National Synchrotron Light Source ◆ Brookhaven National Laboratory

NSLS Newsletter

July 2001

Pseudomartensitic Phase Transformation

JiuHua Chen, Donald J. Weidner, John B. Parise, Michael T. Vaughan, and Paul Raterron

Center for High Pressure Research, Department of Geosciences, SUNY at Stony Brook

Phase transformations play an important role in the behavior of materials, as a material behaves very differently in different forms of phase. When graphite changes its crystal structure into a cubic structure, it transforms into diamond, greatly increasing its value even though both graphite and diamond consist of the same element, carbon. When iron-carbon steel is quenched from high temperature, the steel becomes much harder, increasing its industrial value. Many other phase transformations happen every day, changing the properties of a material to either increase or reduce its value. All the phase transformations have been classified into two groups, diffusional (e.g. graphite-diamond) and diffusionless (e.g. martensitic transition in iron-carbon steel), for decades. Diffusionless transformation, known as martensitic transformation, has been well documented for metallic materials[1]. However, studies of this type of phase transformation in oxides are less extensive. Both transformation mechanisms have been proposed for the olivine(α)-spinel(γ) phase transition, by theoretical modeling and transmission electron microscopy (TEM) studies[2, 3]. Using a newly developed high pressure time-resolved x-ray diffraction technique[4], we are able to follow the transformation crystallographically by a sequence of structure refinements. The experimental result shows that the α-γ transformation driven by temperature at high pressure is controlled by a third type of transformation mechanisms, pseudomartensitic transition: a diffusionless oxygen sublattice transition coupled with short-range diffusional cation reordering.

Our experiments were performed using the large anvil press SAM85[5] at the superconductor wiggler beamline X17B of the National Synchrotron Light Source (NSLS). We compressed the powdered Fe_2SiO_4 fayalite sample into the spinel stability field (6.9 GPa). Then we drove the α-γ transition by increasing the temperature at a constant rate (0.03 K/sec). Time-resolved x-ray diffraction patterns were recorded by a translating imaging plate[4] at a photon energy of 41.12 keV during the phase transformation.

Eight diffraction patterns (each integrated over 100 pixels on the imaging plate) of two coexisting phases were analyzed by Rietveld refinement using General Structure Analysis System (GSAS)[6]. **Figure 1** shows a typical result with 420 diffraction lines from two phases contributing to the pattern. The space group of the spinel phase is

Inside This Edition . . .

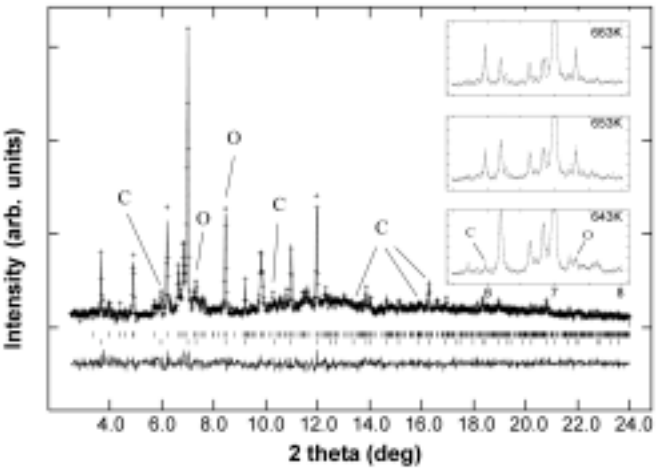


Figure 1. Structure refinement for two coexisting phases during the olivine-spinel phase transformation. The calculated and observed intensities are represented by a continuous line and crosses, respectively. A difference curve ($I_{obs} - I_{cal}$) is plotted at the bottom on the same scale. The olivine and spinel allowed reflection positions are indicated respectively by the first and second row of vertical bars. Letter C and O indicates the diffraction peaks of spinel which are mainly contributed from Fe^{2+}/Si^{4+} cations and oxygen sublattice, respectively. A near-unrecognizable increase in relative intensity of C to O with temperature is illustrated in the inset.

Fd3m; Z=8. For the standard origin at a center of symmetry, the three atoms in the asymmetric unit are Fe^{2+} at (1/2, 1/2, 1/2), Si at (1/8, 1/8, 1/8), and O at (*u, u, u*) with $u \approx 0.24$. During preliminary refinements, the occupancies of all sites in the spinel phase are allowed to vary. This yields a full occupancy (1.0 to 1.1) for the oxygen site while the occupancies of Fe^{2+} cations at octahedral sites and Si^{4+} at tetrahedral sites are significantly below 1. In the final refinements, the oxygen site is constrained at full occupancy while the occupancies of the cation Fe^{2+} and Si^{4+} together with lattice parameter, oxygen positional parameter, isotropic thermal parameters and phase fractions are refined. Diffraction peaks of the olivine phase are significantly broadened due to deviatoric stress developed during the cold compression; the intensities are not sufficiently precise to derive accurate crystallographic data for the olivine phase in the presence of this deviatoric stress. Results of the final refinements for the spinel phase are summarized in **Table 1**.

The spinel phase starts to appear at about 638 K, and the transition is complete by 683 K (**Figure 2**). Occupancies of all the sites and unit cell volume of the spinel phase during the phase transition are plotted as a function of temperature in **Figure 3**. The full occupancy of the oxygen site at the beginning of the transformation indicates that an ideal oxygen framework is formed when the phase transition starts. The tetrahedral (Si^{4+}) sites start with an occupancy of 69% in the first analyzed pattern, and become fully occupied earlier than the octahedral (Fe^{2+}) sites which start with an occupancy of 74%. Although the structure refinement does not yield a full occupancy for the octahedral sites when the transition is completed, the R-factor (12.18%) yielded from this refinement is not significantly smaller than that (12.55%) from the refinement with a forced full occupancy for the octahedral sites. On the other hand, forcing full occupancies of tetrahedral and octahedral sites in the refinement at the beginning of the transition increases the R-factor from 9.98% to 12.99%. An increase in relative intensity of cation dominated diffraction peaks to anion dominated diffraction peaks with temperature is illustrated in the inset of Figure 1. Therefore the cations can be considered less ordered at the beginning, and they gradually find their sites as the transition proceeds. The disordered cations probably exist in the crystal as transitional defects during the process. Refined unit cell volumes plotted in Figure 3 show that the increase in cell volume with temperature as the cation site occupancy increases is much faster than that after the sites are full occupied. In other words, the cell volume increases with the cation site occupancy. However the volumetric difference is much less significant energetically than the consequence of defects caused by disordered cations, because $\Delta V \cdot P$ is

Table 1

Result of the structure refinements for spinel during the olivine-spinel phase transformation

T(K)	a (Å)	V (Å^3)	U	F*(Si)	F*(Fe)	Ph†	R‡(%)	U(Si)(Å^2)	U(Fe)(Å^2)	U(O)(Å^2)
643	8.1834(4)	548.03(4)	0.2416(12)	0.69(5)	0.74(5)	0.26	9.98	0.005(2)	0.016(2)	0.040(1)
648	8.1853(2)	548.42(3)	0.2425(9)	0.87(4)	0.85(3)	0.43	9.53	0.007(4)	0.018(2)	0.024(4)
653	8.1872(2)	548.78(2)	0.2424(7)	0.94(3)	0.90(2)	0.62	10.40	0.012(3)	0.015(1)	0.017(4)
658	8.1880(2)	548.94(2)	0.2445(6)	1.00(3)	0.90(2)	0.77	11.01	0.018(2)	0.018(1)	0.025(3)
663	8.1890(1)	549.15(2)	0.2431(7)	1	0.91(1)	0.82	10.91	0.017(2)	0.019(1)	0.027(3)
668	8.1938(1)	550.12(1)	0.2418(6)	1	0.95(1)	0.92	11.84	0.018(2)	0.020(1)	0.022(3)
673	8.1948(2)	550.31(2)	0.2416(6)	1	0.94(1)	0.95	12.12	0.020(2)	0.020(1)	0.018(2)
683	8.1953(2)	550.41(3)	0.2418(6)	1	0.94(1)	1	12.18	0.020(2)	0.017(1)	0.018(3)

* F: site occupancy. The integer 1 indicates that the site is constrained at full occupancy in the refinement.
† Ph: spinel phase fraction in the sample.
‡ $R = \Sigma \left| I_{obs} - I_{cal} \right| / \Sigma I_{obs}$

about two orders of magnitude smaller than the estimated defect energy[7]. Therefore the phase transformation proceeds to the stable state, at which all the cations order into their respective sites.

Neither the pure diffusional nor the diffusionless transformation mechanism can properly interpret the experimental observation. The diffusional mechanism describes a nucleation and growth process[8], in which both nucleation and growth involve destruction of the old structure and generation of a new structure with fully occupied crystallographic sites. The diffusionless (martensitic) transformation is always associated with displacement of atoms in the old structure and formation of a new structure without diffusion. The two structures have to be crystallographically closely related with a similar atomic arrangement along a corresponding orientation (macroscopic invariant plan) in each structure, as in fcc-bct, fcc-hcp, bcc-hcp transitions in metals and alloys[1]. The transformation is very stress sensitive because it involves only systematic shears of the atomic arrangement. In such an ideal martensitic transformation, no delayed atomic rearrangement is expected. However, in the case of fayalite, the coupled crystallographic orientations in the olivine and spinel structures are defined by their oxygen sublattices (i.e. hcp and fcc in olivine and spinel structure, respectively); the cation arrangement is distinct from the oxygen sublattice, and the required shuffling of cations from olivine to spinel structure is directional divergent[2]. Therefore the cation rearrangement may not synchronize with the anion displacement. Most of the previous studies on mechanism of the olivine-spinel phase transition are TEM investigations. Observations of an epitaxial relationship in the recovered sample are not enough to characterize the cation reordering. Few x-ray diffraction studies on the transformation mechanism in fayalite have been done[9,10,11]. Due to the technical limitation, none of these studies achieved good quality data for structure refinements. Furnish and Bessett[10] observed an early appearance of the (400) and (440) spinel diffraction peaks. Since the (400) peak is mainly contributed by the oxygen sublattice they inferred that the oxygen sublattice in spinel may form earlier then the cation sublattice. However, their results are questioned by a later study[11].

Investigating a recovered sample which was quenched during the phase transition in a separate experiment by TEM , we are able to find the typical epitaxial relation (i.e. $(100)_{Ol}$ is parallel to $(111)_{Sp}$) between adjacent olivine and spinel phases. As modeled by Poirier[2] for a pure martensitic transition, such a epitaxial relation indicates that atomic displacements along the (100) planes of olivine structure transform the hcp oxygen arrangement of olivine into the fcc oxygen sublattice of the spinel structure. Combining this fact with other experimental observations, the stress sensitivity of the transformation and the significant delay of Si^{4+} and Fe^{2+} cation reordering with respect to the oxygen anions, we believe that the olivine-spinel phase transformation in fayalite is controlled by a new pseudomartensitic mechanism, i.e. a diffusionless martensitic transition of anion sulattice coupled with diffusion controlled cation reordering.

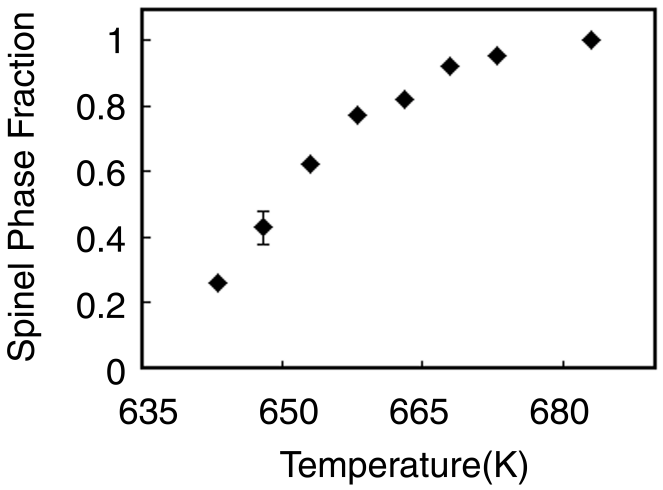


Figure 2. Refined spinel phase fraction in the sample during the olivine-spinel phase transition in fayalite.

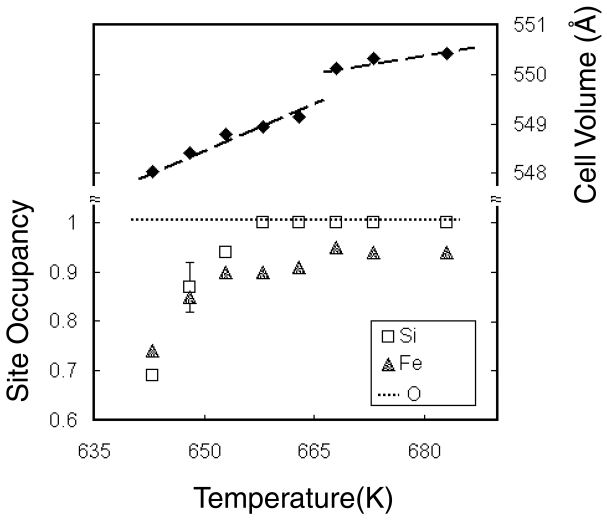


Figure 3. Site occupancies and unit cell volume of spinel during the olivine-spinel transformation. Bars on the symbols indicate the experimental uncertainties. The dashed lines in the volume data are drawn to demonstrate a higher rate of volume increase with temperature during the cation reordering.

This research is carried out (in part) at the NSLS, Brookhaven National Laboratory, which is supported by the U.S. Department of Energy, Division of Materials Sciences and Division of Chemical Sciences. We thank J. B. Hastings and Z. Zhong at the NSLS for their technical support at the X17B1 beamline and D. Lindsley for offering the fayalite sample. The Center for High Pressure Research is jointly supported by the National Science Foundation (NSF) and the State University of New York

at Stony Brook. The research is also supported by the NSF research grant to JC and DW. This paper represents a part of the original publication in Physical Review Letters (Vol. 86, 4072-4075, 2001).

References

1. *Z. Nishiyama,* Martensitic Transformation. *M. E. Fine, et al., Eds. (Academic Press, New York, 1978); D. A. Porter and K. E. Easterling,* Phase Transformation in Metals and Alloys *(Chapman & Hall, London, ed. Second, 1992).*
2. *J.P. Poirier, in* Anelasticity in the Earth *F. D. Stacey, et al., Eds. (American Geophysical Union/Geological Society of America, Washinton D.C./Boulder, Colorado, 1981), vol. 4, pp. 113 .*
3. *A. Lacam, et al.,* Nature ***288****, 155 (1980); P.J. Vaughan, et al.,* Nature ***298****, 357 (1982); N. Hamaya and S.-i. Akimoto,* Physics of the Earth and Planetary Interiors ***29****, 6 (1982); J.N. Boland and R. Liebermann,* Geophysical Research Letters ***10****, 87 (1983); D.C. Rubie, et al.,* Journal of Geophysical Research ***95****, 15 (1990); Y. Wang, et al.,* Science ***275****, 510 (1997); M. Madon, et al.,* Physics and Chemistry of Minerals ***16****, 320 (1989); J.N. Boland and L.-g. Liu,* Nature ***303****, 233 (1983); L. Kerschhofer, et al.,* Mineralogical Magazine ***62****, 617 (1998); P.C. Burnley and H.W. Green(II),* Nature ***338****, 753 (1989); P.C. Burnley,* American Mineralogist ***80****, 1293 (1995); A.R. Remsberg, et al.,* Physics and Chemistry of Minerals ***15****, 498 (1988); P.J. Vaughan, et al.,* Tectonophysics ***108****, 299 (1984); I. Martinez, et al.,* Journal of Geophysical Research ***102****, 5265 (1997); D. C. Rubie and P. E. Champness,* Bull Mineral ***110****, 471 (1987); L. Kerschhofer, et al.,* Physics of the Earth and Planetary Interiors ***121****, 59 (2000).*
4. *J. Chen, et al.,* Review of High Pressure Science and Technology ***7****, 272 (1998).*
5. *D.J. Weidner, et al., in* High-pressure research: Application to Earth and Planetary Sciences *Y. Synono and M.H. Manghnani, Eds. (Terra Scientific Publishing Company / AGU, Tokyo / Washington, D.C., 1992) pp. 13 .*
6. *A.C. Larson and R.B. Von Dreele, "GSAS, General Structure Analysis System"* LAUR 86-748 *(Los Alamos National Laboratory, 1986).*
7. *R.L. Stocker,* Physics of the Earth and Planetary Interiors ***17****, 108 (1978).*
8. *C.-M. Sung and R. Burns,* Tectonophysics ***31****, 1 (1976).*
9. *K. Inoue, Ph.D. Thesis, Tokyo University (1975).*
10. *M.D. Furnish and W. A. Bassett,* Journal of Geophysical Research ***88****, 10 (1983).*
11. *T. Yagi, et al.,* Journal of Geophysical Research ***92****, 6207 (1987).*

Appointment of Steven Dierker as NSLS Chairman

Richard Osgood
Assistant Laboratory Director for Basic Energy Sciences

On May 7, Steven Dierker arrived at the NSLS to assume his new role as Chairman of the NSLS. He took the "baton" from Sam Krinsky who served as Interim Chair while still carrying out his responsibilities as Head of the Accelerator Section. Steve comes to Brookhaven after having been Professor of Physics at the University of Michigan. Steve's career includes many years of experience working at synchrotron facilities. In fact, his work in synchrotron measurements started at the NSLS where he pioneered the new field of X-ray Photon Correlation Spectroscopy. As a result, he is deeply familiar with the NSLS, and many of its staff and support personnel.

Upon receiving his PhD at the University of Illinois at Urbana-Champaign in 1983, Steve joined the Technical Staff in the Physics Research Division of the Semiconductor and Chemical Physics Dept. at AT&T Bell Laboratories, where he used various visible light scattering and small angle neutron scattering techniques in his research. He focused on studies of soft condensed matter systems, most notably the 2D hexatic phase of freely suspended liquid crystal films and activated dynamics of binary fluid mixtures in porous media.

In 1990, Steve left Bell to join the Physics Dept. at the Univ. of Michigan as an Associate Professor of Physics and Applied Physics, where he set up a light scattering laboratory. In 1991, he began to shift his research from visible light scattering to synchrotron based x-ray scattering. He began a program to develop the technique of X-ray Photon Correlation Spectroscopy (XPCS) in a series of experiments at the NSLS and ESRF. He has served as one of the leaders of the Center for Real-Time X-ray scattering of the MHATT-CAT at the APS and also on many committees that review research at synchrotron facilities. He has recently begun to assume a national leadership role in synchrotron matters and last year organized a visit to Congress by the Chairs of the Users Groups for the four DOE synchrotron facilities.

His experience as a researcher in materials science and as a member of the synchrotron community will serve the NSLS well in the coming years.

Letter from the Chairman

Steven Dierker, NSLS Chairman

The NSLS is currently the world's most productive synchrotron light source, and possibly the most productive of all major science user facilities today. It is unique in the range of tools it provides to its users as well as in the energy range these techniques cover, and its capabilities continue to be in high demand. We enjoy an ever increasing number of users, nearly 2600 in 2000, and the scientific impact of their work continues to be strong. As we approach the challenges of the future, our aim must be to continue to develop the NSLS to maintain its excellence as well as its usefulness to the scientific community. I believe we have three fundamental challenges facing us.

First, we need to ensure the strength of our current user base and the successful continued operation of the existing facility. We are challenged by an aging infrastructure and beamlines while attempting to meet the demands of the largest user program of any facility, but we must remain committed to this goal. This also includes working with our user groups to assist in their efforts to maintain appropriate funding levels to operate at the NSLS. At the same time it is urgent that we aggressively pursue the future.

One lesson of recent years is that increasingly diverse areas of science have recognized the power of synchrotron radiation and the number of users continues to grow. Thus, our second task is to encourage and nurture new user communities and to be responsive to the changing ways in which scientists use the facility, emphasizing activities that lead to the highest quality science. An outstanding example of how this might be accomplished is the success of Structural Biology researach at the NSLS in recent years. We are now embarking on several efforts to organize new user groups along the lines of science driven consortia that would utilize multiple beamlines to provide a suite of experimental techniques.

Finally, we must aggressively pursue the development of future source technologies that might form the basis of an upgrade to the existing facility, or perhaps even an entirely new facility. In this area, we have several efforts ongoing. Especially noteworthy are our efforts, in partnership with the user community and other institutions, to make a strong scientific case, and to resolve a number of important technical issues, for a new source based on photoinjected energy recovery linac (PERL) technology.

The laboratory and DOE are strongly committed to the future of synchrotron science at BNL. I look forward to working with all of you to ensure its continued success.

2001-02 Users' Executive Committee and Special Interest Groups (SpIGs)

Standing: Mark Fuhrmann, Mike Becker, Erik Johnson, Tony Lanzirotti, Peter Stephens, John Sutherland, Vince Harris, Dave Mullins, Lisa Miller. Seated: Michael Vaughan, Mary Anne Corwin, Simon Bare, Leemor Joshua-Tor, Dan Fischer. Missing from photo: Mark Chance, Steve Almo, Mahbubul Khandaker, Paul Stevens, Cecilia Sanchez-Hanke, Michael Dudley.

Chair	Simon Bare (UOP, LLC)
Vice Chair	Leemor Joshua-Tor (Cold Spring Harbor)
Past Chair	Mark Chance (AECOM)
Secretary	David Mullins (ORNL)
Member	Michael Vaughan (SUNY/SB)
Member	Steven Almo (AECOM)
Member	Daniel Fischer (NIST)
Member	Tony Lanzirotti (U. of Chicago)
Ex-Officio	Mary Anne Corwin (BNL/NSLS)
Ex-Officio	Erik Johnson (BNL/NSLS)
Bio Scattering	Michael Becker (BNL/Biology)
Imaging	Mark Fuhrmann (BNL/Env Sci)
Industrial	Paul Stevens (Exxon)
Infrared	Lisa Miller (BNL/NSLS)
Nuclear Phys	Mahbubul Khandaker (JLab)
Students/Post	Cecilia Sanchez-Hanke (BNL/NSLS)
Time Resolved	John Sutherland (BNL/Biology)
Topography	Michael Dudley (SUNY/SB)
UV Photo	David Mullins (ORNL)
XAFS	Vince Harris (NRL)
Xray Scattering	Peter Stephens (SUNY/SB)

A User's Perspective

Simon R. Bare
UEC Chair, UOP LLC

On May 22, at the NSLS Users' Meeting, I took over the reigns from Mark Chance as UEC Chair, and the newly elected UEC members became part of the committee. I would like to give a wholehearted thank you to Mark, and to Ken Evans-Lutterodt, Chris Jacobsen, and Barbara Illman, the three outgoing members of the UEC, for all the work and effort they have put in to making the Light Source a better place for all of us to work at.

This has been a challenging year, with the departure of Michael Hart, and the search for a new NSLS chair. The good news is that we have a new Chair of the NSLS. Dr. Steven Dierker, Professor of Physics at the University of Michigan, took up the position earlier this month (see article about Steven in this Newsletter). On behalf of all the users at the NSLS, I welcome Steve to his new position. We look forward to his leadership and to working with him over the years.

On May 7, Mark Chance and I, together with the chairs of the three other U.S. DOE synchrotron facilities visited Washington, D.C. to lobby for increased budgets for the Office of Science and for the physical sciences overall. This visit follows up on a lobbying visit initiated last year by the same group of people. These visits will be ongoing and an essential part of the activities of the User Committees from the ALS, APS, SSRL and NSLS. We met with staffers of the Senate and House Energy & Water Appropriations Committees, House Science Committee staff, and legislative directors for several key members of the House Energy & Water Appropriations Committee. We briefed them on the significant technological, educational and economic impact the four synchrotrons have, and stressed how important it is to have balanced funding in the sciences. We requested that the overall research budget for the physical sciences be increased at 15% over current levels, and that the budget should be doubled over a five year period. Additional meetings were held on May 8 with representatives from the Office of Science and Technology Policy and the Office of Management and Budget. The aim of these meetings was to begin working on budgets for FY2003. This meeting was then followed by a series of meetings with local (NY and NJ) Congressional and Senate representatives. You will be hearing more about additional visits later in the year. One point that was stressed to us time and time again was not to underestimate the impact of visits to the local offices (not D.C.) of your Representatives and Senators. I urge you to make contact with your local Congressperson. Go visit their office, begin the educational process of explaining to their local office staff the importance of the NSLS, and other synchrotrons. This is especially crucial for those of you that live or work in districts outside of Congressman Grucci's district. Your local Congressperson probably has no idea that the NSLS exists, far less the importance of the science that is conducted here. If you would like help on formulating the argument, or would like additional resources before your visit, please do not hesitate to contact me.

I would also request your help when you receive an e-mail from me requesting that you take action regarding the federal budget process. Most often this action will entail writing a letter to the appropriate party requesting support of the Office of Science budget. I quote from an article written by past-UEC chair, John Parise, in July 1998: "Believe it or not, 'I contribute by doing good science and publishing articles acknowledging the Light Source' really is not good enough. You know – it never really was". This is more true now than it ever was. We need your active involvement in this ongoing lobbying process.

I would also like to draw your attention to another serious issue. There is going to be a review of the NSLS by a DOE-appointed committee in July. You may be asked to contribute to this review in some form or other – maybe it will be replacing the poster at your beamline with updated research, or maybe it will involve something else. Please take this request seriously. These reviews do matter. As Users of the NSLS we all have obligations that we have to meet. When you get a request from the User Office for a list of publications, or list of invited talks, or whatever information is requested, please heed this request. The statistics garnered through these requests are used to compile critical information to present to the DOE on the productivity of the NSLS.

I would like to close by saying that the UEC exists to provide an independent forum for the interests of you, the User of the NSLS. Please do not hesitate to contact me if you have any concerns, questions, or ideas about all aspects of the NSLS. We can't act on your concerns if we don't know about them. Please send comments to me at srbare@uop.com.

2001 Annual Users' Meeting

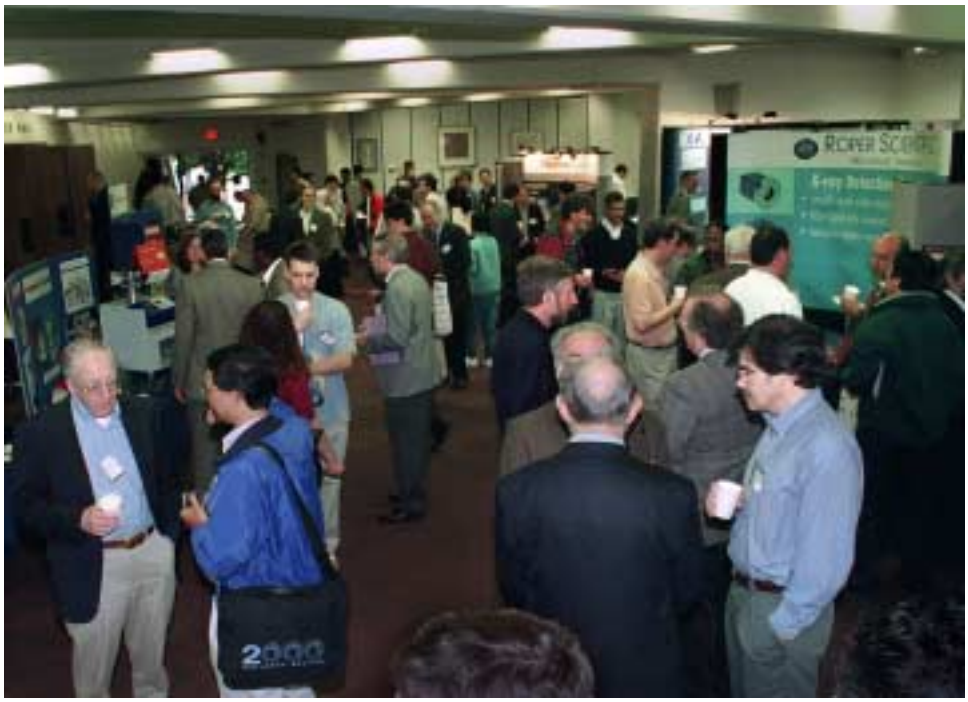
Simon R. Bare
UEC Chair, UOP LLC

The National Synchrotron Light Source Annual Users' Meeting was held at Brookhaven National Laboratory on May 21-24, 2001. This was a record-breaking meeting in several respects: there was an all-time high attendance (370 registered attendees). In addition to the main meeting, there were seven workshops and one forum extending over four days, attended by a total of 480

people, and the cafeteria at Berkner Hall was transformed into the island of Maui for the Hawaiian luau banquet! This truly shows the vibrancy, interest and impact of the research being performed at the NSLS. The poor weather did not stop anyone from enjoying and participating in a stimulating meeting.

The week's events began early in the morning on Monday May 21 with a forum on the needs for advanced detectors for synchrotron science, organized by Peter Siddons. This was followed by three workshops held in parallel: "Environmental Molecular Sciences," organized by Richard Reeder & Tony Lanzirotti, "Synchrotron Techniques for Catalytic Studies," organized by Jingguang Chen, and "Frontiers in Structural Biology at High Brightness X-ray Sources, organized by Mike Becker & Lonny Berman. The meeting poster session, reception, and opening of the vendor exhibits were held on the Monday evening. At the poster session, 60 posters were presented. Judges had a difficult time deciding on the eventual winners of the students & postdocs poster competition. After some deliberation, the following were awarded $75 prizes: Kaveh Adib (Columbia University), "Chemistry of carbon tetrachloride on natural single-crystal hematite surfaces;" Cecilia Sanchez-Hanke (Brookhaven National Laboratory), "Exchange bias studies with soft x-ray circular polarized light;" Raymond Huang (Albert Einstein College of Medicine), "Characterization of bone mineral composition in the proximal tibia of cynomologus monkeys: the effect of ovariectomy and nandrolone treatment;" and Zikri Yusof (University of Connecticut), "Quasiparticle liquid of highly overdoped Bi2212." They are all congratulated on their excellent work.

Poster Session at Monday Night's Reception in Berkner Hall, where 60 posters were presented.



NSLS Users' Meeting attendees and vendor exhibitors at Berkner Hall. 370 attendees registered for this year's meeting.

On Tuesday, the main meeting began with an update on the activities at BNL by Dr. John Marburger, BNL Lab Director. He commented that the Light Source is by far the most productive facility in the laboratory, and with the recent closing of the medical reactor the lab is now an accelerator laboratory with a future based on accelerator science. He re-

ported that there is good support for the lab as a whole by local members of Congress and Senate. He is guardedly optimistic about the future of the lab, particularly the NSLS.

Dr. Richard Osgood, Jr., Associate Laboratory Director for Basic Energy Sciences, followed Marburger's welcome with a presentation on the lab's Nanoscience Initiative, emphasizing how a proposed Nanocenter at BNL could foster new collaborations with university and industrial partners, as well as interdisciplinary work among scientists at BNL. He described the proposed center as a building attached to the NSLS that would serve as "a bridge connecting technology in the Instrumentation Division with the NSLS and with the research on materials science on site." The building would house laboratory clusters focused on topics such as materials synthesis, proximal probe microscopy, and ultrafast optical science.

Recurring themes throughout the meeting were the excitement about nanoscience, increasing the number of users, and concerns about the funding for Basic Energy Sciences, and of Office of Science overall.

Dr. Pat Dehmer, DOE's Assoc. Director of Science for Basic Energy Sciences, gave a fascinating presentation illustrating the connection between public attitudes and funding for scientific endeavors. She emphasized that while public confidence in scientific leadership has remained fairly stable through the years, it is not based on fundamental knowledge of science, which makes that support tenuous. "This puts a burden on us to educate the public and Congress," she said if the budget picture is to improve. She commented that the NSLS will have to operate with a very tight belt in the next year. However, she was optimistic about the potential for scientific advances. "We are on the verge of really explosive discoveries in chemistry and materials science," she said.

Dr. Steve Dierker, the recently appointed new chair of the NSLS, was introduced to the user community and shared his initial views of the facility. He gave an upbeat presentation, emphasizing that while there are challenges ahead, "Our aim must be to continue to develop the Light Source in order to maintain its excellence and usefulness to scientists". He predicted that the number of users will continue to grow and that one avenue for this growth may be in collaborative research, for example in catalysis, materials science, soft matter physics, or studies of magnetic materials. "Many of these fields would benefit by combining suites of beam lines to provide access to many techniques and mechanisms for rapid access and quick turnaround," he said. Such a setup could also serve a larger number of users and provide for more cost-effective maintenance of facilities.

Dr. Jane "Xan" Alexander, Acting Director of the Defense Advanced Research Projects Agency (DARPA), was the meeting's keynote speaker. She commented that while the other speakers had mentioned the bleak-funding outlook in this year's budget, her agency's funding picture was looking good! Her presentation was a look into the future – a world beyond silicon-based electronic devices. Today's technology has a natural limit in the number of devices, and thus the speed that can be fabricated using silicon. She gave several examples of ways to go through, around, or under this wall. Xan talked of "molecular electronics" where it might be possible to develop computer chips on a scale that is 10,000 times smaller than today's. One idea would be to have these circuits assemble themselves from the molecular level, a fascinating thought!

Another approach would be to develop electronics based on spin rather than charge, which she said, could yield much faster devices and unbreakable codes.

Dr. Thomas Weber, Director of Division of Materials Research at the NSF, presented an overview of the national nanoscience initiative, with an emphasis on NSF's role. He stressed that the real driver

Left to right: Simon Bare (UOP LLC), Iran Thomas and Pat Dehmer (DOE/BES), Rick Osgood, John Marburger and Steve Dierker (BNL)

behind the initiative is economic, not just scientific.

The day rounded out with excellent technical presentations. Mark Chance discussed the development of small gap undulator technology at the NSLS. Sam Krinsky presented an overview on source development at the NSLS, and the photoinjected energy recovery LINAC (PERL) upgrade project for the NSLS was discussed by Ilan Ben-Zvi. Seth Darst showed how crystallography is leading to new insights in structural biology with a talk on structural studies of prokaryotic RNA polymerase. He commented that such insights would not have been possible without the advances in synchrotron radiation-based crystallography over the last few years. Jingguang Chen demonstrated the power of soft x-ray absorption spectroscopy in elucidating surface reaction mechanisms in catalysis, and Jim Penner-Hahn presented recent results on the local structure around the active zinc site in enzymes.

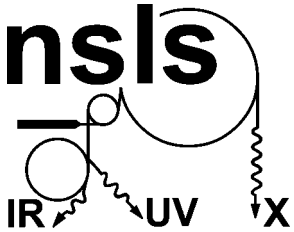
Left to right: Mary Anne Corwin (NSLS), Sue Wirick (SUNY/SB), Chi-Chang Kao (NSLS), Simon Bare (UOP LLC), Lisa Miller, Lydia Rogers and Nancye Wright (NSLS), and Dan Fischer (NIST).

This year the conference banquet was held onsite with the theme of a Hawaiian luau. The cafeteria at Berkner Hall was transformed into an island paradise with tropical decorations and music. Attendees truly got into the theme: colorful Hawaiian shirts were worn by many, and leis were presented as one entered "the island." There was entertainment in the form of Hawaiian dancers and fire dancer – along with some entertaining dancing by some of the attendees!

The meeting continued on Wednesday morning with three more workshops: "Advanced methods and tricks of EXAFS data modeling," organized by Anatoly Frenkel, "IR micro-spectroscopy: a molecular probe with micron resolution," organized by Lisa Miller, and "Applications of synchrotron radiation in nanoscience & technology," organized by Peter Johnson and Chi-Chang Kao. The meeting concluded on Thursday with a workshop on "XAFS data reduction and analysis using WinXAS" by Thorsten Ressler, and the hands-on portion of the IR workshop.

The success of the meeting was only possible with the help of many people. I would personally like to acknowledge the hard work and dedication of this year's organizing committee: Lydia Rogers, Nancye Wright and Mary Anne Corwin, from NSLS User Administration, Lisa Miller, Dan Fischer, Sue Wirick, and Chi-Chang Kao. I would also like to thank the workshop organizers and all the speakers for such a stimulating and enjoyable meeting. I would also like to acknowledge both the vendors that chose to participate in the vendor exhibit and also the corporate sponsors. It is only with support of these companies that meeting costs can be held at a reasonable level. The planning for next year's meeting has already begun – please block out the dates May 20-23, 2002 on your calendars!

Mark your calendar!
2002 NSLS Users' Meeting
May 20-23, 2002



X-Ray Ring Long Range Schedule

X-RAY SCHEDULE - September 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1 Holiday
2 Holiday	**3** Holiday	**4** Holiday	**5** 00-1200 Cond. 1200-2400 Ops.	**6** 00-2400 Ops.	**7** 00-2400 Ops.	**8** 00-2400 Ops.
9 00-2400 Ops.	**10** 00-1200 Ops. 12-2400 Studies	**11** 00-0600 Studies 06-1200 Intlk. 12-2400 Studies	**12** 00-1200 Studies 12-2400 Ops.	**13** 00-2400 Ops.	**14** 00-2400 Ops	**15** 00-2400 Ops.
16 00-2400 Ops	**17** 00-1200 Ops. 12-2400 Studies	**18** 00-0800 Studies 08-2400 Maint.	**19** 00-2400 Maint.	**20** 00-1200 Studies 12-2400 Ops.	**21** 00-2400 Ops.	**22** 00-2400 Ops.
23 00-2400 Ops.	**24** 00-2400 Ops.	**25** 00-0800 Ops. Template 08-2400 Ops.	**26** 00-2400 Ops.	**27** 00-2400 Ops.	**28** 00-2400 Ops.	**29** 00-2400 Ops.
30 00-1200 Ops. 12-2400 Studies						

X-RAY SCHEDULE - October 2001

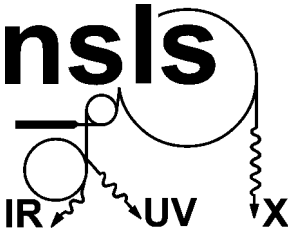
Sun	Mon	Tue	Wed	Thu	Fri	Sat
	1 00-0600 Studies 06-1200 Intlk. 12-2400 Studies	**2** 00-1200 Studies 12-2400 Ops.	**3** 00-2400 Ops.	**4** 00-2400 Ops.	**5** 00-2400 Ops.	**6** 00-2400 Ops.
7 00-2400 Ops.	**8** 00-1200 Ops. 12-2400 Studies	**9** 00-2400 Studies	**10** 00-1200 Studies 12-2400 Ops.	**11** 00-2400 Ops.	**12** 00-2400 Ops.	**13** 00-2400 Ops.
14 00-2400 Ops.	**15** 00-1200 Ops. 12-2400 Studies	**16** 00-0800 Studies 08-2400 Maint.	**17** 00-2400 Maint.	**18** 00-1200 Studies 12-2400 Ops.	**19** 00-2400 Ops.	**20** 00-2400 Ops.
21 00-2400 Ops.	**22** 00-2400 Ops.	**23** 00-0800 Ops. Template 08-2400 Ops.	**24** 00-2400 Ops.	**25** 00-2400 Ops.	**26** 00-2400 Ops.	**27** 00-2400 Ops.
28 00-1200 Ops. 12-2400 Studies	**29** 00-0600 Studies 06-1200 Intlk. 12-2400 Studies	**30** 00-1200 Studies 12-2400 Ops.	**31** 00-2400 Ops.			

X-RAY SCHEDULE - November 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1 00-2400 Ops.	**2** 00-2400 Ops.	**3** 00-2400 Ops.
4 00-2400 Ops.	**5** 00-1200 Ops. 12-2400 Studies	**6** 00-0800 Studies 08-2400 Maint.	**7** 00-2400 Maint.	**8** 00-1200 Studies 12-2400 Ops.	**9** 00-2400 Ops.	**10** 00-2400 Ops.
11 00-2400 Ops.	**12** Holiday 00-2400 Ops.	**13** 00-0800 Ops. Template 08-2400 Ops.	**14** 00-2400 Ops.	**15** 00-2400 Ops.	**16** 00-2400 Ops	**17** 00-2400 Ops.
18 00-2400 Ops.	**19** 00-0800 Ops. 08-2400 Maint.	**20** 00-2400 Maint.	**21** 00-2400 Maint.	**22** Holiday	**23** Holiday	**24** 00-2400 Maint.
25 00-2400 Maint.	**26** 00-2400 Maint.	**27** 00-2400 Maint.	**28** 00-2400 Maint.	**29** 00-2400 Maint.	**30** 00-2400 Maint.	

X-RAY SCHEDULE - December 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1 00-2400 Maint.
2 00-2400 Maint.	**3** 00-2400 Maint.	**4** 00-2400 Maint.	**5** 00-2400 Maint.	**6** 00-2400 Maint.	**7** 00-2400 Maint.	**8** 00-2400 Maint.
9 00-2400 Maint.	**10** 00-2400 Maint.	**11** 00-2400 Maint.	**12** 00-2400 Maint.	**13** 00-2400 Maint.	**14** 00-2400 Maint.	**15** 00-2400 Maint.
16 00-2400 Maint.	**17** 00-2400 Maint.	**18** 00-2400 Maint.	**19** 00-2400 Maint.	**20** 00-2400 Maint.	**21** 00-2400 Maint.	**22** 00-2400 Maint.
23 00-2400 Maint.	**24** 00-2400 Maint.	**25** 00-2400 Maint.	**26** 00-2400 Maint.	**27** 00-2400 Maint.	**28** 00-2400 Maint.	**29** 00-2400 Maint.
30 00-2400 Maint.	**31** 00-2400 Maint.					



VUV Ring Long Range Schedule

VUV SCHEDULE - September 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1 **Holiday**
2 **Holiday**	**3** **Holiday**	**4** **Holiday**	**5** 00-2400 Studies	**6** 00-2400 Ops.	**7** 00-2400 Ops.	**8** 00-2400 Ops.
9 00-2400 Ops.	**10** 00-2400 Ops.	**11** 00-2400 Ops.	**12** 00-2400 Ops.	**13** 00-2400 Ops.	**14** 00-1800 Ops. 18-2400 Studies	**15** 00-2400 Ops.
16 00-2400 Ops.	**17** 00-2400 Ops.	**18** 00-0800 Ops. 08-2400 Studies	**19** 00-2400 Maint.	**20** 00-2400 Maint.	**21** 00-1800 Ops. 18-2400 Timing	**22** 00-2400 Ops.
23 00-2400 Ops.	**24** 00-1800 Ops. 18-2400 Timing	**25** 00-2400 Ops.	**26** 00-2400 Ops.	**27** 00-2400 Ops.	**28** 00-1800 Ops. 18-2400 Studies	**29** 00-2400 Ops.
30 00-2400 Ops.						

VUV SCHEDULE - October 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
	1 00-2400 Ops.	**2** 00-0800 Ops. 08-2400 Studies	**3** 00-2400 Studies	**4** 00-2400 Ops.	**5** 00-2400 Ops.	**6** 00-2400 Ops.
7 00-2400 Ops.	**8** 00-2400 Ops.	**9** 00-2400 Ops.	**10** 00-2400 Ops.	**11** 00-2400 Ops.	**12** 00-1800 Ops. 18-2400 Studies	**13** 00-2400 Ops.
14 00-2400 Ops.	**15** 00-2400 Ops.	**16** 00-0800 Ops. 08-2400 Studies	**17** 00-2400 Maint.	**18** 00-2400 Maint.	**19** 00-1800 Ops. 18-2400 Timing	**20** 00-2400 Ops.
21 00-2400 Ops.	**22** 00-1800 Ops. 18-2400 Timing	**23** 00-2400 Ops.	**24** 00-2400 Ops.	**25** 00-2400 Ops.	**26** 00-1800 Ops. 18-2400 Studies	**27** 00-2400 Ops.
28 00-2400 Ops.	**29** 00-2400 Ops.	**30** 00-0800 Ops. 08-2400 Studies	**31** 00-2400 Studies			

VUV SCHEDULE - November 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
				1 00-2400 Ops.	**2** 00-2400 Ops.	**3** 00-2400 Ops.
4 00-2400 Ops.	**5** 00-2400 Ops.	**6** 00-0800 Ops. 08-2400 Studies	**7** 00-2400 Maint	**8** 00-2400 Maint	**9** 00-1800 Ops. 18-2400 Timing	**10** 00-2400 Ops.
11 00-2400 Ops.	**12 Holiday** 00-2400 Ops.	**13** 00-2400 Ops.	**14** 00-2400 Ops.	**15** 00-2400 Ops.	**16** 00-1800 Ops. 18-2400 Studies	**17** 00-2400 Ops.
18 00-2400 Ops.	**19** 00-2400 Ops.	**20** 00-2400 Ops.	**21** 00-2000 Ops. 20-2400 Maint.	**22** **Holiday**	**23** **Holiday**	**24** 00-2400 Maint
25 00-2400 Maint	**26** 00-2400 Maint	**27** 00-2400 Maint	**28** 00-2400 Maint	**29** 00-2400 Maint	**30** 00-2400 Maint	

VUV SCHEDULE - December 2001

Sun	Mon	Tue	Wed	Thu	Fri	Sat
						1 00-2400 Maint
2 00-2400 Maint	**3** 00-2400 Maint	**4** 00-2400 Maint	**5** 00-2400 Maint	**6** 00-2400 Maint	**7** 00-2400 Maint	**8** 00-2400 Maint
9 00-2400 Maint	**10** 00-2400 Maint	**11** 00-2400 Maint	**12** 00-2400 Maint	**13** 00-2400 Maint	**14** 00-2400 Maint	**15** 00-2400 Maint
16 00-2400 Maint	**17** 00-2400 Maint	**18** 00-2400 Studies	**19** 00-2400 Maint.	**20** 00-2400 Maint.	**21** 00-2400 Maint	**22** 00-2400 Maint
23 00-2400 Maint	**24** 00-2400 Maint	**25** 00-2400 Maint	**26** 00-2400 Maint	**27** 00-2400 Maint	**28** 00-2400 Maint	**29** 00-2400 Maint
30 00-2400 Maint	**31** 00-2400 Maint					

VUV Ring Status

Stephen Kramer
VUV Ring Manager

The VUV ring, after several vacuum leaks in a fast bump magnet's ceramic gap, has been limping along at lower current (I< 600mA) and ring energy at the injection energy (E=737MeV). A remanufactured gap has been built to replace the leaking unit during the May shutdown. Analysis of the thermal pattern of the synchrotron radiation on this gap has shown high temperatures on the transition (tapered) section made of stainless steel. This thermal loading induces high stresses in the transition metal rings, causing them to fail more often than the other two identically built gaps in the ring. The other gaps have reduced thermal loading, as a result of their placement in the ring. The repaired gap will have a clamped on water cooling channel, which should reduce the temperature rise and therefore the thermal stresses in the rings. This change should greatly extend the lifetime of this ceramic gap. However, armed with this new information, an improved gap design effort is underway to provide a spare gap that will have even greater expected lifetime at the higher power levels expected in the future.

In addition to replacing the ceramic gap, the vertical stripline kicker will be replaced. This kicker had a damaged electrical connector that could have been the source of beam instabilities seen during non-stretched bunch (timing) operations. By electrically shorting out this kicker, during the winter shutdown, the current limiting instability was greatly reduced during the spring operating period. This allowed timing operations to be scheduled up to the current limit imposed by the ceramic gap.

The digital feedback system has been performing quite well for at least 6 months. This system, although not significantly improving the beam stability achieved with the older analog system, has proven to be more flexible for operations when problems arise. If a Beam Position Monitor (BPM) or trim power supply fails during operations, the digital system can have the offending unit disabled from the algorithm and the system returned to operations as quickly as possible. After the problem equipment has been repaired offline, it can then be re-enabled in the system during the next injection. At present, the old analog feedback system is being maintained as a backup system. However, after about a year's experience with the digital feedback system, the decision will be made as to when to eliminate the older system, freeing up valuable rack space.

The U3B and U12IR beam lines have been used to study the high frequency (microwave) impedance that the beam sees. This impedance is the source of numerous effects on the beam that increases the beam emittance as the current in the bunch is increased. These effects are hard to calculate in detail, since it is difficult to calculate this high frequency impedance. These measurements will help to improve the modeling of the impedance and possibly the source of high intensity, coherent synchrotron radiation emission in the mm to micron wavelength range.

X-Ray Ring Status

Jeff Rothman
X-Ray Ring Manager

Problems with the Linac resulted in significant downtime for the X-ray ring in March and April. The output power from Klystron #1 began to deteriorate during March. Every effort was made to compensate for the problem in an attempt to delay repair work until the May shutdown. On March 28th the output power dropped so low that operators could no longer inject into the booster ring. Emergency repairs began immediately to replace the failed one year old tube with a spare. The Power supply group and Vacuum group worked in shifts to complete the repairs as quickly as possible. The bake-out required after klystron replacement occupied much of the downtime. Once operations resumed the spare tube functioned well, until April 5th when a water leak in the high voltage tank caused it to fail. The spare was removed, dried out, and reinstalled with the water disconnected. Due to the low duty cycle of the Linac the klystrons can operate without cooling water. As of this writing we have not had any more problems with the Linac. The failed tube is being rebuilt and an investigation is underway to locate a more reliable klystron supplier.

The trim upgrade project has grown larger than originally expected. Operating at 2.8GeV and low emittance requires many of the trim magnets to operate near maximum current. This limits our ability to provide orbit corrections for users. Monitoring has shown that some trims near saturation now were not near saturation four months ago. This indicates that any of the X-ray trims could be a problem. We anticipate upgrading all 69 horizontal trim magnets and their power supplies. The task is complicated by the fact that there are six different types of trim magnets in the X-ray ring. The various upgrade options include, adding windings, lengthening the magnet, and increasing the current and adding cooling to the magnet. In many cases other components will need to be moved to make room for the upgrade. The mechanical group has completed a comprehensive survey of the trim magnets and made upgrade recommendations on a magnet-by-magnet basis.

The RF group installed a circulator in RF system 2 during the May shutdown. This device isolates the output power amplifier from the RF cavity, allowing the amplifier to drive a constant 50 ohm load regardless of beam current. Without isolation the amplifier can become unstable with small changes in tuning or small changes in the length of the transmission line. The circulator greatly simplifies tuning of the RF system, increases efficiency, and improves reliability.

Marching up the Periodic Table with Magnetic Scattering

G.H. Lander[1] and W.G. Stirling[2]
[1]European Commission, Institute for Transuranium Elements, Karlsruhe, Germany
[2]University of Liverpool, UK, and ESRF, Grenoble, France

The field of magnetic scattering with photons began with pioneering experiments by de Bergevin and Brunel [1] more than 20 years ago on a sealed tube source at the CNRS in Grenoble. Within a few years, with the advent of synchrotron sources, the photon fluxes were sufficiently large that such experiments could go beyond the demonstration phase. The study of the magnetism of holmium then became the benchmark with experiments being conducted by the BNL group of Doon Gibbs and his collaborators. The group performed some of their research at Cornell and Stanford while they waited for the NSLS to become fully operational in the mid-1980's. After making contributions to understanding the magnetic configuration in holmium, they observed the enhancement of the intensity of the magnetic scattering when the photon energy was tuned to close to that of the L_3 absorption edge of holmium [2]. This was rapidly understood in terms of atomic physics and standard resonance theory [3], and all rare-earth metals and many compounds have now been examined [4]. The theory suggested also that very large effects might be observed in actinides at the M edges, in which a 3*d* core electron is promoted directly into an empty 5*f* orbital. Within a year, experiments by Eric Isaacs and collaborators on UAs at the X14 beamline at NSLS had indeed shown [5] that enhancements as great as 10^6 were obtained with uranium, and presumably with other actinides. The magnetic signal from the single crystal of UAs as a function of incident photon energy is shown in **Figure 1**. These experiments opened the way for many studies of uranium compounds at BNL, Daresbury, and the ESRF, and no doubt at more places in the future.

We started our own experiments on uranium compounds 11 years ago at the NSLS. Many students experienced Long Island and the cooking of one of us for the first time. For the most part, they seem to have survived both experiences! The studies resulted in our development of a better understanding of the M resonances and their use in characterizing magnetic structures and phase transitions, as well as the observation of critical scattering. An additional spinoff was a successful program aimed at observing magnetic surface scattering [6] from UO_2, which was first observed at X22C in 1994 and is now continuing using the high brightness of the ESRF's ID20.

However, an interesting question was how to continue progress up the 5*f* series, as one glance at the periodic table shows that uranium is just at the beginning. As is well known, many compounds of U, Np, and Pu are magnetic even if the first magnetic element of the actinides is curium. In the early 1980s, the Institute for Transuranium Elements in Karlsruhe, Germany, had developed, in collaboration with ETH, Zürich, technology for making single crystals of Np and Pu compounds [7], and it seemed natural to try synchrotron experiments on them. In 1990, we presented BNL safety staff with the idea that we could work on encapsulated samples containing mg quantities of transuranium isotopes. After some discussion and modifications our proposal was accepted. In 1992, we saw our first signal from the Np resonance in an antiferromagnetic compound NpAs [8]. The resonances at Np are similar to those in U, except that, as expected, they are at higher energy. A good example (**Figure 2**) showing clearly the element specific nature of the probe is taken from our recent study [9] at the ESRF where we show the spectra for a pure Np compound and then one in which both U and Np atoms are randomly distributed. The lower figure (b) is interesting as one can tell without further data treatment that the U atoms in this alloy carry a magnetic moment. There would otherwise be no signal at the U M resonances. A careful treatment of the data also shows that the "branching ratio" (the ratio of the amplitudes at the M_4 and M_5 edges for each element) is decreasing in going from U to Np. This is expected and gives hope that the electronic structure may be accessible with this technique once the systematics are established. Further, one can hope to determine the ratio of the Np and U moments and their individual temperature dependencies.

Having succeeded with Np, it seemed reasonable to try the next element – plutonium. Unfortunately, there are special problems with Pu, both its handling, and especially the bureaucracy needed for its transportation. The discussions this time at BNL were more difficult – any accident concerning Pu would inevitably have enormous repercussions on the facility – and we only succeeded due to the extremely positive attitude of the NSLS and Lab safety staff. We had our first run in late 1996, and to our great concern, we did not succeed. In fact, not only did we fail to observe *magnetic* scattering, but we failed even to find a strong *charge* peak from the crystals. The only signal we observed was a strong absorption at the M position of Pu from what was a rather weak charge Bragg peak. One has to remember that the penetration of the photons at the M edges is about 2000 Å into the material. The strong absorption suggested an amorphous overlayer containing Pu that was absorbing but not diffracting. Steve Wasserman and Lynne Soderholm of Argonne National Laboratory [10] performed an EXAFS analysis on the Bragg intensity (essentially a DAFS spectrum) and showed that this was indeed the case and that

Pu-O was a likely contaminant on the surface of the crystals to the extent of perhaps a micron. Another attempt on the same samples a year later (1997) failed even to see the weak signal of a year before. It was clear we were losing further credibility [11].

Of course, failing at Pu certainly drew the attention of the safety staff and all the administrators who were monitoring our progress! We struggled over the cause of the problem. The mounting of the crystals used a modification of a method developed many years ago for neutrons, and there has never been any problem with neutron experiments on Pu-containing samples. However, even with the strongly absorbing isotope ^{239}Pu, the neutrons penetrate into the sample about 60 μm, so this is far greater than the x-ray experiments, and it seemed reasonable to believe that was the root of our difficulties – but why only with Pu? We then started the lengthy process of returning the samples to Karlsruhe to examine the crystals. Given the bureaucracy involved, this took nearly a year.

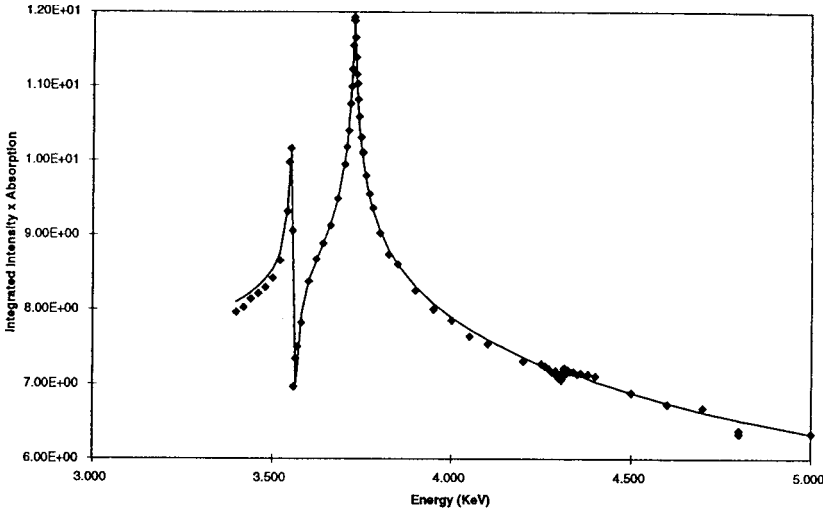


Figure 1: The antiferromagnetic signal from UAs as a function of photon energy. The solid line is a fit to three Lorentzians representing the M_5, M_4, and M_3 (in ascending energy) absorption edges. Data taken at X14 at the NSLS, Ref. [5].

Serendipity then took a hand. In discussions at the 1997 Baden-Baden Actinide Conference between Gerry Lander and John Haschke, a chemist from Los Alamos and an expert on plutonium, John was initially stumped over our problem. Finally, somewhat as an afterthought, he pointed out that if we were using some type of epoxy (which we were) there was a strong possibility (known to chemists in the Pu business for years!) that the alpha particles emitted from ^{239}Pu would radiolyse the epoxy. This would release a whole slew of chemical products, some, such as HF, being highly reactive and capable of attacking the crystal surface. This idea seemed so simple that it had to be correct. It also made us look rather foolish. But what should we use instead? Pierre Haen from the CRNS in Grenoble suggested (over dinner in Lisbon) that indium could be used to solder the crystals to the Cu plate, but we were still waiting to examine the crystals and see if the damage was visible. Indeed it was. **Figure 3** shows photos of the crystals on their return from BNL, indicating severe degradation and interesting nanoscale "stripe" structure on the surface. That this did not prevent neutron experiments (which attach the samples with the same epoxy) can be attributed to the greater penetration of neutrons as well as the larger (than the x-ray case) containers used in the encapsulation and hence the lower concentration of gases.

So it was back to the drawing board! Finally we had new samples, attached with indium, shipped to BNL. Unfortunately, this was in late 1998, and coincided with the severe problems at the HFBR at BNL. Safety hurdles were even greater than before. After additional documents and discussions, we got the green light and settled in at X22C in April 1999. Success! We saw beautiful resonances from Pu and proceeded to look at a mixed U–Pu composition to exploit the element specific nature of the probe. For these experiments, we prepared single crystals of $(U_{1-x}Pu_x)Sb$ since we knew the properties of the end members and there were some interesting questions about how the magnetic configuration would evolve across the series. A second successful run was made on another composition in April 2000. The intensity of

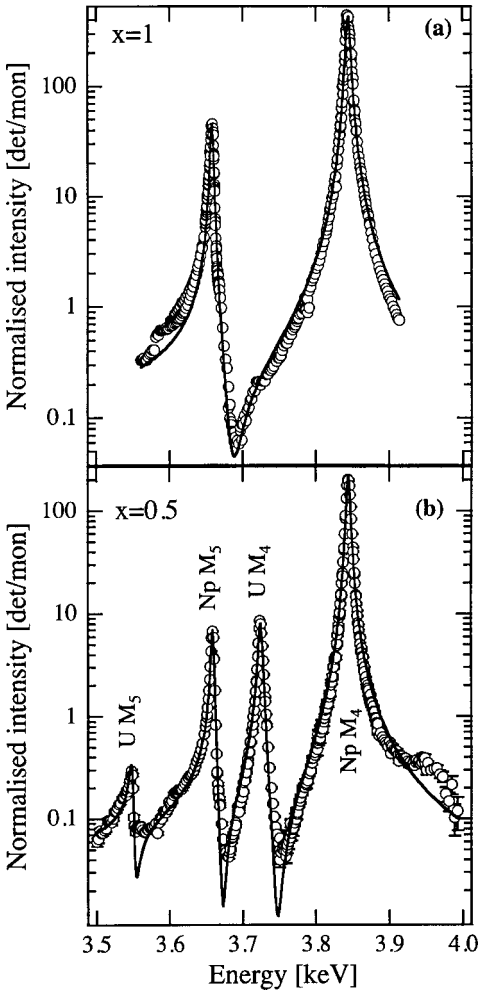


Figure 2: The energy dependence of the photon intensity from an antiferromagnetic peak in (a) $NpRu_2Si_2$ and (b) $(U_{0.5}Np_{0.5})Ru_2Si_2$ taken at the ID20 beamline at the ESRF, see Ref. [9].

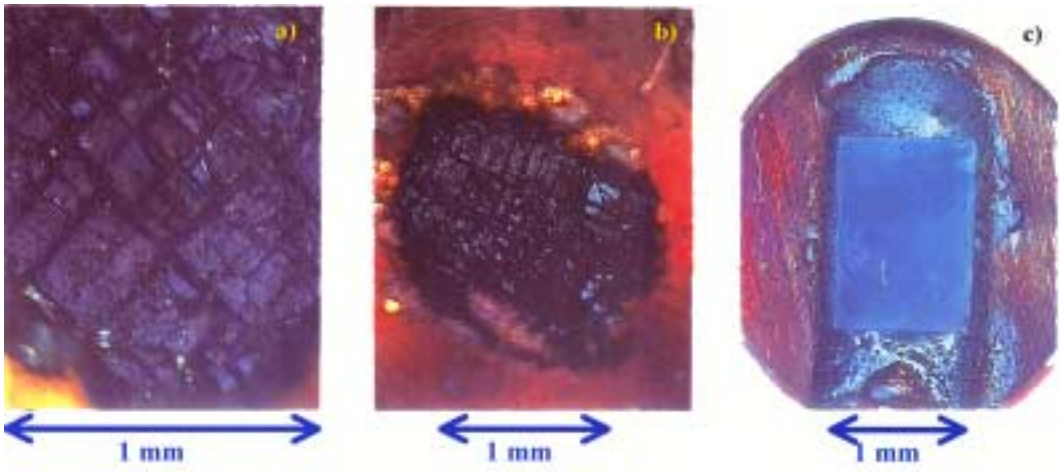


Figure 3 (color): Photos of the crystals of PuSb. (a)and (b) The crystal as it was returned to ITU after shipping to BNL and a total of about 2 years allowing the radiolysis of the epoxy by the ^{239}Pu . (c) The virgin crystal surface after attaching with indium at ITU.

tra of Pu compounds. Such an effort is now planned at the APS.

We have been able to use a combination of the x-rays (at NSLS) and neutrons (at the ILL in Grenoble) to unravel the magnetic phase diagram of the x = 0.75 sample. This is shown schematically in **Figure 5**. The magnetic configuration changes from the so-called 3*q* configuration at high temperature to a single-*q* configuration at low temperature. These changes are related directly to the electronic structures and, so, are becoming of increasing interest to theorists [12].

the antiferromagnetic reflection as a function of incident photon energy for the x=0.50 sample is shown in **Figure 4**. There are two points of interest. First, the ratio of the M_5 to M_4 intensities is much greater in Pu than it is in U. This is expected from the greater number of 5*f* electrons (5) in Pu as compared to the 3 in U^{3+}. Second, there is a new spectral feature on the high-energy side of the Pu M_5, which was also seen in the x = 0.75 sample and is probably a feature of many Pu spectra. While there are a number of speculations of what this might come from, there does not yet appear to be a clear winner. This motivated the need to look at the M-edge absorption spectra of Pu compounds. Such an effort is now planned at the APS.

We can expect more experiments on transuranium samples in the years to come and, indeed, experiments at the ESRF have provided new evidence [13] on NpO_2, which is one of the most puzzling materials despite first measurements of the specific heat almost 50 years ago. With a clearer picture of resonances from the first three actinide elements, theoretical advances are now being made to relate these intensities to the magnetic moments – a challenging task that could widen the application of this technique [9]. In addition, there is the need to under-

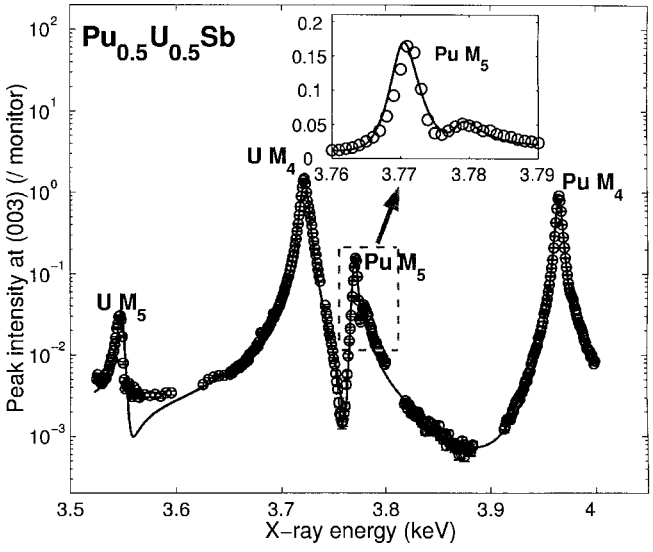


Figure 4: Intensity of the (003) magnetic reflection from $(U_{0.5}Pu_{0.5})Sb$ as a function of incident photon energy taken at the X22C beamline at the NSLS. Note the peaks attributed to U and Pu. In addition note the new spectral feature on the high-energy side of the Pu M_5 resonance. Taken from Ref. [15]

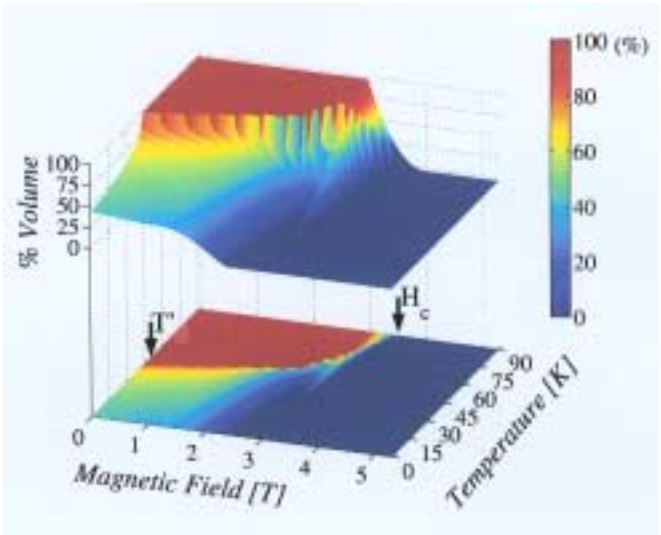


Figure 5: Schematic phase diagram for the $(U_{0.25}Pu_{0.75})Sb$ sample as a function of field (H) and temperature (T). Note that T_N is ~ 90 K. The plot has been made to show the percentage of the crystal in the 3q magnetic configuration at any H and T. Zero 3q implies that the configuration is 1q. Taken from Ref. [15]

stand the new spectral feature in the Pu spectra as seen in Fig. 4, as well as continuing up the periodic table. Curium awaits us, and now that we have seen magnetic scattering from ~ 40 μg of sample, thin films of Cm, if they could be grown epitaxially would be fascinating as Cm is reported to be antiferromagnetic. Diffraction peaks have been easily observed on a 80 Å thin film containing uranium [14], so the whole field of thin film, and multilayer, magnetism is another area waiting to be exploited.

Many people have played a role in this success story. At BNL we thank Andy Ackerman, Tom Dickinson, Rudy Zantopp, Gerry Shepherd and his crew, and Michael Hart, Denis McWhan, and, above all, Doon Gibbs for their support. Scott Coburn and Bill Schoenig in the scattering group never failed in their enthusiasm to help us. John Hascke from LANL made a crucial contribution and Lynne Soderholm and Steve Wasserman from ANL helped us understand our "rotten" crystals. At Karlsruhe Jean Rebizant and Franck Wastin were responsible for the crystal growth and encapsulation. The students/postdocs involved in the story from first Keele, and then later Liverpool, Universities were Chiu Tang, Debbie Jones, Sean Langridge, Bill Nuttall, Danny Mannix, Matt Longfield, and Peter Normile. We all survived!

References

*[1] F. de Bergevin and M. Brunel, Acta Cryst **A37** 317 (1981); ibid **A37**, 324 (1981)*

*[2] D. Gibbs et al., Phys. Rev. Lett. **61**, 1241 (1988); D. Gibbs, Synchrotron Radiation News **5**, #4, p. 18 (1992)*

*[3] J. P. Hannon et al. Phys. Rev. Lett. **61**, 1245 (1988); J. P. Hill and D. McMorrow, Acta Cryst. **A52**, 236 (1996);*

*[4] D. F. McMorrow, D. Gibbs and J. Bohr, in "Handbook on the Physics & Chemistry of Rare Earths", edited by K. Gschneidner and L. Eyring, Elsevier Science, Amsterdam, 1999, Vol. **26**, p. 1*

*[5] E. D. Isaacs et al., Phys. Rev. Lett. **62**, 1671 (1989); D. McWhan et al., Phys. Rev. B **42**, 6007 (1990); D. McWhan, J. Alloys & Cmpds. **271-273**, 408 (1998)*

*[6] G. Watson et al., Phys. Rev. Lett. **77**, 751 (1996); Phys. Rev. B **61**, 8966 (2000)*

[7] J. C. Spirlet and O. Vogt, in "Handbook on the Physics and Chemistry of the Actinides", edited by A. J. Freeman and G. H. Lander, North-Holland Physics, Amsterdam, Vol. 1, p. 79, (1984)

*[8] W. G. Stirling and G. H. Lander, Synchrotron Radiation News, Vol. 5, #4, pp 17-21 (1992), S. Langridge et al., Europhysics Lett. **25**, 137 (1994); Phys. Rev. B **49**, 12010 (1994); ibid **49**, 12022 (1994)*

*[9] E. Lidström et al., Phys. Rev. B **61**, 1375 (2000)*

*[10] S. Wasserman et al., J. Alloys and Compounds **271–273**, 882 (1998)*

*[11] D. Mannix et al., Physica B **262**, 125 (1999)*

*[12] L. M. Sandratski, Adv. in Physics **47**, 91 (1998); H. Yamagami, Phys. Rev. B **61**, 6246 (2000); K. Knöpfle and L. M. Sandratskii, Phys. Rev. B **63**, 014411 (2001)*

*[13] D. Mannix et al., Phys. Rev. B **60**, 15187 (1999)*

*[14] N. Bernhoeft et al., Phys. Rev. Letters **81**, 3419 (1998)*

[15] P. Normile, Thesis, Univ. of Liverpool, 2001, and to be published

New Rules for Transport of Hazardous Materials

Andrew Ackerman, NSLS

The Laboratory is working on clarifying the rules for transport of hazardous and radioactive materials. A group was formed to develop guidelines for both on- and off-site movement of these materials and their recommendations will soon become effective. Those involved in moving materials for an experiment are to know and follow these requirements for transport. There are requirements for both shipping and for personally driving chemicals and radioactive materials to, around, and off the site.

The NSLS experimental program is a significant contributor to the volume and variety of materials moved around and off the laboratory site. Users bring many materials that are subject to Department of Transportation (DOT) restrictions and the new guidelines are designed to help everyone comply with those rules when packaging and transporting chemical and radioactive materials needed for an experiment at the Light Source.

While the details are still being resolved, the basic policies have been described and they are sensible for our operations. Users can move small quantities of materials in personal vehicles as long as the transporter knows and meets the DOT, "Material of Trade (MOT)" rules for packaging and labeling. The MOT rules are easy to follow and help simplify transport of experimental samples and many common materials for Users traveling to BNL by car. For those who come by air, and for materials classified as more hazardous, transport must be handled by a commercial carrier and requires coordination with your facility shipping people or with the BNL shipping staff. The guidelines were developed to help determine which requirements are to be used. They can be a valuable reference for use when planning an experiment to identify the transport requirements applicable to individual circumstances.

Efficient movement of equipment and materials has obvious importance to a User facility. The new rules are well organized. They will help improve our efficiency and help us meet our obligation to keep the NSLS experimental program at minimum risk to incident and in compliance with regulation. More information will be available soon.

Facility Report

Gerry Van Derlaske

Here is a quick overview of some of the events transpiring in and around the NSLS Complex this spring.

Movement of LS Users from Bldg. 510-E: Recently, vacated quarters in Bldg. 535, located within the confines of the Instrumentation Division have opened up. LS Users are being moved as space becomes available into ground level offices found in the North Corridor and the Modular Building of 535A. Upon completion of this move, over thirty offices and their occupants from the third floor of Physics Bldg. 510 will have been transferred to this new location.

Acquisition of Bldgs. 820A & 820B: 820A-Physics and CAD personnel are in the process of vacating the 820A facility. Upon transfer of ownership and NSLS occupancy of this area, cages will be constructed for the storage of departmental equipment. 820B-An exit review was performed to address legacy issues prior to full transfer and ownership to the ATF Facility of a building for storage purposes. As punch-list items from the Exit Review are addressed, we move closer to occupancy of this much needed space.

New Custodial Supervisor/ NSLS Carpenter: After 9 years of dedicated service to our department, Muriel Olenick, area Custodial Supervisor, has been named General Laboratory Custodial Supervisor. Muriel has been promoted to fill the slot vacated by Oscar (Slim) Blevins, who has retired after 37 years of Laboratory Service. Debbie Doyle has been named to fulfill the duties to our department previously held by Muriel. We wish Debbie, Muriel and Slim the best of luck in their new endeavors.

Bill Gildersleeve, our resident carpenter, has been placed on light duty arising from complications of a nagging back injury. Paul Humbert has been assigned to carpenter duties within the NSLS Complex. For any Custodial or Carpenter needs, please schedule your requests via myself at ext. –3476, or Bob Kiss, ext. –4926.

Monitoring Wells: The placement of approximately 22 Tritium monitoring wells began the week of June 4th. The installation of these wells will be located on the east lawn of Bldg. 725, south of the Picnic Area, near the east concrete walkway. No interruption of egress to Bldgs. 726, 727, 728 or 729 is anticipated.

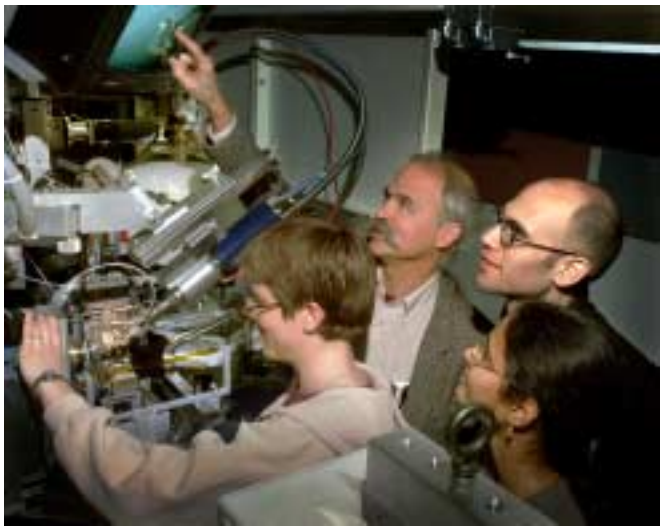
Little Flower update: All empty deposit soda cans placed in the metal recycle cans and the Blue Receptacles near the Vending machines are pick up and cashed in on a regular basis, with all proceeds going directly to the Little Flower Children's Services in Wading River. Please remember to place all recyclable cans in any of these containers, and not in the regular trash bins. Not only is it better for the environment, you are helping out someone less fortunate than yourself. Congratulations to Nick Gmur and Johnny Kirkland, who collected enough cans to make a recent donation to Little Flower of over $275.00!!!

Course in X-Ray Crystallography Draws Students from Around the World

Karen McNulty Walsh
BNL Public Affairs

The National Synchrotron Light Source (NSLS) was extra busy the week of April 23-27 as a group of 48 students from around the world participated in a week-long, hands-on training course. "Rapid Data Collection and Structure Solving at the NSLS: A Practical Course in Macromolecular X-Ray Diffraction Measurement" was developed by the Biology and NSLS Departments to introduce students to the best people, newest equipment, and latest techniques in the field of x-ray crystallography. "There's probably no better experience than this for someone who's trying to learn how to use a synchrotron and how to use the software," says participant Mark Walter, an associate professor of chemistry at Oakton Community College in Des Plaines, Illinois. Walter last did crystallography work when earning his Ph.D. at Northwestern University. Like a Rip Van Winkle who¹s been asleep for eight years while the field has advanced dramatically, he wanted to update his skills and possibly set up a similar hands-on learning experience for his students. Like Walter, half the students

Students and teachers participating in the x-ray crystallography course gather outside the NSLS.



At BNL's National Synchrotron Light Source, looking at equipment that is being used for the recent week-long course are: (clockwise, from left) Emma Jakobsson from Uppsala University, Sweden; Bob Sweet of the Biology Department, who designed and helped run the program; Oren Rosenberg from Rockefeller University & Yale University; and Srikripa Devarakonda from the University of Virginia.

came to the BNL course to observe and learn everything they could about x-ray crystallography. The other half came with specific projects in mind, such as solving the structure of a particular enzyme. "Half a dozen of them are leaving with a publishable result," said Bob Sweet of BNL's Biology Department, who designed and helped run the program. The students started their week with two days of lectures and tutorials on the techniques used to prepare crystal samples, and the software and other tools needed to collect and analyze data. These talks were given by BNL scientists, including Sweet, John Skinner, Dieter Schneider, Lonny Berman, and Howard Robinson, and other experts chosen from industry, academia, and other national labs. "We recruit people who we think are leaders in the field," says Sweet, "and they are really happy to come because they and their competitors are all together at the same time and are able to show their wares. There were several volunteer instructors from various industries, including Bram Schierbeek, who works for Bruker/Nonius, a Dutch and German company that makes a variety of crystallography equipment, including detectors and software. Says Schierbeek, "We think it's a good thing to meet people who use the equipment and see what problems they are encountering so we can use this in our design of software -- and tell them about the good things we make." The BNL scientists and these volunteers then served as hands-on scientific supervisors when the students moved to the NSLS beam lines to begin collecting data on their crystal samples. Emma Jakobsson, a Ph.D. student from Uppsala University in Sweden, particularly appreciated this aspect of the program. "Usually when you are working, you don't have somebody that can take their time to help you . . . and set up the experiment in the optimal way," she said. But in this program, "there is always someone you can ask." According to Sweet, the plan was for the students to run 24 experiments using six of the NSLS macromolecular crystallography beamlines for 60 hours straight. At about 9 p.m. on Tuesday, however, after just one group had begun to collect data, the synchrotron went down.

It took until about 1:30 a.m. to get it up and running. But the students took the delay in stride, working in groups of eight with their scientist advisors to plan how best to use their time. When last seen, they were huddled around beam-line computer terminals, clustered in conversation, and getting on with their work. "It's really nice to be able to concentrate on this aspect -- collecting data and solving structures -- which is the last part of experimentation in x-ray crystallography," said Srikripa Devarakonda, a Ph.D. student from the University of Virginia. This was her first trip to a synchrotron. And with a background in physics, she seemed genuinely excited to see how it worked, like a kid in a candy store.

BNL Science Featured at March 2001 APS Meeting

Karen McNulty Walsh and Diane Greenberg
BNL Public Affairs

More than 30 BNL scientists presented their research at the March 2001 American Physical Society (APS) meeting held this week, March 12-16, at the Washington State Convention Center in Seattle, Washington. These stories feature a sampling of the promising and varied research reported at the meeting.

[Editor's Note: The following research was performed in part or in whole at the National Synchrotron Light Source.]

Revealing the Secret of High-Performance Transducers

Beatriz Noheda, Physics Department, reported on new advances in the study of piezoelectric Materials -- materials that can be deformed by the application of an electric field, or that produce an electric current when physically deformed. One of the most important piezoelectric materials is a ceramic known as PZT. It is used as a transducer for transforming the vibrations of sound waves, for example, into electrical current and vice versa in devices such as telephones, sonar systems, and ultra-


Beatriz Noheda

sound machines. Noheda described the discovery of a previously unknown phase, or crystalline shape, for certain compositions of PZT, which explains their very high piezoelectric response. "With this new 'monoclinic' phase, you no longer have to apply the electric field in the exact direction of the deformation. This material has a lot more freedom to deform," Noheda says. Scientists may now look for this monoclinic phase in other materials and use them as well as PZT to make the next generation of solid-state transducers, which could result in much more sensitive devices. This work was done at the National Synchrotron Light Source.

Exploring Electronic States in High-Temperature Superconductors

Tonica Valla, Physics Department, presented his group¹s latest efforts to understand the underlying mechanism for superconductivity in copper-based materials - cuprates - that act as high-temperature superconductors. Like traditional superconductors, these materials carry electrical current with no resistance while in their superconducting state. But Valla's studies at the NSLS reveal that they do not use the same mechanism. In both traditional and high-temperature superconductors, pairs of electrons carry the electric current, but the "glue" that holds the pairs together may be different. Valla's experiments give direct information about electronic states in these materials and can uncover the interaction that causes pairing of electrons. The new materials become superconducting at warmer temperatures than do conventional superconductors, which must be kept super cold by surrounding them with expensive liquid helium. Cuprates, however, are superconducting at temperatures "warm" enough to be chilled by less-expensive liquid nitrogen. "If we understand how these high-temperature superconductors work, then we might be able to make them more efficient so that they can take the place of the more expensive kind in magnets for accelerators, electronic circuits, or even more exotic applications as superconducting railroads and motors," Valla says.

Using New X-ray Technique to Improve Breast Imaging

Zhong Zhong, National Synchrotron Light Source (NSLS) Department, and North Carolina State University researchers Miklos Z. Kiss and Dale E. Sayers are investigating a new technique called diffraction-enhanced imaging (DEI) to detect and study calcifications of breast tissue. As Kiss reported at the APS meeting, using DEI, the collaboration looked at a sample of breast tissue with at least ten calcifications and made computer models of the new imaging process to study its contrast mechanisms. This new method, compared to x-rays used in mammography, significantly improves pictures of breast tissue. Calcifications are associated with breast cancer, and their early detection is crucial for diagnosis and treatment. DEI was developed and tested at the NSLS by researchers from BNL, the Illinois Institute of Technology, North Carolina State University, and the University of North Carolina. DEI reduces the x-ray scattering that makes for blurry images and lack of contrast in mammograms. The new patented method may one day replace mammograms.


Tonica Valla


Zhong Zhong

NSLS User Administration Office
Brookhaven National Laboratory
NSLS Building 725B
P.O. Box 5000
Upton, NY 11973-5000

Important Upcoming Dates

September 1, 2001	Deadline for submissions, November Newsletter
October 1, 2001	Deadline for General User Proposals (Jan-Apr 2002)
October 1, 2001	Deadline for Beamline Progress Reports
October 1, 2001	Deadline to submit Publication References for FY2001 (The system doesn't close!)
October 1, 2001	Deadline to submit title for Activity Report science highlights
October 31, 2001	Deadline to submit Abstracts for FY2001 (Submit soon! The system is always open!)

Call for NSLS General User Proposals

For Beam Time in Cycle	Deadline
January - April 2002	Monday, October 1, 2001

General User Proposal and Beam Time Request Forms including instructions (and PX forms) can be found at URL:
http://nslsweb.nsls.bnl.gov/nsls/users/procedures/proposals.htm
Proprietary Proposal Forms including instructions can be found at URL:
http://nslsweb.nsls.bnl.gov/nsls/users/procedures/proposals-prop.htm

The *NSLS Newsletter* is published tri-annually by the National Synchrotron Light Source Department, Brookhaven National Laboratory

Mary Anne Corwin, Editor
Nancye Wright, Production Assistant

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

http://nslsweb.nsls.bnl.gov

NSLS User Administration Office
General Information, User Registration, Training:
Phone: (631) 344-7976 Fax: (631) 344-7206

User Administrator
Mary Anne Corwin corwin@bnl.gov
Annual Users' Meeting
Lydia Rogers lrogers@bnl.gov
General User Proposals
Gretchen Cisco cisco@bnl.gov
Lydia Rogers lrogers@bnl.gov
Publications/Newsletters
Nancye Wright wright1@bnl.gov