can be a bewildering place for novice users and users who are not here often, and the Operations Coordinators with their far reaching knowledge of operating and safety procedures are a gold mine of information and can normally help and answer questions about procedures at NSLS. The wide variety of experiments in almost all scientific fields that are conducted at the NSLS imply a wide variety of different types of beam line configurations and

NSLS Operations Coordinators. Top row (left to right): John Klug, Tom McDonald, Kathy Warburton, Ray Raynis, Lenny Pharr. Bottom row (left to right): Peter Ratzke, Billy Jew and Randy Church (Control Room Supervisor). Missing from photo: Bob Chmiel and Steve Kemp.

users from many different backgrounds. The Operations Coordinators are familiar with the operating procedures, personnel and machine safety interlock systems and other special conditions of all of the almost 90 beam lines.

Before beginning their experiments, a user or group of users need approval of the Operations Coordinator in order to enable their beam line safety shutter. Enabling the safety shutter allows access to the synchrotron radiation. Before an Operations Coordinator enables a beam line safety shutter, certain events must take place: All conditions of the safety approval forms must be met, beamline safety checklists and padlock checklists are satisfied, all users are wearing radiation badges and have received all appropriate training, the beam line information green board is updated, all yellow tags restricting beamline operations are removed and a beamline lockout responsibility form has been signed by a user present to ensure proper lockout procedures at the end of the experiment. All this sounds like police work, and that there must be constant friction between the experimenters and the Operations Coordinators. However, quite the opposite is true. The Operations Coordinators are committed to make the stay of an experimental user at the NSLS a pleasant one, and allow access to the synchrotron radiation in the most efficient and safe manner. They are always ready to help in any way they can.

There are 8 Operations Coordinators at the NSLS, working in four shifts of two. The NSLS operates 24 hours a day and 7 days a week except for the Fourth of July, Thanksgiving, Christmas and New Years. Two Operations Coordinators are always on duty during operations. The accelerators are shut down for the winter maintenance period, which include Thanksgiving, Christmas and New Years, and for a spring maintenance period of one to two weeks. During these long shutdown periods, an Operations Coordinator is still available to provide service for the users from 0830 to 1700 hrs.

In addition, the Operations Coordinators are an important part of the NSLS emergency response effort. For example, activation of a single automatic fire alarm first rings in the control room (and also notifies the BNL Fire Department), allowing the Operations Coordinators to investigate the nature of an alarm, and at their discretion cancel the next level of response, which is a building evacuation. During a fire, they guide the firemen to the emergency site and help with the evacuation procedures.

The Operations Coordinators are well versed in all aspects of safety, safety management and emergency response, and in addition to being a valuable source of information for the users, are on constant vigil, so that a user's stay at the NSLS is a safe and productive one.

About Radiation Badges . . .

Wear your Radiation Badge as shown at the left. . .

which means it must be . . .

- fully visible (not covered)
- between your neck and belt
- with the yellow or blue color bar facing out

Always wear your radiation badge when working in a Controlled Area.

For Temporary Radiation Badge users: If you are working at the NSLS over a badge exchange weekend, go to User Admin. or the NSLS Control Room to return your old badge and sign out a new one. When your experiment is finished and before you leave, always put your radiation badge in any "Returned Badge" box located near the badge boards.

CALL FOR GENERAL USER PROPOSALS

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

Wednesday, May 31, 2000 for scheduling September - December 2000

Prior to Submitting a Proposal

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

Preparing Your Proposal

The same form is used for both new proposals and beam time requests against existing proposals. Follow the instructions on the information sheet and complete and submit all the required sections. Type or print all information legibly. MAIL OR FAX **ONE COPY** of the proposal form and any attachments to the NSLS User Administration Office. Only **one copy** is required - *do not mail a hard copy if you have already faxed one.*

Macromolecular Crystallography (PX) Requirements

NEW PROPOSALS: The proposal represents a two-year program. Provide an overall plan for your research according to the instructions on the proposal form. If you can, estimate the number of crystals you plan to measure over the two years. If you require the use of an insertion device beamline like X25, be sure to indicate your need for the enhanced performance. New proposals must also include your plans for the upcoming cycle for which you are requesting time (below).

BEAM TIME REQUESTS: Be specific about what you plan to study in the upcoming cycle. Submit PX Forms only for the crystals you plan to study in that cycle. Answer all the questions, use the back of the form if you need more space. Be clear about what crystals you already have, which you expect to have, and how you would use the beam time you requested if you were unable to obtain the planned crystals in time (i.e., other crystals described in your program).

*The **NSLS Newsletter** is printed on paper containing at least 25 percent recycled materials, with 10 percent post-consumer waste.*

Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Standard Time Wednesday, May 31 in order to be considered for the September - December cycle. **The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on May 31**. We encourage submitting new proposals by mail or fax prior to the deadline. Beam time requests for active proposals will be accepted after the deadline, but will be allocated beam time only after requests received on time have been allocated. Late requests are not eligible for a rating upgrade if beam time could not be allocated to them.

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

Proposal Forms and Additional Information

Blank proposal forms and instructions are available on the World Wide Web. From the home page at http://www.nsls.bnl.gov, go to User Information, then Forms. **The PX form must now be completed online**. A guide to the NSLS beamlines and more information about the General User Program can be found through our homepage, **http://www.nsls.bnl.gov** or by contacting E. Pinkston or L. Rogers in the NSLS User Administration Office. Office hours are Monday through Friday, 8:00 a.m. to 5:00 p.m. Eastern Standard Time (EST). Contact information is on the back page of this Newsletter.

Safety Approval Form

Reminder. Principal Investigators must submit a Safety Approval Form (SAF) online ***at least one week before*** the scheduled beam time. You must answer all questions on the Safety Approval Form and you must include all experimenters who will be performing work at the NSLS with respect to your experiment. The online Safety Approval Form can be found at:

http://130.199.76.52/safety/

Do not send in SAFs with your proposal.

Registration Form ◆ 2000 Annual Users' Meeting & Workshops

Complete a separate registration form for each registrant. Return with payment to User Admin. (address and fax on back cover). INFO: www.nsls.bnl.gov or e-mail lsusrmtg@bnl.gov

Annual Users' Meeting on Tuesday, May 23, 2000

The Meeting Registration fees include attendance at meeting sessions, meeting materials, vendor exhibits, poster sessions, welcoming reception on 5/22, barbeque, and coffee breaks. (Banquet optional.)

Social Events

Welcoming reception with open bar and live music, featuring a vendor exhibit and poster session at Berkner Hall on Monday, May 22. Users' Meeting Banquet, Tuesday May 23 will be held at Giorgio's Restaurant at The Fox Hill Country Club. Guests of meeting participants are always welcome. The non-refundable ticket price for meeting registrants and guests is $45. Bus transportation between BNL and Country Club will be provided with $10 prepaid advance reservation.

Users' Meeting Cancellation Policy

If you need to cancel your registration, please notify the Conference Coordinator in writing by April 28 in order to receive a refund. If your cancellation notification is received after that date, the registration fee will not be refunded.

Free Transportation to/from LIRR Train Station

BNL Chauffer service is free, but advance reservation is required. **For daily chauffer schedule and reservations**:

8:57 a.m. pickup from Ronkonkoma Train Station to BNL
(Train leaves NYC Penn Station daily at 7:39 a.m.)
☐ Pick me up from Ronkonkoma Train Station on:
Date: ____________ Number of People ______

4:00 p.m. from BNL to Ronkonkoma Train Station
(The 4:46 train arrives at Penn Station at 6:08 p.m.)
☐ Take me to Ronkonkoma Train Station on:
Date: ____________ Number of People ______

Housing Information

BNL onsite housing is a first-come first-served basis. Charges must be paid to the Housing Receptionist on arrival. *Residence Houses* have single rooms and shared baths. *Apartments* have 2 twin beds per bedroom and shared baths. 1-rm efficiencies have 1 queen bed. Housing is very limited and choices of accommodation may not be available. *Make reservations early!* Alternate assignments will only be made with the approval of the Conference Coordinator. *If a reservation is not canceled by 1:00 p.m. on the date of arrival, you will be billed for that night's lodging.* Check out time is 3:00 p.m.

Housing Reservations: Pay Upon Arrival

Arrival Date: ___________ Departure Date: ___________

☐ Residence: Male [ ] or Female [ ]. $18 per night.
☐ 1-BR APT ☐ 2-BR APT ☐ Efficiency
to be shared with: ____________________
Apartment/Efficiency rates: $64 for 1 person per bedroom per night or $70 for 2 persons per bedroom per night.

Full name: ________________________________
Affiliation: ________________________________
Mailing Address: ________________________________
____
Phone: ________________ Fax: ____________
Email: ________________________________

Part I: Meeting Fees

Registration Fee (check one box):

☐	Registration, General	1	x	$150	=	______
☐	Registration, Student	1	x	$70	=	______
☐	Late Fee (after 4/30)		add	$25	=	______

Workshops (check all workshops you wish to attend):

☐ 5/22:Environmental & Geological Sciences
☐ 5/22 :XAFS of Dilute Systems
☐ 5/24:Very Bright Infrared Sources and Applications
☐ 5/24:New Approaches to Solving Protein Crystal Structures
☐ 5/24:Chemical Applications of Synchrotron Radiation

Workshop Fees (check one box):

☐	1 workshop, General	1	x	$70	=	______
☐	2+ workshops, General	1	x	$100	=	______
☐	1 workshop, Student	1	x	$40	=	______
☐	2+ workshops, Student	1	x	$60	=	______

Total Part I Fees (allowable expenses) $ ☐

Part II: Social Events

[The following are unallowable costs and cannot be paid for with government credit cards or funds] **(check all that apply):**

☐	5/22 reception, registrant	1__	x	$0	=	______
☐	5/22 reception, guest(s)	___	x	$20	=	______
☐	5/23 lunch, registrant	1__	x	$0	=	______
☐	5/23 lunch, guest(s)	___	x	$15	=	______
☐	5/23 Banquet	___	x	$45	=	______
☐	5/23 Bus to Banquet	___	x	$10	=	______

Total Part II Fees (unallowable expenses) $ ☐

Payment Information

Part I and Part II expenses must be paid for separately. Part II expenses cannot be paid for with government funds or government credit cards. Fees may be paid as follows:

[ ]Checks or [ ]Money orders enclosed. Make separate checks for Part I and Part II expenses. Checks must be drawn on a U.S. bank, payable to "Brookhaven Science Associates."

[ ]BNL Account. Users with signature authority on a BNL account may charge Part I expenses. Overhead charges apply. I authorize $ to be charged to BNL Acct #

[ ]Credit Card: I authorize my credit card(s) to be charged:

Part I:
[ ]Visa [ ]MasterCard [ ]AmExpress $____________
Card No.________________________ Exp.___/___/___.

Part II:
[ ]Visa [ ]MasterCard [ ]AmExpress $____________
Card No.________________________ Exp.___/___/___.

Authorizing signature for BNL Acct or for Credit Cards:

____

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In This Issue

Important Upcoming Dates

Apr. 9-14, 2000	Rapid Data 2000 Course, "Rapid Synchrotron Macromolecular Data Collection"
April 15, 2000	Deadline for submissions, July Newsletter
April 28, 2000	Deadline to register for the NSLS 2000 Annual Users' Meeting
May 22-24, 2000	NSLS Annual Users' Meeting and Workshops
May 31, 2000	Deadline for General User Proposals (May-Aug. 2000)

The *NSLS Newsletter* is published triannually by the National Synchrotron Light Source Department, Brookhaven National Laboratory.

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For additional information about the NSLS (including this Newsletter in electronic format) see the NSLS Home Page on the World Wide Web at

www.nsls.bnl.gov

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