MARCH 1999 NSLS Newsletter

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Resonant X-Ray Scattering from Liquid Crystals

R. Pindak (Bell Laboratories)

P. Mach (U. of Minnesota)

A.-M. Levelut (Laboratoire de Physique des Solides, Univ. Paris-Sud)

P. Barois (Centre de Recherche Paul Pascal, CNRS)

C. C. Huang (U. of Minnesota)

L. Furenlid (NSLS, current address: U. of Arizona)

The existence of a helical symmetry axis is widespread in systems exhibiting liquid crystalline order, especially in systems comprised of chiral molecules. Because these systems usually lack three-dimensional positional order, the helical symmetry axis cannot be observed using conventional x-ray scattering. It is well known that in crystalline systems, resonant x-ray scattering can be used to observe such `hidden' symmetries [1]. In our recent research at NSLS, we demonstrated that polarization-analyzed resonant scattering could also be applied to observe the "hidden" helical symmetry axis in liquid crystals [2]. Since the nature of the helical ordering impacts the electro-optic response of the liquid crystal phase, it is a crucial structural feature to establish.

The liquid crystal systems which we studied are the chiral smectic-C (SmC*) phases. In these phases, the rod-shaped molecules form a stack of fluid-like layers. Within each layer, the molecules, on average, tilt in the same direction. Moreover, the local symmetry environment is consistent with a hindered rotation of the molecules about their long axes resulting in a spontaneous local polarization parallel to the layer plane and perpendicular to the tilt plane. The different SmC* phases are distinguished by different interlayer sequences of changes in the molecular tilt direction. This interlayer orientational order of the molecular tilt varies from the molecules in each layer being tilted in the same direction (SmC* phase) to the molecules in alternate layers being tilted in opposite directions (SmC_A* phase). Since this interlayer orientational order also applies to the local spontaneous polarization direction, the electro-optic response of the phases varies, respectively, from ferroelectric to antiferroelectric. Intermediate between these extremes are SmC* phases that exhibit a ferrielectric response (SmC_{FI1}*, SmC_{FI2}*, and SmC_{alpha}* phases). Although these intermediate SmC* phases had been utilized in high-speed displays and tristable optical switching devices, their detailed structure remained unconfirmed.

To elucidate the structure of these intermediate SmC* phases, we worked with thiobenzoate and thiophene liquid crystal compounds which, as shown in Figure 1, contained a sulfur atom in their cores. Utilizing a two-crystal Si(111) monochromator on beamline X19A, we tuned the x-ray energy to the

sulfur K-edge at E_O=2.48 KeV and did specular diffraction from free-standing films. When the energy of the incident x-rays is close to the absorption edge of the sulfur atom, the excitation of core electrons to the molecular orbitals results in a tensorial atomic scattering factor. The polarization state and the phase of the scattered x-rays depend on the orientation of the local environment of the scattering sulfur atom, and therefore, helical modulation of the orientational order can result in diffraction peaks forbidden in conventional diffraction. Moreover, the polarization state of the diffracted x-rays can differ from that of the incident x-rays. The tensorial atomic scattering factors for crystalline helical symmetry axes were calculated by Dmitrienko[3].

RESONANT SCATTERING PEAKS REVEAL SHORT-PITCH HELICES

By changing the temperature of the liquid crystal films, we observed the resonant scattering features associated with each of the SmC* phases [2]. We discovered that the scattering was consistent with a helical interlayer orientational ordering with a short pitch = nu d where d is the layer spacing and 2pi/nu is the rotation angle between successive layers. Levelut and Pansu [4] extended Dmitrienko's analysis to incommensurate helical structures as well as commensurate, but non-helical, orientationally ordered structures. Their analysis demonstrated that a helical structure of pitch = nu d results in resonant peaks at $Q_Z/Q_O = l + m\ (1/nu)$ where Q_Z is the scattering wavevector normal to the layers, Q_O = 2pi/d , *l* and m are integers. Moreover, because there are only five independent terms in the tensorial atomic scattering factor, $-2 \leq m \leq 2$. The evolution of the helical pitch in the different SmC* phases can be seen in Figure 1 which shows the non-integer resonant peaks associated with each phase. It is important to note that all of the non-integer peaks appearing in Figure 1 vanish when the x-ray energy differs from the sulfur resonant energy by more than 20 eV.

Starting with the lowest scan in Figure 1, taken in the SmC_A phase of the non-chiral racemate of the thiobenzoate compound, half order peaks were clearly observed. This indicates a commensurate 2-layer helical pitch. In the chiral SmC_A* phase, the molecular chirality induced an additional small rotational change per layer making the helical structure incommensurate and splitting the half order peaks. In the ferrielectric phases of the thiobenzoate compound, one-third order peaks were observed in the SmC_{FI1}* phase while, in the SmC_{FI2}* phase, quarter and half order peaks were observed. These observations indicated, respectively, 3-layer and 4-layer pitches. In the SmC_{FI2}* of the thiophene compound, the half order peak was observed to split due, again, to an additional small rotational change per layer. At higher temperatures, in the thiobenzoate compound, a phase transition into the SmC_{alpha}* phase occurred. This phase exhibited an incommensurate pitch, which varied between 5 to 8 layers with increasing temperature. Finally, in the SmC* phase of the thiophene compound, first and second order satellite peaks (m = ±1 and ±2) were observed on either side of the second order layer peak. The spacing between these peaks was consistent with a helix having a 290 nm pitch.

HELIX INDUCED sigma arrow pi POLARIZATION CHANGE

The analysis by Levelut and Pansu [4] also predicted intriguing effects for the polarization state of x-rays diffracted from helical structures. As one example, a helical axis with nu ~ 4 would induce a sigma arrow pi change of the polarization state! To measure the polarization state of the diffracted x-rays, we used a

pyrolytic graphite (PG) analyzing crystal. At the sulfur resonant energy, the 2 theta Bragg angle for PG is close to 90° . Hence, rotating the analyzing crystal about the axis of the x-ray beam through the angle, chi_a , will diffract most of the beam if the beam is polarized perpendicular to the PG diffraction plane, but will diffract nearly zero beam if polarized parallel to this plane. The top plot in Figure 2 illustrates this for the incident beam. The incident beam was sigma-polarized and was polarized perpendicular to the PG diffraction plane when $c_a = \pm 90°$ giving maxima in the diffracted intensity. We applied a similar polarization analysis to the resonant peaks in the SmC_{FI2}* phase (lower two plots in Figure 2) by rotating the analyzer crystal about the diffracted beam. For the half order peak, the diffracted beam was s-polarized, same as the incident beam, giving c_a maxima at ± 90°. On the other hand, for the quarter order peak, the diffracted beam was pi-polarized so the maximum in chi_a occurs at 0° as predicted.

SUMMARY

Polarization-analyzed resonant x-ray scattering provides a valuable and unique probe of the orientational correlations that occur in liquid crystalline systems. These orientational correlations can be studied from Angstrom to micron length scales. In the case of the SmC* phases, the observed resonant diffraction peaks as well as their polarization states were consistent with these phases having a short helical pitch. This structure was in agreement with a clock-model of the SmC* phases which had been proposed by several authors [5]; but, inconsistent with a variety of other models, in particular the popular Ising-like models which constrain the tilt direction to a plane [6].

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Chairman's Introduction

Michael Hart

NSLS

A Linac RF modulator was destroyed by a fire in the modulator cabinet just after midnight on the morning of January 25 at the National Synchrotron Light Source. About 100 pounds of plastic capacitors burned and created a large volume of smoke that was distributed through much of the building. Burning plastic creates heavy black smoke with a complex chemical composition that includes irritating gasses and particulate. This was a typical electrical fire as it involved only common electrical equipment. There were no unusual toxic materials, no PCB oils, no nearby chemical storage, and no radioactive materials involved. The BNL emergency response and fire rescue personnel responded to the alarm, took control of the scene, and extinguished the fire. They assessed what had burned and determined that they had controlled any imminent danger hazards. Following the standard protocol the emergency response team stabilized and then released the scene to the facility once the fire was out and the smoke had cleared.

By dawn the Investigation Team had started its work and the cleanup had begun.

By the evening of Friday 29 January the Investigation Team was able to make an interim report to me, which was attended by members of the BSA Directorate and DOE Brookhaven Group. Preparation for restart was authorized after new safety measures and procedures, recommended by the NSLS Investigation Committee, were implemented. Operations were resumed at 13:00 on February 1, when beam was restored in the 800 MeV electron storage ring providing ultraviolet and infrared light for users. X-ray injection followed with stable 2.548GeV beam at 16:30 for X-Ray user experiments.

At the time of going to press the Investigation Team has not finished its task. Evidence has been collected from the scene of the fire, from staff on duty, users who were present, the emergency response team, and technical experts. Thank you all for your cooperation which is crucial if the cause of the incident is to be understood, is prevented from happening again, and if we are all to benefit from the lessons learned. We will keep users advised on the lessons learnt from the fire.

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Greetings from the New NSLS Associate Chair for ESH&Q

Bob Casey

Associate NSLS Chairman for ESH&Q

Stop-Work Procedures at BNL	NSLS Gets High Marks in DOE Audit of Chemical Handling	Transport of Hazardous and Radioactive Materials To, From and Within BNL

As a new member of the NSLS staff, I want to introduce myself to you and let you know a little about who I am. First, please call me Bob. If you look me up in the phone book or the email addresses, you will find me listed as William R. Casey. I always blame this confusion on my Mother.

I started at the Light Source on January 4. I am glad to become a part of this organization and I look forward to working closely with you in the future. The NSLS has established a good reputation for its Environmental, Safety and Health programs, and even more so for the quality of its scientific research. I feel a distinct privilege in becoming a member of the Department; and I am grateful for the very capable work of my predecessor, Bill Thomlinson.

I am not a newcomer to the Lab. I started work at BNL in 1973 and have worked in a number of different positions, including serving as the head of the Lab's Safety & Environmental Protection Division for 10 years. My professional career began in radiation protection, but I have had in-depth involvement in all ESH areas _ safety, industrial hygiene, environment, fire protection, and emergency planning.

Much of my career has been involved with large accelerator facilities, both at BNL and elsewhere. I have worked at the AGS on two separate occasions, and I was involved with NSLS in the early 1980s as an ESH representative on the initial steering committee. Over the years, I have served on DOE or institutional panels reviewing ESH programs at Fermi Lab and SLAC on several occasions. This spring I will serve on a review committee examining the Integrated Safety Management Systems at Thomas Jefferson Laboratory.

For the past year, I have worked closely with the new BSA management at BNL, and I have established a good working relationship with them. In the past several months, I have had responsibility to organize and manage a major effort to improve radiation protection performance at BNL, an assignment that will carry over for the first several months of my new assignment at NSLS.

My background and experiences will help in my new position at the NSLS. I understand the Lab's strengths and its weaknesses in its current ESH programs, and I have a good grasp of the expectations for ESH that DOE and BSA have for our performance. In the next few months, I will work hard to develop a good understanding of NSLS strengths and weaknesses in seeking to determine what the next steps should be for the NSLS ESH programs.

A major priority for me is to work closely with the research user community. I welcome your thoughts and input. Please contact me at 344-4654 or at Casey1@bnl.gov, or stop by to see me in my office at the NSLS.

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A User's Perspective

John B. Parise

SUNY @ Stony Brook

UEC Chair

This will be my last "perspective" as UEC chair and I begin by thanking those, particularly in the User Administration Office, who have been such a help over the past year. 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 Eva Rothman, Linda Feierabend, Nancye Wright, Eileen Pinkston, and Lydia Rogers. As it turned out, all these activities were far more enjoyable thanks in large part to these individuals. The UEC relies heavily on the corporate history invested in them.

After a harrowing 1997, the UEC was able to refocus on more "bread and butter" issues during 1998. The 97-98 period was dominated by uncertainty due to the change-over in administrations at BNL as well as the implications of the "Birgeneau report" (see last year's *User's Perspectives*, particularly Joel Brock's farewell of March of 1998). There are still issues arising from these events, some of which I touch upon below. While questions linger, however, the larger picture for synchrotron research is bright, with new users continuing to come to the NSLS and new areas of research continuing to open up. Nowhere is this growth more evident than in the life sciences - especially in molecular biology and protein crystallography. I urge these new users to continue to involve themselves in the UEC. As their community grows, so their beam and infrastructure requirements will evolve as well. The UEC provides a direct, and noticed, route of communication to both NSLS and BNL administrations. And as the independent representative body of the users it requires the support of a broad cross-section of them.

For those who missed it in the December 30 NSLS News e-mail, a form of weekend food service has been restored and should now be available at the Brookhaven Center (opposite the Police Station). This development is due in large part to work of the UEC in conjunction with a task force headed by Denis McWhan (BNL Associate Director and previous NSLS Chairman), and follows a couple of vigorous town meetings dealing with this subject. In response to NSLS users' requests for continued/improved food service on the BNL site during weekends, BNL Management has announced the following: "We are going to upgrade the service in Brookhaven Center, offering more food, better supervision of the area as well as of the food preparation, and an earlier start. We are also going to invest in an upgrade of the space at the Center to make it more congenial and functional as a dining place. Weekend service will be provided." As of January 9, 1999, the Brookhaven Center will have begun serving breakfast foods and sandwiches from 7:30 am until 2:00 PM, both Saturdays and Sundays. Now that this is official I trust users will take advantage of this service for weekend breakfast and lunch.

BNL Weekend Food Service

Brookhaven Center now serves breakfast foods and sandwiches from 7:30 am until 2:00 pm, both Saturdays and Sundays.

On the national level, more coordination between user groups is needed to promote synchrotron radiation in general. The "Birgeneau report" provided a platform for the synchrotron community and emphasized the importance of our science to a broader audience at DOE. Several letters to the DOE, extolling the virtues of synchrotron radiation and its importance to the nation's scientific infrastructure, were co-signed by the chairs of the NSLS, APS and SSRL user groups. More of this type of coordination will be required in the future. In response to the need for synchrotron radiation in their communities a number of science-specific interest groups formed. The last time I checked, I am involved in at least two of these, the Geosynch and HPsynch groups formed to promote the use of synchrotron radiation research in the Geo- and high-pressure communities. While these, and like-minded efforts, are aimed at informing particular sub-disciplines within the NSF, DOE, DOD and NIH, there may also be a need for a national synchrotron radiation users group to press the case for continued funding. Just such a group exists for neutron scattering, for example. We can, and must, continue to press individually to enhance the facilities for our science at the NSLS. A national organization, however, not tied to a particular source, might well be a more appropriate way to state the case for increased overall support for synchrotron radiation. The growth in importance of synchrotron radiation in a number of scientific communities has meant that many users travel to a number of specialized facilities to carry out their research. We have a special interest in maintaining the excellence of the NSLS - the most user-friendly and cost effective of the nation's sources (efficiency, though, does not seem to be one of the DOE's more important funding criteria). It is also important, however, that we band together with other UEC's to promote synchrotron radiation in general. Joint letters are one small step. Increased coordination through a national body may ultimately make the community even more visible.

The year saw the usual number of requests of NSLS users, asking for input to reviews, responses to budgetary concerns and participation in community outreach through media interviews and alike. Some of the issues addressed in these requests are still with us of course and will require our continued attention. My thanks to all those who wrote letters, who have taken time out to answer survey questions and who provided feedback on a number of these issues. Thoughtful and collegial responses to concerns can produce results. If they do not, we can always revert to outrage. Continued lobbying for our interests and reminders of our achievements will bring results. Perhaps the Long Island media could be persuaded to report on some of the more positive aspects of BNL science.

Finally, my best wishes to you all over this year and see you `round the rings.

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X-Ray Ring Report

Roger Klaffky

X-Ray Ring Manager

Since the last NSLS Newsletter status report there have been reliable 2.8 GeV operations preceding the December shutdown, and the start-up of 2.584 GeV operations on January 19 with the low emittance lattice. This report will primarily focus on the tasks completed during the December 1998 shutdown.

The major task planned for the shutdown was the replacement of the existing XRF1 cavity with a second all-copper cavity. The new cavity was delivered to BNL on September 23, 1998, and transported to the RF Test Area for frequency calibration, mode damping measurements, and high power conditioning and testing. The front cover seal and port seals were found to be vacuum tight. After baking the cavity, it was necessary to use a smaller cross-section seal and a 3.5 mm thick shim to achieve the proper cavity resonant frequency (52.987 MHz). Initial low level rf conditioning in early November revealed many multipactoring levels. The cavity input loop and window was successfully tested to 140 kW input power for 8 hours. To reduce low level multipactoring levels, a bake was carried out before further rf conditioning. After baking the base pressure was higher than expected and helium leak checking revealed a leak from the mushroom water cooling channels into the cavity. After meeting with ACCEL representatives the decision was made to ship the cavity back to Germany in December for careful inspection. Members of the NSLS engineering staff visited ACCEL during the week of January 24-28 to examine options for repair or replacement of the defective mushroom. At the ACCEL facility visual inspection revealed a 20 cm long band on the inside surface of the hollow mushroom stem with markedly enlarged grains. This indicated that this area had experienced elevated temperatures at or above the recrystallization point. During a visit to an ACCEL subcontractor KME, the supplier of the RF cavity copper forgings, it was suggested that the failure occurred during the final machining of the mushroom by the improper adjustment of a "steady rest" which caused local heating in the area of the enlarged grain size and the vacuum leak. ACCEL confirmed that this was a strong possibility. Agreement was reached with ACCEL to remove and replace the defective piece. The cavity should be shipped back to the NSLS at the end of the summer, in time to prepare it for installation during the December 1999 shutdown. One RF task that was completed during the December shutdown was the addition of a coaxial line to equalize the output impedance of the two 125 kW amplifiers in the XRF2A and XRF2B hybrid transmitter system. This will increase the system efficiency.

As reported in the November NSLS Newsletter, the new Oxford Instruments X17 superconducting wiggler was returned to Oxford for repair of a leak between the nitrogen and helium vessels and a short in the power leads. At the present time, the problems have been isolated and repairs are underway. Oxford expects to complete the repairs and re-test the magnet before shipping it in early spring. After

receiving the magnet, the NSLS will conduct acceptance tests prior to installation in the December 1999 shutdown.

The next upgrade in the X-Ray Ring operations parameters will be a boost in the maximum allowable operating current to 438 mA at 2.584 GeV (or to 300 mA at 2.8 GeV). Before this could occur a life cycle test had to be completed on the dipole magnet exit chamber crotch radiation absorber. A life cycle test was devised utilizing an electron beam from an EBW welding machine to simulate the thermal loading from the X-ray beam. A crotch was subjected to 8000 thermal cycles to simulate a 20 year life. An additional 1000 cycles at a 20% higher power level have been recently completed to demonstrate a suitable safety margin. The test crotch will be carefully examined for microscopic cracks. There is no damage evident to the eye. Upon recommendation of the Thermal Limits Task Force and approval by NSLS management, operation at increased currents can commence.

Over the last year there has been a concerted effort to identify and reduce sources of floor vibration. The vibrations have adversely affected the VUV Ring IR beamlines, X1A, and X16C. Prior to the shutdown, the replacement of isolation springs under concrete pads supporting the MER2 aluminum and chilled water pumps led to a substantial reduction in 59.5 Hz floor vibrations. The water cooling pumps for the RF3 and RF4 cavities were also found to be introducing 58.5 Hz vibrations into the X-ray and VUV beamline floors. During the December shutdown, spring mounts were installed for these pumps and flexible couplings were installed on the inlet and outlet lines for both of these pumps. These improvements have substantially reduced the 58.5 Hz vibrations.

A substantial amount of work took place on the NSLS water systems. Controls were installed on the biological cold rooms so that a switch-over to domestic water cooling of the cold room compressors will automatically occur upon a disruption of water from the BNL Central Chilled Water Facility. Also, the cold box dehumidifier controls were modified so that a shutdown of the compressors will turn off the heat sources in the cold rooms (dehumidifier, lights, etc.) to prolong the time before they warm up. Another major job was the transfer of the LEGS magnet supply and target cooling from the low pressure copper system to the experimental water system. In the future, the LEGS magnet cooling will also be transferred from the high pressure cooling system to the experimental water system. Valves were installed to prepare for this change. Other water system work included the installation of a pressure control valve for the experimental water system, of additional experimental water spigots (at X19, X20), and of new expansion tanks for all water systems.

Prior to the shutdown, the X1 Horizontal Active Interlock system was commissioned so that water cooling would not be necessary for the X1 insertion chamber "speed bump". Monitoring of the pump pressures in the vicinity of this component and of the X1 straight section bremsstrahlung radiation had suggested that a small water-to-vacuum leak had developed. During the shutdown the water was drained from the speed-bump cooling channel, which was then pumped out and back filled with an atmosphere of helium for the start-up of operations in January. When operations resumed, the bremsstrahlung radiation from the X1 straight had decreased by a factor of 5, suggesting that the water channel had been leaking. A leak check performed in late January with the water channel pressurized to 20 psi of helium clearly confirmed the leak. A more severe leak had developed a year earlier in the X17 insertion device chamber speed-bump. Unlike the X1 speed-bump, it had to be replaced because it constantly intercepts high power X17 wiggler radiation, thereby protecting front-end components further downstream. In the case of X1, the active interlock system now provides this protection.

There was substantial progress towards rendering all X-ray front end fast valves operational. Fast valve electronic systems have been installed on all front ends except X12, X27, and X28. Eighteen of the 28 front ends now have operational systems (X1, X3, X4, X5, X7, X8, X9, X10, X11, X13, X14, X19, X20, X21, X22, X24, X25, X26) utilizing front end sensors to trip the valves. Of these, eight have molybdenum blades which remove the necessity of dumping the ring when the fast valve fires. Four of the Phase I front ends having stainless steel blades (X15,X16,X18,and X23) require either a molybdenum blade or a modification of the beamline interlock system so that the ring will dump if the fast valve fires. Other interlock work included a testing of the global alarm system for critical devices cooled by aluminum water. Individual heat sensors on each component were heated to ensure that the system trips.

There was a complete check of the X-Ray Ring bremsstrahlung shielding configuration. Lead blocks were restacked as necessary and all shielding was banded. Photographs of each shielding arrangement were taken and placed in the vicinity of the shielding. Each front end area was tagged indicating that work permits are required before shielding is removed.

Another future upgrade will be the implementation of the low emittance lattice during 2.8 GeV operations. Operations at 2.584 GeV with the low emittance lattice commenced last September. Before implementing the lattice at 2.8 GeV several tasks must be accomplished. A 500 V, 400 Amp sextupole magnet power supply must be replaced with a 700 V, 1000 Amp supply. A specification was written for a new supply and bids were solicited in the middle of January. During this shutdown a plan for running new higher current cables and cable trays was developed in order to install cable during the Spring 1999 shutdown. Before this work proceeds, temperature measurements will be performed on a test sextupole magnet at higher currents to ensure that it has adequate water cooling.

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FOCUS ON.....NSLS Experimental Systems Group

Erik D. Johnson

Group Leader - Experimental Systems Group

Project Manager DUV-FEL

The Experimental Systems Group was created a year ago in a merger of the Beamline R&D section with the Long Range R&D group. It came about as a consequence of changes in roles for their respective group leaders. Peter Siddons was preparing for a sabbatical leave that he is now enjoying at the ESRF, and the Laboratory needed to call on the expertise of Jerry Hastings to help in the area of neutron science. The timing for the reorganization also seemed propitious, since on March 1st 1998 Brookhaven Science Associates formally took over management of the laboratory.

While it would be tempting to make broad claims of novelty for the ESG organization, its core mission remains essentially the same as it was when the NSLS project created a beamline group nearly twenty years ago. What has changed, is that we now have it (and other useful information) available on the ESG web site (http://www.nsls.bnl.gov/ESG/ESGFrontPage.html).

There are three major aspects to our mission

Developing new capabilities to maintain the facility at the state-of-the-art;

Encouraging new user communities from all fields to use the NSLS capabilities;

Operating key facilities for the benefit of the general user community.

The strategy to achieve these goals is also embedded in the history of the group and the Department. We participate in programs at a number of beamlines on both storage rings. Some are aimed at serving specific user communities with well-defined technical requirements. Some are configured to be more flexible to allow the development of new instruments, techniques, and diagnostics. Others are allocated for development by the group with the aim of promoting the growth of new user communities. We share our expertise with the general user and PRT communities both to aid them in their research, and to broaden our knowledge of activities in synchrotron radiation research. We also participate in development projects for new sources and instruments that may ultimately be beneficial to the NSLS users of the future.

All of these activities require talented, creative and well-recognized staff. To attract these individuals to the NSLS, we make resources available for them to conduct research relevant to the missions of the NSLS. Their successes are then translated into state-of-the-art resources for the NSLS user community.

Our role in the various beamlines can be described in four capacities:

#Beamlines	Capacity

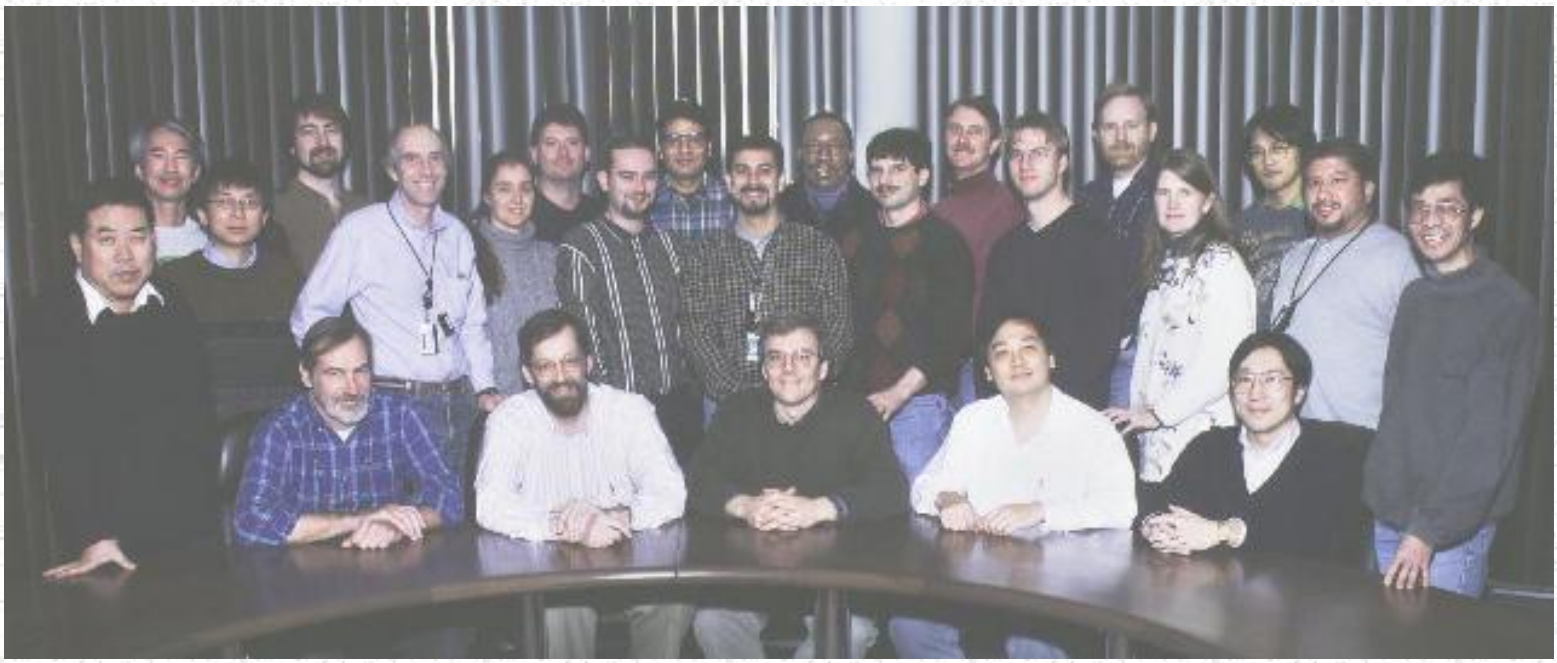
12 **Steward**; ESG assumes full responsibility for operation and maintenance.

13 **Participant**; ESG has membership as part of a PRT.

10 **Advocate**; NSLS is attempting to promote the use of a beamline.

4 **Custodian**; ESG maintains the beamline for the NSLS (e.g. diagnostics).

In all, the 39 beamlines mentioned in the inset have the attention of a staff comprised of 12 scientists, 5 science associates, 3 engineering professionals, 7 technical specialists and 1 administrative secretary. Currently working with us are ten post-docs, students, and visitors partially supported by the NSLS, who make a strong contribution to the intellectual environment. In the last year we also started joint appointments with scientists in other BNL departments. At present they are Louis DiMauro (Chemistry) and John Sutherland (Biology). In addition, we participate in the scientific life of the laboratory through collaboration with other departments and institutions. There is of course other glue that holds the enterprise together. The NSLS is a matrix organization, and staff from other sections in the Department support many of our activities (particularly construction and special projects).



Standing, left to right: Qing-Li Dong, Shu Cheung, Zhijian Yin, Phil Pietraski, Gwyn Williams, Cecilia Sanchez-Hanke, Dennis Carlson, James Ablett, Syed Khalid, Paul Montanez, Skip Thomas, Elio Vescovo, Gary Nintzel, Dieter Lott, Barrett Clay, Michelle Ramotowski, Hayashi Hisashi, Clay Carle, Edson Kakuno.

Seated, left to right: Tony Lenhard, Erik Johnson, Steve Hulbert, Ivan So, and Chi-Chang Kao.

Absent from photo: Jerry Hastings, D. Peter Siddons, Lonny Berman, Larry Carr, Jaehoon Park, Zhong Zhong, Betsy Dowd, Louis DiMauro, John Sutherland, Hyun-Jo Kim, Alex Weiss, Wan Chan Zhang, Anthony Balon, Roger Hoeft, Rick Greene, Gerry Van Derlaske, Pamela Ciufo.

In the past year, there have indeed been many changes at BNL and the NSLS. With the advent of BSA we have tried to organize the ESG to be attuned to the new management culture of the laboratory. Department wide, there has been some turn-over in staff which we view as an opportunity to strengthen our capabilities. What has not changed has been the commitment of the ESG staff to make the NSLS a scientifically productive facility through a cooperative relationship with our user community.

It doesn't matter how new an idea *is*,
What matters is how new it *becomes*.

-Elias Canetti

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MARCH 1999 NSLS Newsletter

Synchrotron X-ray Microbeam Diagnostics of Combinatorial Synthesis

E.D. Isaacs ***(Bell Laboratories)***

G. Aeppli ***(NEC Research)***

X.-D. Xiang (LBNL, Materials Sciences Division)

Combinatorial synthesis has the potential for revolutionizing a broad range of disciplines from biotechnology[1] to material science[2,3] because of the efficient and systematic way in which it searches for new materials. Application of combinatorial techniques to making libraries of solid-state thin films is of particular current interest because so much of modern electronics is thin film-based. In such libraries, each member can be individually addressed by adaptations of standard characterization techniques, such as transport and photoluminescence, potentially making trial-and-error discovery of new materials very efficient.[2] Whether combinatorial techniques can yield useful electronic materials ultimately depends on the accurate and rapid characterization of up to thousands of films. However, because of small sample volumes most characterization has been limited to bulk powder samples prepared by standard solid-state synthesis to replicate selected combinatorial films. In response to this challenge we have adapted recently developed synchrotron-based x-ray microprobe techniques (spot size = 2 x 20mm^2) to directly characterize individual films of as-grown combinatorial libraries. In particular, we have used the x-ray microprobe to characterize a library of rare earth activated tricolor phosphors.[4] Using x-ray fluorescence, diffraction and near-edge x-ray absorption fine structure we establish the composition, structure, and valence state of the Eu activator which determines the color of the phosphor. The key result is that the films are primarily single phase, despite the large compositional spread. However, there is also evidence for a cubic epitaxial phase.

Combinatorial Synthesis of Phosphor Films

Oxide-based phosphors, such as Y_2O_3:Eu^{3+},[5] have become important in advanced display technologies such as projection TV because they are stable, relatively easy to synthesize, and can have high quantum efficiencies. However, the useful number of phosphors available for displays is quite limited. Recently, combinatorial techniques have been used to search for new rare-earth activated $Gd_xGa_yO_{3-delta}$:RE phosphors. This search lead to the discovery of a new red phosphor with good color saturation at the desirable wavelength of 621 nm and with the high quantum efficiency of 85% competitive with Y_2O_3:Eu^{3+}, an industry standard.[6] In order to directly evaluate the quality of this material, we examined a library containing the new red phosphor (inset in Figure 1). The library was fabricated on a polished Si(100) single crystal substrate. A sequence of rf sputtering sources (e.g., Ga_2O_3, Eu_2O_3, Y_2O_3, Gd_2F_3 and ZnO), masks and annealing steps were used to generate a 1024 member libraries of 1 mm thick thin films each with lateral dimensions 0.7x0.7 mm^2. Two annealing steps were required; an extended period

of low temperature post-anneal (100 °C - 400 °C) to ensure uniform mixing of the sputtered layers and a subsequent high temperature anneal (T>1100 °C) allowed for the formation of a single phase. Even so, some minority phases were possible due to inadequate mixing of the sputtered layers, as was observed in earlier generation combinatorial libraries, [7] or substrate interactions. As we now show, x-ray microbeam is an ideal analytical tool to identify and characterize such minority phases in solid state combinatorial libraries.

Synchrotron X-ray Microbeam

Our measurements were carried out on the x-ray microprobe (XMP) at the Bell Labs beamline X16C at the National Synchrotron Light Source (NSLS).[8] The principal components are a horizontally collimating, double Si(111) crystal monochromator with an energy range from 5 - 20 keV, a pair of glancing incidence, elliptically bent mirrors[9] for focusing independently in the vertical and horizontal, respectively, and a two-circle diffractometer with micron XYZ sample positioning capability. The mirrors produce a spot size on the sample of approximately 2 mu m x 20 mu m with a flux of 10^8 photons/sec at 8 keV. This represents a gain in flux of 65 over a pinhole with the same area. The composition of each phosphor film was determined by measuring the x-ray fluorescence (XFS) spectra with an energy dispersive solid-state detector with approximately 250 eV energy resolution at 8 keV. X-ray diffraction (XRD) was performed by scanning the angle of the sample (Theta) and the position of the detector (2Theta). In order to determine the valence state of the Eu activator atom, we used near-edge x-ray absorption spectroscopy (NEXAFS). All three techniques were applied to nominally the same location on the film.

X-ray Microanalysis - Structure

Structural characterization with x-rays is crucial in evaluating the product of any materials growth process. It is particularly important when considering the large number of phases possible given the large set of sputtering targets, masks and annealing steps[4] used in the combinatorial synthesis of a solid-state library. For instance, since all of the films on a library chip are typically processed identically, it is important to ask whether more than a few of the films will yield the desired single phase. A partial answer to this question can be seen in Figure 2 in which we show x-ray diffraction data for six films from the library shown in the inset in Figure 1. All but two of the significant Bragg peaks on each of these films corresponds to the Gd_2O_3 C2/m monoclinic structure. This is the target host structure which along with the correct rare-earth ionization state (see below) gives rise to red or blue emission. The variability of the relative intensities of the Bragg peaks, and missing Bragg peaks, were attributed to the small number of grains within the focused x-ray spot. The strong reflection at 1.366 Å is the Si(400) substrate peak which served as a fiduciary for d-spacing. The single, bright peak at 2.722 Å in most of the films cannot be ascribed to the monoclinic structure. It may be due to epitaxial growth of cubic Gd_2O_3 which has almost exactly twice the Si(400) d-spacing (Si(200) is forbidden). A theta-rock (fixed 2Theta) shows this peak to be resolution limited giving evidence for its' epitaxial origin. In contrast, the monoclinic grains are textured, showing several discrete peaks with an angular spread of ~2° within the 2 x 20 mu m^2 spot. Many of the other films examined by XRD, which are not shown here due to lack of space, also showed the desired C2/m monoclinic host structure.

X-ray Microanalysis - Composition

Figure 3 shows x-ray fluorescence data for the red phosphor film labeled (20,8) which we examined with XRD above (see inset in Figure 1). The highest energy peak is due to the elastic scattering of the 11 keV incident x-rays from the thin film and substrate. Strong fluorescence is observed from Gd (L alpha at 6.057 keV and Lb at 6.713 keV), Ga (K alpha at 9.252 keV and K Beta at 10.264 keV) and Zn (K alpha at 8.639 keV). In contrast, the emission from the relatively low concentration of 3% Eu (E_{La} = 5.846 keV), is almost entirely obscured by the Gd. Eu emission could be observed by tuning the incident energy to 7.000 keV where the Gd L emission lines were not excited. To quantify the sample compositions we solved four coupled equations which connected the observed XRF intensities (obtained with gaussian fits to the data) to the concentrations of the elements Gd, Ga, Zn, and Eu. The equations included sample absorption, which itself depends on the concentration of each element, and air absorption. The results were normalized so that $N_{Gd} + N_{Eu} + N_{Zn} \equiv 2$ for the expected Gd_2O_3 monoclinic structure (see above), where N_i is the concentration of the i^{th} element. The composition of film (20,8) is $Zn_{0.36}Gd_{0.52}Ga_{1.12}\ O_{3-d}:Eu^{3+}_{0.03}$.

X-ray Microanalysis - Valence State

Since the color of the refractory oxide metal phosphors are determined by the energy levels of the rare earth activator, which depend on the crystal fields and valence bands of the host material, we used NEXAFS to determine the valence state of the Eu atom. Figure 4 shows NEXAFS data for the same red phosphor film (20,8) examined above. These curves show the characteristic "white line" peak at the threshold for absorption which at the L_{III}-edge is due to the excitation of an electron from the Eu 2p core levels to the unoccupied 5d-bands. The position of the white line peak is characteristic of the Eu^{3+} valence state. This valence state, in the environment of the monoclinic structure as observed by XRD, is what gives rise to the red color under broad-band UV illumination.

We have demonstrated the use of an x-ray microprobe to characterize a combinatorial library of perovskite based phosphors. Utilizing the small spot size and the sample positioning capabilities of our microprobe, we have characterized individual films with x-ray fluorescence spectroscopy, x-ray diffraction and near-edge x-ray absorption fine structure spectroscopy. The XRF data are used to measure the composition. The XRD data show that the combinatorial technique does indeed produce the desired Gd_2O_3 monoclinic host structure for the rare earth activated phosphor for many of the films in a single library. At the same time, the XRF show unambiguously that an additional, possibly epitaxial cubic phase coexists within each film.

The Future

Combinatorial synthesis is a technique with tremendous promise for new materials discovery. Progress within the last year has been so rapid that new compounds are being synthesized at a much more rapid rate than they can be characterized. A particular problem is that many of the classic analytic tools, such as x-rays, require bulk samples rather than the ultra-low mass thin films which are the basic constituents of combinatorial solid state libraries. In this paper, we have demonstrated the feasibility of an analytical

probe which provides detailed chemical and structural information for the individual elements of an as-fabricated library. In the future, we envision combining a dedicated x-ray microfocus beamline with a computer controlled wafer stepper and advanced data management. This will allow complete structural and compositional characterization of > 1000 element libraries on time scales of less than an hour. While analogous instruments employing other probes are conceivable, x-rays are unique both because their penetrating power allows the libraries to be examined under arbitrary environmental conditions and their collimation and weak-coupling nature allow extraordinarily precise quantitative analysis.

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MARCH 1999 NSLS Newsletter

VUV Ring Report

Stephen Kramer

VUV Ring Manager

The VUV Ring came back from the winter shutdown in record fashion. A new policy was instituted for the turnover of the ring's systems to the operators by the groups responsible for those systems on the VUV Ring. This change helped to prevent some of the turn-on delays which have occurred after previous shutdowns. With the rapid conditioning experience obtained from the recovery after the vacuum accident in November, the ring conditioning proceeded ahead of schedule and the ring was returned to operations two days ahead of schedule. Beam lifetime was still improving when the fire in the linac modulator stopped operations.

The major work performed on the VUV Ring during the winter shutdown was the improvement of the radiation shielding for the Booster to VUV Ring transport line and the Ring shielding from U11 to U16. Although a detailed comparison of the reduction in the radiation levels with the new shielding will take sometime to develop, initial measurements show a factor of 1.6 to 3.5 reduction in the levels at the VUV shutter during normal injections into the VUV Ring.

Other changes to the VUV Ring made during this shutdown included: new vacuum gauges and RGA electronics, a new higher resolution bunchlength monitoring system and replacement of the active filter on the dipole power supply. Also installed was the support girder, vacuum chamber and bremsstrahlung shielding for the U3B diagnostic beam port. This beamline will make use of an unused portion of the U3 bending magnet beam to measure the energy distribution and changes of the VUV electron beam. A pair of mirrors will bend the beam back toward the bending magnet, where a detector system will measure the synchrotron light from the storage ring beam radiated from a high energy dispersion point in the ring. This beamline will require very little user floor space, while utilizing a previously unused portion of this bending magnet beam. We wish to thank Mike Sagurton (U3C) and Anthony Burek (U3A) for their cooperation and assistance during the installation of this beamline. The first mirror in this beamline should be installed within the next couple of months and the final portion of the beamline could be installed during the spring shutdown. When completed the measurements possible from this beamline should provide a unique insight into the energy structure of the VUV Ring beam and its impact on the synchrotron light beams seen by the users. We gratefully acknowledge the cooperation of the PRT's on the U3A and U3C for allowing this beamline to make use of the U3B beam port. Hopefully this will be the first of many more cooperative interactions between the PRT's and the accelerator systems.

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MARCH 1999 NSLS Newsletter

Facility Update

Frank Terrano

Assistant to Chairman

Head of Administration

Experimental Floor Lighting

The new mercury vapor lighting fixtures to be installed around the X-Ray and VUV Rings have been purchased and are in house. Having previously tested this lighting in several locations around the rings, we found user opinions to be more favorable towards the new whiter "daylight" illumination than the dimmer "yellowish" light cast by the existing sodium lamps. The installation process will include some reconfiguring of the lighting plan to take better advantage the fixture locations with regard to the working areas around the experimental floor. Several of the new fixtures have already been installed, and the job will continue until all of the old fixtures have been replaced.

New Card Reader on Roll-Up Doors

The roll-up doors opening onto the delivery ramps on the east side and west side of building 725 will have card readers installed in the immediate future. The card readers will replace the "push" buttons on the inside of the building, which currently activate the doors allowing entry for deliveries into the building. Presently anyone on the inside of the building may open the roll-up doors at any time. This presents a potential entry point for unauthorized personnel or individuals not having a proper radiation badge and training to roam the area without escort. The new card reader will be set to recognize NSLS staff ID cards and will record the activation of the door. Users needing to use either roll-up door during off-hours or on weekends may contact the Control Room for assistance.

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Work Procedures at BNL

The Laboratory recently established new procedures for issuing "Stop-Work" orders for situations involving "Imminent Danger" or violations of Radiological Requirements. All BNL employees are being trained on these procedures, as well as all guests who hold appointments to work in our facilities.

Any users who receive the NSLS Safety Orientation or request radiation dosimeters in User Administration will receive this brief training. The training involves reading two short pamphlets and answering 5 multiple-choice questions.

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MARCH 1999 NSLS Newsletter

NSLS Gets High Marks in DOE Audit of Chemical Handling

Recently, the local DOE Area Office conducted a review of the management of chemicals at all BNL facilities. We are happy to report that NSLS received high marks for our chemical management practices. "Nice going!" to Andy Ackerman and John Aloi, who manage our programs, and to all the users at NSLS who work with us to keep our facility safe and in compliance with BNL requirements.

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MARCH 1999 NSLS Newsletter

Transport of Hazardous and Radioactive Materials To, From and Within BNL

Occasionally, problems have developed in the transport of hazardous materials between user institutions and BNL. As you prepare for your work at BNL, please keep the following requirements in mind:

1. All chemicals as well as other hazardous or radioactive materials must be listed in detail on NSLS Experimental Safety Approval Forms. We will advise you if any chemicals require barcoding by the BNL Chemical Management System (CMS) or if any special handling at BNL will be required.
2. Packaging, labeling and transport of chemicals and hazardous materials to and from BNL must be done in strict accordance with the Department of Transportation regulations. Contact your institution's Safety Office for guidance. Shipping of chemicals and hazardous materials from BNL must be done through BNL's Shipping Division and a Material Safety Data Sheet (MSDS) must accompany each item.
3. Transport of any radioactive material to, from and within BNL must be done through the BNL Isotopes & Special Materials Group. You are not allowed to transport these materials in your private vehicles or suitcases. Contact I&SM at least 2 weeks in advance of your arrival at BNL: 516-344-5233. Federal law 49CFR170-180 governs transport of hazardous materials to and from BNL.
4. As a DOE-operated institution, BNL falls under the Price Anderson Amendment Act (PAAA). We must therefore follow the regulations set out in the Code of Federal Regulations 10CFR835, "Occupational Radiation Protection". All handling of radioactive material must be in compliance with these regulations. Not following these regulations makes BNL or an individual liable for civil and/or criminal penalties.
5. When legally transporting any scientific equipment or samples, you should always carry information on your institution's letterhead stationary describing the equipment or samples and what hazards they may pose to other individuals, if any. Presenting such information up front at airport terminals, for example, will make your travels considerably smoother.

If you have additional questions as to what constitutes a hazard or if you know you are bringing material of significant hazard, contact Andrew Ackerman (516-344-5431; ackerman@bnl.gov) or Chris Weilandics (516-344-2593; weil@bnl.gov) well in advance of your arrival to determine agreed upon procedures while at the NSLS. Lack of approved procedures could result in postponement of your experiment.

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MARCH 1999 NSLS Newsletter

CALL FOR GENERAL USER PROPOSALS

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

Tuesday, June 1, 1999 for scheduling Sept-Dec 1999

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 arrangements for equipment. Your chance of getting beam time is improved by being able discuss any special to use more than one beamline.

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 to us.*

Special Notes for Macromolecular Crystallography (PX)

NEW PROPOSALS:two-year program. Provide an overall plan for your research according to The proposal represents a 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 scheduling cycle. Submit PX Forms only for those 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).

Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Tuesday, June 1 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 June 1.** 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.

Additional Information and Forms

Blank proposal forms and instructions, a guide to the NSLS beamlines, and more information about the General User Program are available on the World Wide Web at **www.nsls.bnl.gov**, or by contacting E. Pinkston or L. Rogers in the NSLS User Administration Office. Office hours are Monday through Friday, 8:00 am to 5:00 pm Eastern Time. Contact information is on the back page of this Newsletter.

Safety Approval Form Reminders:

Submit via the Web ***at least one week before*** your scheduled beam time. Do not send in a SAF with your proposal.

Complete all fields on the SAF Web form - use "NA" or "not applicable" if you need to, but answer each question!

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IMPORTANT UPCOMING DATES

April 18-23, 1999	Rapid Data Collection & Structure Solving at NSLS: A Practical Course in Macromolecular X-Ray Diffraction Measurement
May 22, 1999	Deadline for submissions, July Newsletter
May 25, 1999 May 24 & 26	Annual Users' Meeting Workshops
June 1, 1999	Deadline for General User Proposals (Sept. - Dec. '99)

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