

# MARCH 1998 NSLS NEWSLETTER

*Editor: Eva Z. Rothman*

*Production Assistant: Nancye Wright*

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## TABLE OF CONTENTS

- [Chairman's Introduction](#)
- [Observation of a Half-Metallic Ferromagnet](#)
- [Spin Density Waves in Epitaxial Cr Layers](#)
- [SOLVE - Automatic Real-Time Structure Solving at the NSLS](#)
- [ES&H: Enhanced Work Planning at BNL](#)
- [ES&H: Involving Everyone in Safety Self-Assessment](#)
- [Focus On ..... The NSLS RF Group](#)
- [A User's Perspective](#)
- [X-Ray Ring Report](#)
- [VUV Ring Report](#)
- [Detailed and Long Range Operating Schedules](#)

## NEWS and ANNOUNCEMENTS

- [Special Instructions for Foreign National Users](#)
  - [Reminder about the NSLS Annual Users' Meeting and Workshops](#)
  - [EXAFS Equipment Pool](#) and [Mail-in EXAFS Service](#)
  - [Call for General User Proposals](#)
- 

## [Important Upcoming Dates](#)

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[NSLS Newsletters Page](#).....[NSLS Home Page](#)..... [BNL Home Page](#)

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February 27, 1998

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# Introduction by the Chairman

### Michael Hart

The recent Winter shutdown was much more hectic than usual. It began with a major electrical power failure two days before the planned start of the shutdown and ended with the discovery of a water to vacuum leak in the X17 wiggler straight which forced the suspension until May of all X-17 experimental runs. Heroic efforts by staff from NSLS, BNL Plant Engineering and contractors achieved an almost on time startup in January.

On March 1<sup>st</sup>, Brookhaven Science Associates (BSA) became the new Contractors to D.O.E. with the task of Management and Operation of Brookhaven National Laboratory under Contract No. DE-AC02-98CH10886. Since January a transition team has been working with staff, management, A.U.I., and D.O.E. groups to achieve an orderly change of leadership.

The vision of our new contractor, an outline of some of new organization, and a partial list of senior staff can be found through the BNL web page at [www.bnl.gov](http://www.bnl.gov). BSA intends " *to preserve the best elements of the academic culture that underpins BNL's scientific excellence, while implementing lasting culture changes that will lead to necessary improvements in operations, ES&H, and community involvement within the Laboratory.*"

To achieve this goal, BSA will introduce several significant changes within the Laboratory:

- Establishing two Deputy Director positions: one to plan, direct, and oversee BNL's scientific and technical programs, and one to lead the operations and technical support services within the Laboratory.
- Consolidating all applied science and technology programs along with BNL's economic development and technology transfer efforts into a new Applied Science and Technology organization to provide a focal point for future growth
- Creating a new ES&H and Quality organization responsible for providing technical support services to the line organizations and maintaining the Laboratory's independent oversight function
- Creating a new Environmental Management organization responsible for leading the current waste management and clean-up efforts, identifying future environmental restoration and waste management needs, and inserting new technologies into the current program
- Consolidating all community relations, public affairs, communication, and educational outreach activities into a new Community Involvement and Public Affairs directorate
- Placing the High Flux Beam Reactor Restart Project at the first level of the organization (in the event that the decision is made to restart the HFBR).



Some of the new directorate will already be well known to NSLS users and biographies are given in more detail than I can do here on the above web pages. Those most likely to be directly in contact with NSLS staff and users are:

John Marburger III, Laboratory Director:

He was, for 14 years, President of Stony Brook U.

Peter Paul, Deputy Director, Science and Technology:

Chairman and leading professor, Department of Physics, Stony Brook University

Thomas Sheridan, Deputy Director, Operations:

Former manager, TWRS Regulatory Compliance Unit, Battelle at Pacific Northwest National Laboratory

Kenneth C. Brog, Assistant Laboratory Director, ES&H/Quality:

Former Battelle corporate vice president, ES&H; and director, ES&H, PNNL

J.T. Adrian Roberts, Associate Laboratory Director, Applied Science and Technology:

Former Battelle director, PNNL Economic Development Office

Michael Schlender, Assistant Laboratory Director, Environmental Management:

At Battelle, led a Laboratory-level initiative at PNNL to produce an environmental service organization with field-deployed environmental professionals

Denis McWhan continues as Associate Laboratory director for Basic Energy Sciences and Richard Setlow as Associate Laboratory Director for Life Sciences.

BSA's vision for BNL's scientific future includes:

- "• The successful delivery and operation of RHIC as a preeminent nuclear physics facility;
- Continued focus on vital high energy physics, nuclear physics, and neutron science programs;
- Maintenance of the NSLS as a state-of-the-art user facility;
- Significantly strengthened capabilities and programs in molecular biology and other life sciences areas built around BNL's unique instrumentation;
- A strengthened and diversified applied science and technology portfolio contributing significantly to all DOE missions; and
- Strengthened partnerships in research, education and technology transfer with academia and industry."

[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

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February 23, 1998

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# Observation of a Half-Metallic Ferromagnet

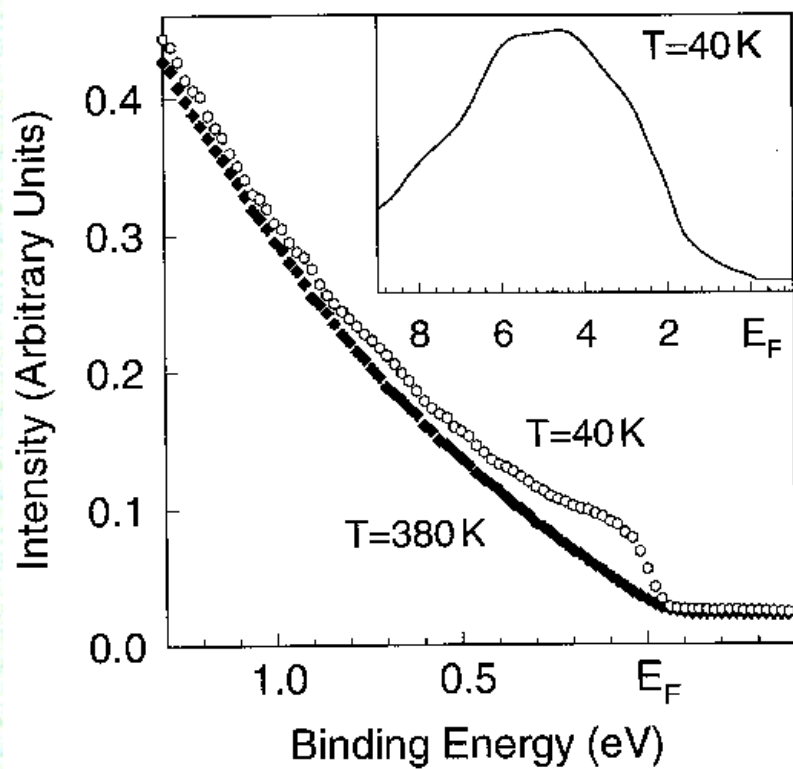
*J.-H. Park, E. Vescovo, H.-J. Kim (NSLS)*

Half-metallic systems are new class of materials which are characterized by the coexistence of metallic behavior for one electron spin and insulating behavior for the other. The density of states (DOS) has 100% spin polarization at the Fermi level ( $E_F$ ), and the conductivity is completely dominated by the metallic single-spin charge carriers. This exotic physical property could have a significant impact on technological applications related to magnetism and spin electronics. The half-metallicity was theoretically predicted for some ferromagnetic materials such as Mn-based Heusler alloys and chromium dioxide. However, the half-metallic nature has never been demonstrated directly and the predictions are still in doubt. We have performed spin-resolved photoemission measurements on a ferromagnetic manganese perovskite,  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ , which is well known to yield a large negative magnetoresistance, the so-called "colossal" magnetoresistance (CMR), near the Curie temperature ( $T_C$ ). This system is expected to have highly spin-polarized conduction electrons, and the possibility of 100% spin-polarization (i.e. a half-metal) has also been discussed. However, band structure calculations and experimental spin-resolved tunneling studies have not confirmed the half-metallic nature of this system.

A high quality  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  thin film, which was epitaxially grown on a (001)  $\text{SrTiO}_3$  single crystal substrate by a pulsed laser deposition method, was provided by C. Kwon, R. Ramesh, and T. Venkatesan at the University of Maryland. The  $T_C$  of the sample was determined to be  $\sim 360$  K. The measurements were performed at the new U5UA undulator beamline at the NSLS. The sample was introduced into an ultra high vacuum, better than  $1 \times 10^{-10}$  Torr, and the surface was cleaned *in situ* in a sequence of annealing processes.

The successful achievement of the clean surface is confirmed in the valence band photoemission spectra near  $E_F$  shown in **Figure 1**, which were measured well below  $T_C$  ( $T = 40\text{K}$ ) and above ( $T = 380\text{K}$ ). The spectra clearly exhibit the metal to non-metal transition associated with the ferromagnetic transition: at  $T = 380\text{K}$ , the spectrum shows negligible spectral weight at  $E_F$  while at  $T = 40\text{K}$ , it clearly shows the metallic Fermi cutoff. The wide range spectrum shown in the inset is also very similar to that reported for polycrystalline  $\text{La}_{1-x}\text{Sr}_x\text{MnO}_3$  samples.

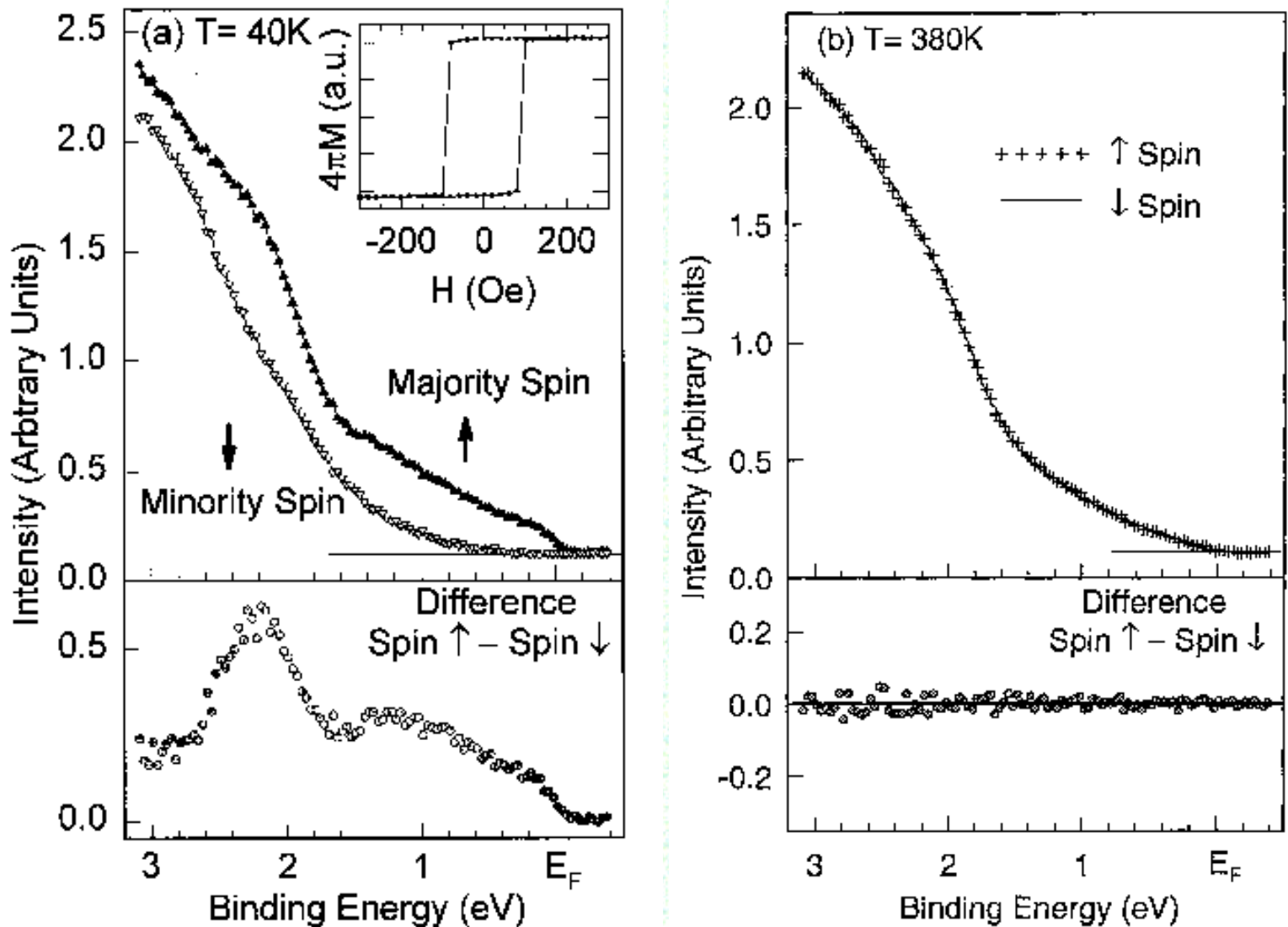
**Figure 1. High resolution photoemission**



**spectra of a  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  ( $T_C \sim 360\text{K}$ ) thin film very near the Fermi energy ( $E_F$ ) at  $T = 40\text{K}$  ( $\ll T_C$ ) and at  $T = 380\text{K}$  ( $> T_C$ ). The photon energy and the experimental resolution were set to  $h(\nu) = 40\text{ eV}$  and  $(\Delta)E = 0.1\text{ eV}$ , respectively. The inset shows a wide scan valence band spectrum at  $T = 40\text{K}$ .**

**Figure 2** shows the valence band spin-resolved photoemission spectra near  $E_F$  of the  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  thin film measured at (a)  $T = 40\text{K}$  and (b)  $T = 380\text{K}$ . At  $T = 40\text{K}$  ( $\ll T_C$ ), all the Mn  $3d$  electron spins are aligned ferromagnetically, and the spectra in **Figure 2(a)** clearly show the striking *half-metallic* feature; the majority spin (up arrow) band extends up to  $E_F$  and shows the metallic Fermi cutoff, while the minority spin (down arrow) band has almost disappeared at  $\sim 1\text{eV}$  binding energy and the spectral weight is vanishingly small near  $E_F$ , reflecting the insulating gap. The resulting spin-polarization near  $E_F$  is  $\sim 100\%$  and the half-gap on the occupied state (photoemission) side of the minority spin states is estimated to be  $0.6\text{eV} \pm 0.2\text{eV}$ . The minority spin states still show large spectral weight at higher binding energies owing to O  $2p$  states, which are fully occupied for both spins. The Mn  $3d$  state spectrum can be obtained by subtracting the minority spin spectrum from the majority spin spectrum. Such a difference spectrum at  $T = 40\text{K}$  (**Figure 2a**, bottom panel) shows the metallic Fermi cutoff at  $E_F$  and two features around  $1.2\text{ eV}$  and  $2.2\text{ eV}$  binding energies, which can be interpreted as the Mn  $3d$   $e_g$  and  $t_{2g}$  electron removal states, respectively. Upon heating through  $T_C$ , the Mn  $3d$  spins become disordered, and the spin anisotropy disappears. The spin-resolved photoemission spectra above  $T_C$  ( $T = 380\text{K}$ ) shown in **Figure 2(b)** confirm the disappearance of the spin-anisotropy. The difference spectrum of the up (up arrow) spin and down (down arrow) spin spectra, in the bottom panel, exhibits only experimental noise.





**Figure 2.** Spin-resolved photoemission spectra of a  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  ( $T_c \sim 360\text{K}$ ) thin film near the Fermi energy ( $E_F$ ) at (a)  $T = 40\text{K}$  ( $\ll T_c$ ) and at (b)  $T = 380\text{K}$  ( $> T_c$ ). The photon energy and the experimental resolution were set  $h\nu = 40\text{eV}$ , and  $DE = 0.2\text{eV}$ , respectively. The bottom panels show the corresponding difference spectra between the (up arrow)-spin and the (down arrow)-spin spectra. The inset in Figure 2(a) shows the magnetization ( $M$ ) vs. applied magnetic field ( $H$ ) hysteresis loop, which was obtained by monitoring Mn  $L_2$ -edge absorption intensity for circularly polarized incident light at the NSLS U4B beamline.

The results of our spin-resolved photoemission studies of  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$  show (1) the half-metallic behavior well below  $T_c$  and (2) the occurrence of the metal to non-metal transition accompanied by the loss of the ferromagnetic order upon heating through  $T_c$ . The observed half-metallic nature of a doped manganese perovskite,  $\text{La}_{0.7}\text{Sr}_{0.3}\text{MnO}_3$ , is the first proof of the existence of half-metallic ferromagnets. This may open up a new class of technologically important materials.

[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

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# Spin Density Waves in Epitaxial Chromium Layers as Revealed by Synchrotron Radiation

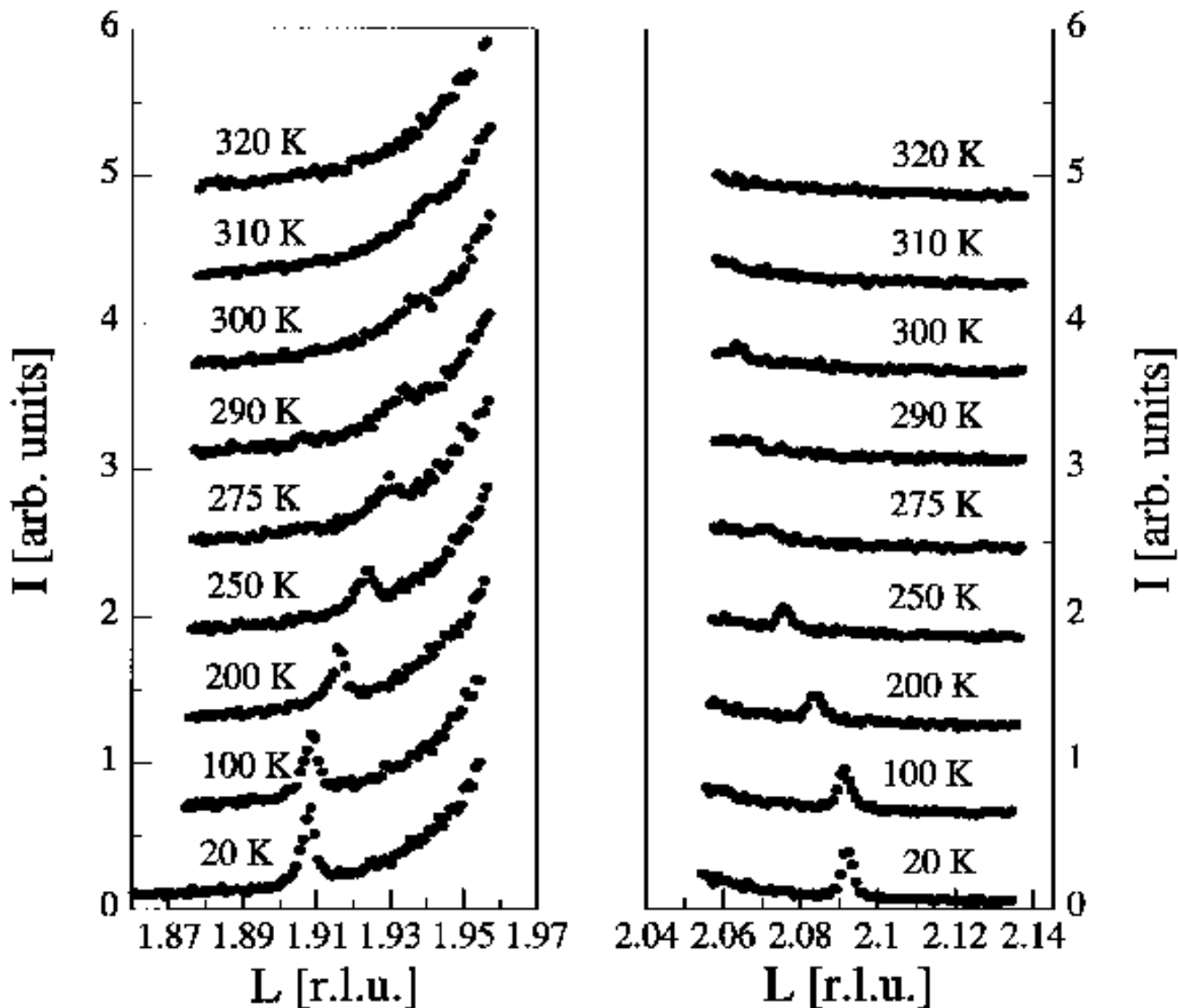
*Hartmut Zabel, Peter Sonntag (currently at Hamburger Synchrotron Lab [HASYLAB]), and Patrick Bödeker (Ruhr-Universität Bochum, Germany)*

The incommensurate spin density wave (SDW) magnetism of Cr has fascinated many researchers since its first discovery via neutron scattering in 1959<sup>[1]</sup>. In thin films the magnetism of Cr has recently become the focus of interest because of its mediating role in exchange coupled superlattices and GMR materials. While the magnetic phase diagram and the Néel temperature,  $T_N=311\text{K}$ , are well established for bulk Cr, the question arises of how these properties are altered in thin films and superlattices either due to dimensionality effects or due to proximity with ferromagnetic boundary layers. The early scattering experiments were performed on large single crystals. Unraveling the SDWs in thin films is a challenge only met by modern synchrotron sources. During the past several years we have studied in detail the SDWs in epitaxially grown Cr(001) films as function of temperature and film thickness using the NSLS X22B beamline. Furthermore, we have investigated proximity effects between ferromagnetic cap layers and the propagation and polarization of the SDWs in Cr.

In bulk Cr, the incommensurate SDW consists of a sinusoidal modulation of the amplitude of the anti-ferromagnetically ordered Cr moments with a wave vector  $Q$  being incommensurate with the lattice periodicity of Cr<sup>[2]</sup>. There exist domains for the different propagation of the SDWs along the three possible  $\{100\}$  directions in the bcc lattice. Usually these domains are equally populated, referred to as a triple domain state. The wave vector  $Q$  determines the propagation direction of the SDWs and from the magnitude,  $|Q| = (2\pi/a)(1-\delta)$ , follows the periodicity  $L = a/\delta$  of the SDW, where  $a$  is the lattice parameter of Cr and  $\delta$  the deviation of the SDW from commensurability. Aside from the propagation, we have to distinguish between different polarizations of the SDW: the magnetic moments may be oriented either parallel or perpendicular to  $Q$  for longitudinal or transverse SDWs, respectively. At low temperatures the SDWs have longitudinal character, whereas between the spin-flip transition at 123 K up to the Néel temperature of 311 K the SDWs are transverse. The period of the SDW is temperature dependent and increases from about 60 Å at 100 K to 78 Å at the Néel temperature. Above the Néel temperature, Cr is paramagnetic.

The sinusoidal modulation of the magnetic moments produces sharp Fourier components in the reciprocal lattice, which can, for instance, be determined by neutron scattering or high energy non-resonant x-ray scattering. Furthermore, the SDW induces a charge density wave (CDW) and a strain wave (SW) in the bcc lattice with half the modulation period of the SDW, which can be observed via x-ray scattering<sup>[3,4]</sup>. The corresponding satellite reflections occur close to the charge Bragg peaks of the bcc lattice. Using non-resonant scattering with synchrotron radiation at not too high energies, the propagation direction of the SDWs can be determined but not the polarization. The most striking features of the SDWs in thin epitaxial Cr films comprise a single  $Q$  - state propagating perpendicular to the growth plane with a modulation period being much longer than in bulk and which scales with the inverse film thickness. Moreover, the spin flip transition from longitudinal to transverse SDW occurs at higher temperatures as the film thickness decreases<sup>[5,6]</sup>. **Figure 1** reproduces the strain wave satellite reflections in a 3000 Å thick Cr(001) for different temperatures as revealed by scattering experiments with synchrotron radiation.





*Figure 1. Scans showing strain wave satellite reflections on both sides of the Cr(002) reflection in a 3000 Å thick Cr[001] film. The temperature dependent period of the strain wave is clearly visible.*

As we cover the epitaxial Cr(001) films with a Fe(001) cap layer, we expect that the Cr moments in the first monolayer at the interface line up antiparallel to the Fe moments due to the antiferromagnetic *inter*-layer Fe-Cr coupling. Since thin Fe layers show an in-plane magnetization, this coupling should force the SDW to propagate normal to the plane and with spins lying in the plane, i.e. a single Q transverse out-of-plane SDW is expected. Contrary to expectations, our scattering experiments clearly show that the SDW propagates parallel to the film plane. Neutron scattering experiments confirm this result and furthermore show that the Cr magnetic moments are oriented at a right angle with respect to the Fe in-plane magnetization<sup>[7,8]</sup>. A typical scattering experiment with synchrotron radiation from a 2100 Å thick Cr(001) film capped with a 20 Å thick Fe(001) layer is shown in **Figure 2**. The scans are taken parallel to the [010] direction across the commensurate (011) Bragg peak position. At low temperatures satellite reflections to the left and right side of the (011) peak can be recognized from the incommensurate SDW existing up the Néel temperature. The satellite reflections occur only along the [010] direction but not along [001] as for uncapped Cr films, proving that the SDW indeed has been reoriented due to the presence of the Fe cap layer.



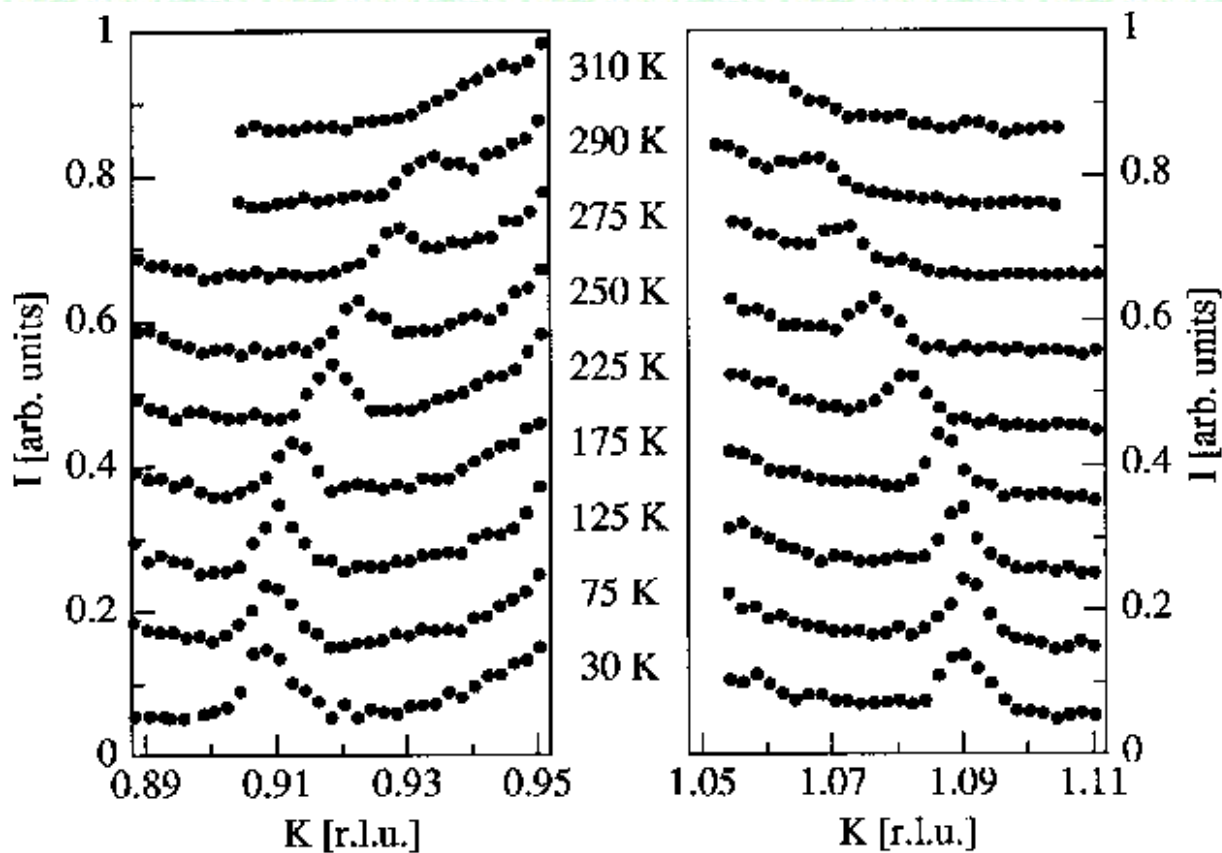


Figure 2. Strain wave satellite reflections in scans along the [010] direction at the (011) Bragg reflection in a 2100 Å thick Cr(001) film capped with a 20 Å thick Fe(001) layer.

The re-orientation of the wave vector  $Q$  from out-of-plane (without magnetic cap layer) to in-plane (with magnetic cap layer) is due to steps and kinks at the interface, causing the antiferromagnetic interactions at the Fe-Cr interface to be frustrated. To overcome this frustration effect, domains could be formed either in the Fe or the Cr layer, which, however, costs *intra*-layer exchange energy. The competition between both, *intra*- and *inter*-layer exchange interactions, determines whether a re-orientation takes place or not. For large Cr layer thicknesses the re-orientation is the energetically more favorable choice, which has recently been confirmed by computer simulations<sup>[7,9]</sup>.

The re-orientation takes also place when the Cr film is covered with very thin Ni or Co layers, whereas a Cu cap layer has no effect on the orientation of  $Q$ . Highly polarizable Pd cap layers have an effect, which is intermediate between the ferromagnetic and the diamagnetic case. In this case we find domains with in- and out-of-plane propagation of the SDW. In none of the cases the cap layers change the Néel temperature.

As we reduce the thickness of the Fe capped Cr films, we observe another transition of the SDW from propagating parallel to perpendicular to the film plane<sup>[7]</sup>. This transition starts at a Cr thickness of about 750 Å and is completed at about 250 Å. Below 250 Å the frustration effects are overcome, and the energy balance favors a transverse out-of-plane SDW, squeezed between the boundary Fe layers. Finally, when the thickness of the Cr layer is reduced to values below the length of one modulation period, a transition from an incommensurate SDW to a commensurate antiferromagnetic structure takes place<sup>[10,11]</sup>. The commensurate state has a Néel temperature which is typically much higher than  $T_N$  for the incommensurate state.

In conclusion, we have shown via scattering experiments with synchrotron radiation that thin Cr films exhibit a single domain spin density wave state. The Néel temperature and the modulation period of the SDW scales with the film thickness, whereas its propagation direction depends on the magnetic boundary condition. As a result of frustrated exchange coupling at rough interfaces, ferromagnetic cap layers on Cr cause the spin density wave to propagate in the plane with magnetic moments pointing at a right angle to the ferromagnetic

layer. However, with decreasing Cr film thickness the propagation direction flips into the out-of-plane direction. Finally, as the Cr film thickness is reduced to below the extension of a spin density wave period, the SDW state collapses and Cr becomes a simple antiferromagnet with a much higher Néel temperature. In this state the exchange coupling in Fe/Cr superlattices is strong and mediates a two Cr-monolayer period of the interlayer exchange coupling<sup>[12]</sup>, as well as a non-collinear spin structures in the Fe sublattice<sup>[11]</sup>.

*We would like to acknowledge the collaborations with our co-workers during synchrotron data collection, in particular Tom Thurston, Ben Ocko and Doon Gibbs (BNL Physics Dept.), and Andreas Schreyer (Ruhr-Universität Bochum) for fruitful discussions. Work was supported by the Deutsche Forschungsgemeinschaft through SFB 166 ("Strukturelle und magnetische Phasenumwandlungen"). H.Z. also acknowledges receipt of a Volkswagen fellowship while this manuscript was written.*

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

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# SOLVE — Automatic, Real-Time Structure Solving at the NSLS

*Thomas Terwilliger and Joel Berendzen,  
Los Alamos National Laboratory*

The pace of protein-structure determination is accelerating rapidly, and synchrotron x-ray sources such as the NSLS are at the center of this action. Structures of protein molecules are in high demand in biotechnology because they are important for applications such as drug discovery and engineering enzymes for commercial use. A structure of a protein molecule can be used to identify what each part of the molecule is doing and makes it possible to design new protein molecules that have improved properties for therapeutic or industrial use. As the genome projects continue to produce thousands of new protein sequences, the demand for structural information on these proteins is increasing. Synchrotron beam lines at the NSLS a key tool for protein structure determination. The multiwavelength anomalous diffraction (MAD) method takes advantage of the intensity and tuneability of x-rays at the NSLS in obtaining x-ray diffraction data that can be used to solve protein structures that contain atoms with accessible x-ray absorption edges.

Until recently, the analysis of MAD X-ray data has been somewhat difficult owing to the number of steps involved and to the need for an expert to make important decisions about how to handle the data. We have developed a new software package called SOLVE that can carry out all the steps and make all the necessary decisions in the analysis of MAD data automatically, and we have installed it at beamlines X12C, X8C, and X4A at the NSLS. The SOLVE software has now been run by a number of users at these beamlines and has proven itself capable of solving protein structures in less time than was necessary to collect the x-ray data.

## How SOLVE Works

SOLVE is an expert system that automatically produces three-dimensional electron-density maps of protein molecules from x-ray diffraction measurements. For a MAD structure determination, a user at the NSLS will measure diffraction intensities from a single crystal at several x-ray wavelengths spanning an absorption edge for an anomalously scattering atom, such as selenium, incorporated into the protein. This is the raw data needed by SOLVE. The user tells SOLVE where these data are located, what the scattering properties of the selenium atoms are at the x-ray wavelengths used, how many selenium atoms are thought to be in the protein, and how big the protein is. SOLVE takes this information and constructs an electron density map that can be displayed using a graphics program.

The approach used by SOLVE is similar to the one that a protein crystallographer would use. The MAD method for structure determination is a kind of bootstrapping operation in which the positions of the anomalously scattering atoms are first deduced and then used to calculate electron density for the entire structure. The hard part is coming up with likely solutions for the positions of the selenium atoms and evaluating the relative quality of different solutions. These steps often have been done manually in the past, while SOLVE can carry them both out automatically. SOLVE generates a list of likely solutions for the locations of the heaviest atoms in the protein using the program HASSP to analyze an optimized Patterson function derived from the MAD data with MADBST. It then evaluates each solution for internal consistency and it compares characteristics of the electron density map obtained from that solution with those of real electron density maps of proteins. Using the best starting solutions, SOLVE bootstraps to generate improved solutions, and when further improvement is not possible produces a final electron density map of the protein molecule.

Several additional features make SOLVE much faster than it otherwise would be and enable real-time structure solving. One, the MADMRG procedure, is a technique for extracting the three essential pieces of information from a set of up to MAD diffraction measurements. Another, the origin-removed Patterson refinement procedure in HEAVY, reduces calculation time by rapidly adjusting a solution to match the x-ray measurements. Bayesian Correlated MAD Phasing, is a comprehensive way to deal with errors in measurement and to deal with large differences between crystals used in the diffraction analysis. The result of this integration of techniques is a fully automated analysis of x-ray diffraction measurements.



# Using SOLVE to Solve a Structure in Real Time at X12C

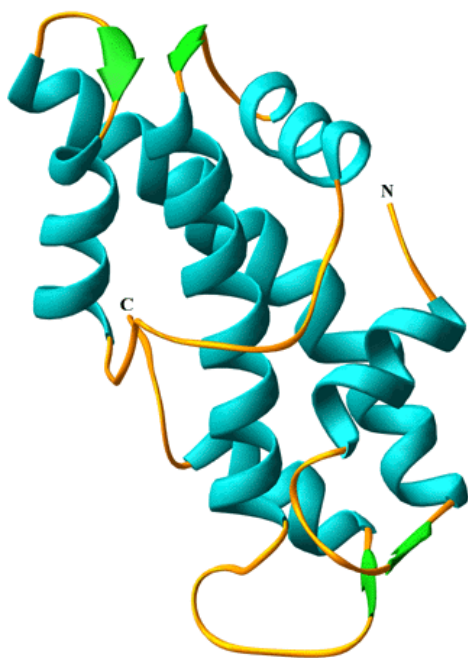
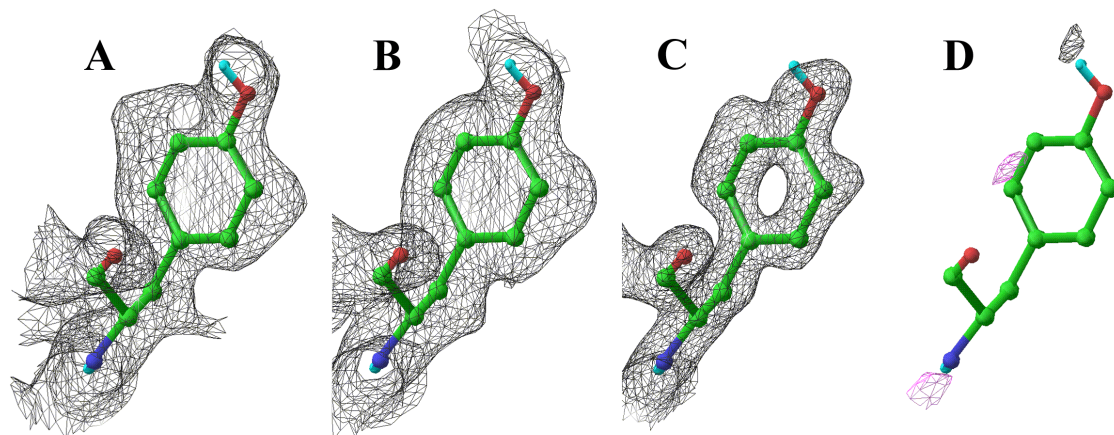
The recent successful structure determination by Richard Fahrner, Duilio Cascio and David Eisenberg (UCLA) at X12C illustrates how useful SOLVE can be. The UCLA group was interested in determining the structure of a histone protein (HMK) from a thermophilic organism. They initially used molecular replacement and multiple isomorphous replacement, two traditional crystallographic methods for phase determination that do not require synchrotron radiation. However, these methods failed to produce interpretable electron density maps. They then turned to MAD phasing. Using established methods for selenomethionine incorporation, they were able to produce selenomethyl histone protein and obtain crystals of the protein. In November 1997, they came to beamline X12C at the NSLS to collect data for a three wavelength MAD experiment.

The histone protein crystallizes as tetragonal bipyramids in space group  $P4_12_12$  ( $a=b=57.72 \text{ \AA}$ ,  $c=97.61 \text{ \AA}$ ) and have little mosaic spread (0.20 degrees) when frozen at 100K. Since accurate anomalous information is required for a good MAD experiment, they collected anomalous pairs using the Friedel flip method: for every wavelength there were two datasets, sweeping the same zones, 180 degrees apart. Their MAD experiment consisted of six complete datasets; two at the fluorescence inflection point ( $0.9798 \text{ \AA}$ ), two at the "white line" peak ( $0.9799 \text{ \AA}$ ), and two at a distant point ( $0.9500 \text{ \AA}$ ). Data collection proceeded smoothly and rapidly, employing the automatic MAD data-collection protocols devised by J. Skinner and R. Sweet (BNL Biology Department). Two factors accelerated high resolution data collection. The size and dynamic range of the new Brandeis CCD detector permitted collection of high resolution and low resolution data in a single pass. Also, careful orientation of the crystal with respect to the major zones allowed collection of a large amount of data in a short period of time. The integrated data was about nine-fold redundant, greater than 99% complete to  $1.4 \text{ \AA}$ , with a merging R of 8.1%. Data collection for the complete MAD experiment required about seven hours.

The software package SOLVE was used to accelerate phase determination and assess the quality of their MAD experiment. The SOLVE package offered several advantages for them. Whereas manual solution of Patterson maps in high symmetry space groups with multiple heavy atom sites is challenging and time consuming, SOLVE offered a simple-to-set-up, automatic phase determination alternative. This was particularly attractive for them because solving a Patterson map in a high symmetry space group with the amount of sleep they had would have been an extreme challenge. The three-wavelength unmerged data with Bijovet pairs were input to SOLVE. Using the anomalous and dispersive differences between datasets, SOLVE determined a single solution for four of five Se-Met sites within 78 minutes running on a 500Mhz DEC ALPHA workstation. SOLVE was even able to distinguish the correct hand of the structure. The time from placing the crystal into the beam until the structure was solved was an astonishing eleven hours, roughly 18 hours after the Brandeis 2x2 detector was put into operation for any data collection at all. (This was the first data set collected on the new X12C Brandeis 2 x 2 CCD-based detector.)

The initial electron density maps were easily interpretable (**Figure 1**). The map quality was then improved through solvent-flattening and histogram-matching routines using the program "dm" in the CCP4 program suite. The solvent-flattened map could be unambiguously traced for 98 of 154 residues. After refinement using XPLOR, the amino acids originally traced were returned in the  $2F_o-F_c$  map and more importantly, most of the remaining residues could be traced. Their current  $2.0 \text{ \AA}$  model includes residues 3 to 154 and 40 water molecules ( $R=26.3\%$ ,  $R_{\text{free}}=33.7\%$ ).





**Figure 1 (top):** Electron density maps from MAD phased Mkh data at 2.0 Å resolution, contoured at 2.0 sigma around tyrosine 108. **A:** Density obtained using phases from solve. **B:** After solvent flattening and histogram matching. **C:**  $2f_o - f_c$  density from current model.

**Figure 2 (bottom):** Ribbon diagram of Mkh structure.

[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 28, 1998

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# **Environment, Safety, & Health (ES&H): Safety and Compliance**

## **Enhanced Work Planning at BNL**

*Tom Dickinson*  
*NSLS Safety Officer*

Most of us at one time or another have set about to do a job only to have it turn out not as we expected. Seldom are these outcomes an improvement on our expectations, and sometimes they represent serious delays, financial loss, or accidental injury. Occasionally this is due to bad luck, but more often it is caused by inadequate planning. Poor work planning often shows up as a contributing factor in incident and injury reports, and for some time the DOE has had a program for improved work control and "Enhanced Work Planning". These systems are in place at several DOE facilities with good results in improving efficiency and safety. BNL is in the process of developing a Work Control Policy, motivated by recommendations coming out of recent safety and management reviews at the laboratory. A draft of the BNL Work Control Policy is nearly ready for distribution, and the NSLS has been chosen for a Pilot Demonstration of Enhanced Work Planning starting the first of February.

The traditional approach to work planning has been for an individual to define and plan a job with little interaction with support organizations or the work force other than to seek approvals and permits. This approach can lead to poor coordination with other activities concurrent with the job, to late identification of hazards or problems which could cause lost time because of needed changes, and to inconsistent or incomplete work packages. Enhanced Work Planning addresses these problems and promotes efficiency and safety with strategies which include diverse planning/review teams including worker involvement, effective screening of work for complexity and hazards which allows a graded approach to work management, and organized communication and follow-up.

The diverse team brings together the disciplines needed for effective planning and review so this can be an integrated effort rather than a sequential process. The expertise is needed for confidence in the screening process which allows the effort to be focused on the work which needs it while permitting routine work to proceed with minimum encumbrance. It is estimated that 80 to 90 percent of assigned work can be done with minimal planning beyond that provided by normal supervision and skill of the craft. However, not all simple jobs are low hazard. In a recent incident, asbestos was dispersed when some stained ceiling tiles were replaced in a building on site. This led to a massive containment and cleanup effort, serious disruption for occupants of the building, and a heavy liability to BNL for violation of asbestos regulations. The people who knew of the residual asbestos condition in the building were not involved in planning the job. Experience at other DOE facilities has shown that with Enhanced Work Planning, this kind of problem can be avoided without bogging down in endless paperwork and safety reviews.

For the pilot demonstration at the NSLS, we will be focusing initially on jobs to be done at the Light

Source by workers from outside the department, for example from Plant Engineering. Mike Kelly (NSLS Building Manager) will act as Work Control Manager and will handle work requests, coordinate work planning, and track jobs. Tom Dickinson will bring together the teams which will do the screening, planning, and review on these jobs. So in the next month or two if you put in a request for some work on your hutch, an electrical installation, or some equipment fabrication, don't be surprised when a small group comprising a craftsman, a safety person, and maybe someone from operations shows up to do a little planning with you on how the work should be done.

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [\*NSLS User Administration Office\*](#) maintains this document. [\*DISCLAIMERS\*](#) : Revised Date : February 23, 1998

---



# Environment, Safety, & Health (ES&H): Safety and Compliance

## Involving Everyone in Safety Self-Assessment

*John Aloï*  
*NSLS Safety Engineer*

The Tier I safety inspections are an essential part of the NSLS ES&H program, and serve as a primary source of information used in our self-assessment of safety performance. BNL is currently working on self-assessment improvements as part of the ongoing reviews of management and safety at the Laboratory. The NSLS has agreed to pilot one of the improvement activities: working to increase employee involvement in our Tier I inspections. One of the guiding principles developed during the reviews at the Lab is that workers are the primary source of self-assessment information and especially safety and process improvements, and therefore should be directly involved in all elements of the program.

In the past, many individuals have interacted with the Tier I inspection team as they have toured the facility and, of course, others have been involved in correcting the findings. We will be trying to improve the participation of the NSLS staff and beamline staff at the time of the tours. This should provide the opportunity for real-time involvement with the safety personnel on the tour. We hope that there will be much more immediate response to the findings, more awareness created with respect to the constant need for vigilance in the workplace, and more participation in the difficult task that our Tier I inspection personnel have to perform.

The plan is to have staff available in the work place while the Tier I safety inspection team comes through on a tour. No one will have to spend a lot of time. We do not plan to have more staff on the entire tour, just more personnel alerted to the fact that the tour will be coming and they will be expected to join in the inspection of their specific work places. We can expect the program to evolve with experience over the six month trial period. The trial period will conclude at the end of June, with an evaluation of the NSLS pilot program and the other programs being piloted around the Lab.

The NSLS Safety Staff recently conducted its first Tier I safety inspection using the new and improved process. Prior notice of the inspection was provided to supervisors and workers in the NSLS Vacuum Shop. All of the Vacuum Group personnel were present during the inspection. Immediate explanations and feedback regarding observed weaknesses were provided. Quick on-the-spot fixes were accomplished. Response to the new Tier I process was very positive. The NSLS Safety Staff would like to thank the Vacuum Group personnel for their whole-hearted participation in the new program.

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 23, 1998

---



# **FOCUS ON . . . . the NSLS RF Group**

[John Keane](#)

*Electrical Systems Manager*

The energy imparted to the electron beam in both the X-Ray and VUV storage rings comes from the RF system. The 52 MHz accelerator cavity is where this energy interchange takes place. In the VUV Ring, 3 kilowatts are needed to supply voltage to the cavity and an additional 16 kilowatts for beam loads. Due to the higher energy of the X-Ray Ring, substantially more power is required, thus there are four accelerating cavities each driven by a 52 MHz RF power system. The total power to supply gap voltage to the accelerating cavities is four x 33 kW = 132 kW with an additional four x 60 kW = 240 kW for beam loading. The RF group maintains and improves the RF power system and accelerating cavities in both the X-Ray and VUV Rings as well as the booster ring.

Manny Thomas is the electrical engineer for the X-Ray Ring and Nathan Towne is the electrical engineer responsible for the VUV RF system. They are supported by the technician staff guided by Roy D'Alsace and his assistant Gloria Ramirez. Included in the task of the group is maintenance, construction, planning, writing test procedures, highlighting safety requirements and diagnosing causes of failures. To aid in the implementation of work are John Vaughn and Tony Rodriguez. Manny Thomas, Roy D'Alsace and Gloria Ramirez have been here since the early days of the Light Source. Nathan Towne recently came from Chalk River Laboratories, John Vaughn and Tony Rodriguez are graduates from DeVry Institute. John is a recent graduate, but Tony came to the RF group from the NSLS Control Room.

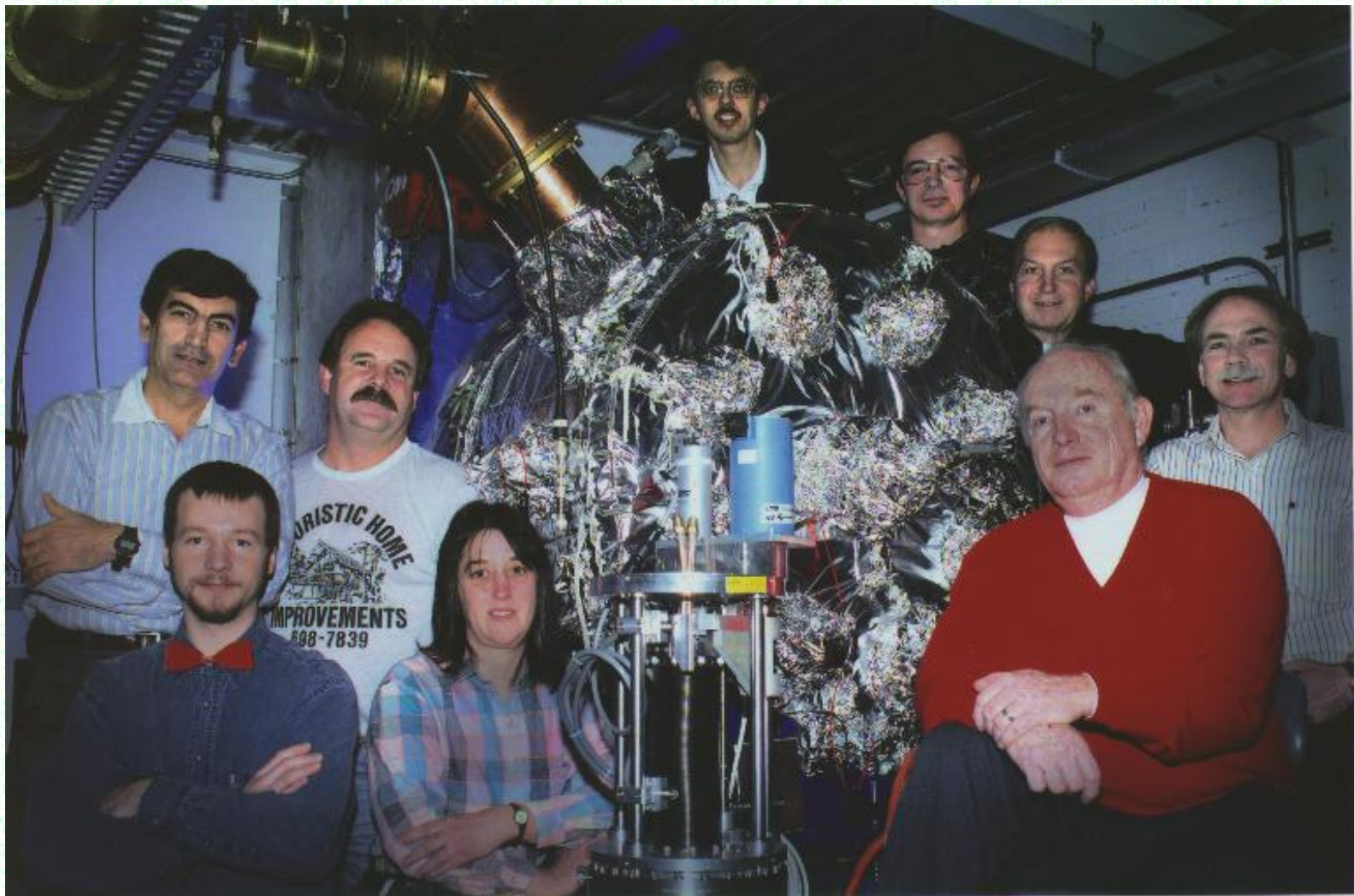
A large portion of the tasks required to be done are mechanical in nature and Payman Mortazavi is the cognizant mechanical engineer with Jimmy Newburgh providing the technician support. Tasks include maintaining cooling systems, mechanical drives, and dampening probes for the seven accelerating cavities in the NSLS RF systems. Payman also is a long-time employee of the Light Source. Jimmy came to the Light Source in 1979.

Numerous changes in the RF system have been made by this group over the last ten years. New accelerator cavity design has been implemented in both the booster and X-Ray rings. Two additional accelerating cavities with appropriate RF high power driving systems have been added to the X-Ray Ring system. The peak power capability in one of these systems has been doubled. The original output RF power amplifier utilized an RCA tetrode. These have been replaced with EIMAC tetrodes and associated tuning circuitry. These changes have increased the peak current capability in both accelerating rings as well as a substantial increase in reliability. The addition of a fourth harmonic debunching cavity and associated power amplifier has increased the beam lifetime in the VUV Ring.

During the May 1998 shutdown one of the accelerating cavities in the X-Ray Ring will be replaced by a new cavity having substantially better cooling capacity as well as peak RF input power capability. It also eliminates potential water-to-vacuum faults that exist in the present cavities.

In summary, the RF group is dedicated to keep the beam in the machine and minimize downtime. Their success rate is rather remarkable when one considers the fact that an un-interruptible 300,000 Watts is required to keep beam in the X-Ray machine.





*(Left front) John Vaughn, Gloria Ramirez.*

*(Left, back) Payman Mortazavi, Jimmy Newburgh.*

*(Clockwise from top) Tony Rodriguez, Nathan Towne, Manny Thomas, Roy D'Alsace, and John Keane.*

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 23, 1998

---



## A User's Perspective

[Joel D. Brock](#)

*Cornell University*

*UEC Chair*

Although it's only early February, my term as Chair of the Users' Executive Committee (UEC) is, in reality, almost over. Most of my official duties and obligations have been completed or are scheduled, organized and will progress through completion more or less on auto-pilot. Looking back on the past two years, I marvel at the changes. When I agreed to stand for election to the UEC, it never occurred to me that I might become its chair. And, even in my wildest dreams (or nightmares), I would not have predicted the dramatic events which would soon engulf the entire Laboratory. By anyone's reckoning, the last eighteen months have been quite tumultuous for both Brookhaven National Laboratory and for the NSLS. The combination of the change in BNL's management and the Basic Energy Sciences Panel which reviewed the four DOE funded synchrotron sources (The Birgeneau Panel) have kept the UEC occupied for most of my tenure.

I am very pleased to be able to report that both the NSLS and BNL are emerging from these challenges strong and poised for future growth. The user community owes a collective debt of gratitude to the Chairman of the NSLS, Michael Hart. Michael skillfully managed NSLS's participation in the Birgeneau Panel's review. The charge to this committee requested that they consider the impact of closing one or more of the DOE funded synchrotron sources. The committee included members who had no familiarity with the NSLS or, in some cases, any synchrotron source. The report is highly laudatory of the NSLS, recommending increased operating funds, additional funds to expand the number of user support personnel and additional funds to pay for capital improvements to the beam lines. (You can view the executive summary from the [NSLS Home Page](#) under "News".) Michael Hart masterfully managed NSLS's participation in this review process. The user community is also to be congratulated for their contributions to this review process. In the end, the best justification of the NSLS is the science done by its users.

At the recent NSLS Town Meeting and the Users' Executive Committee meeting the following day, I had my first opportunity to meet a member of the new management team of BNL, Dr. Peter Paul. I am pleased to be able to report that I was favorably impressed. Dr. Paul engaged the UEC in an open and frank discussion of both the issues and the opportunities both the NSLS and BNL are presented with. I left the meeting with the strong impression that the new Laboratory management is firmly behind the NSLS and willing to lobby both DOE and Congress on our behalf. Furthermore, Dr. Paul demonstrated a strong interest in helping to drive the cutting edge science done at the NSLS by promoting the development of new sources. It is my hope that the NSLS user community, the NSLS management and the BNL management can continue to forge a new and strong working relationship. Obviously, this will take time. However, the survival of our facility depends upon it.

Looking to the future, I am very pleased with the job the Organizing Committee has done getting ready

for this year's Annual Users' Meeting. There are literally thousands of details to attend to before a meeting this size. The Organizing Committee takes care of all of these every year. We owe them a tremendous debt of gratitude. This year's meeting has the promise of being one of our best ever.

In closing, I would like to make one final observation. During the last two years I have been struck, time and time again, by how small the number of users who make active contributions to the NSLS is. This is not the fault of the NSLS administration who routinely has to "shake the bushes" in order to find Proposal Study Panel (PSP) members or to obtain examples of exciting science done at the NSLS for the consumption of our DOE grant officers or Congress. Unfortunately, many users behave as if the appropriate relationship between the user community and the NSLS administration (or the DOE) is an adversarial one. I completely reject this. The users, the NSLS administration and the BNL management are all on the same side. And, the more I interact with these people, the more I am convinced that they also feel this way. Clearly, lack of communication and erroneous assumptions frequently poison the waters. We must remain on guard against this. Frequently, neither side appears to have an accurate picture of the very real pressures and constraints the other is under. I urge everyone, the users, the NSLS administration and the BNL management to approach the continuing challenges we all face in the next several months with a spirit of cooperation and trust.

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 23, 1998

---



## X-Ray Ring Report

[Roger Klaffky](#)

*X-Ray Ring Manager*

On Sunday morning November 23, 1997, two days prior to the end of 1997 x-ray user operations, an electrical failure occurred in the 480 Volt electrical power wiring that connects the power from the four substations in the middle of the NSLS courtyard to equipment inside the building. The fault occurred in a cluster of plastic (PVC) conduits under the concrete floor of Mechanical Equipment Room 2 (MER2). The conduits connect substations 2 and 3 to a junction box in MER2 used to splice the 3 or 4 wires in each conduit to wires leaving this box to other locations in the building. There was a complete melting of all the conductors in one of the conduits from substation 3 over a several foot length, and a partial resultant melting of a conductor in an adjacent conduit from substation 2. There was no damage to any equipment other than the wiring itself. Temporary power was restored to all areas by 6 p.m. on November 27, allowing planned shutdown work to proceed on the X-Ray and VUV Rings. Because of the observed damage, it was considered necessary to replace and reroute all of the wiring in the damaged conduit array using an alternate cable tray installed over the building roof. This also required that a new junction box be installed. This permanent repair was completed December 15. A committee chaired by [John Keane](#) (NSLS Electrical Systems Manager) is presently investigating the causes of the electrical failure.

During the November 18-23, 1997, 2.8 GeV operations period and throughout the December shutdown, there was continued progress in completing 2.8 GeV beamline shielding. At the present time there are 42 beamlines approved for 2.8 GeV operation and one beamline in a self-monitoring mode. Between now and the March/April 2.8 GeV operations period there will be studies periods set aside so that all beamlines can be approved for operation.

There was a considerable amount of work performed on various NSLS systems during the December 1997 shutdown. A primary aluminum water system heat exchanger was replaced and a plugged high pressure copper water system heat exchanger was disassembled and cleaned. The aluminum water system control valve was rebuilt and the three de-ionizer systems were relocated to make them more accessible. Additional 1-inch diameter experimental water spigots were installed at several locations on the x-ray floor. The X-Ray Ring RIA and RIB interlock chains were physically separated in new conduits running around the ring. The Critical Device Overtemperature System (CDOS) was installed in the Control Room and the installation of Klixon temperature sensors at the output of existing Proteus flow meters continued. The CDOS will dump X-Ray beam if the cooling water temperatures from critical ring components (aluminum chamber, crotch, front end components, etc) exceed specified setpoints. Presently there are interlocks that will dump the ring if the global aluminum temperature exceeds 105 degrees F or if the power supply water exceeds 115 degrees F. Towards the goal of 438 mA operation at 2.584 GeV, new Be windows were installed on X3B, X16A, X16B, X16C, X18A, X18B, X20B, X22B, X27A and X27C. There are three Be windows that still need to be replaced: X10B, X11A, and X22C. Of these, X11A must either be leak-checked or replaced before operating above 350 mA. This will occur



early in February. The other two windows can operate at high current before they are replaced during the Spring shutdown. New safety shutters rated for operation up to 500 mA were installed in the X21 and X25 frontends during the shutdown. The final requirement for high current operation is that a simulation and testing be completed before the start of high current operations to confirm a lifetime projection for the 10 degree crotch assembly.

During the April 1998 shutdown, a new all-copper cavity will replace the existing RF2 cavity. A new copper coupling loop brazed to a beryllia ceramic window will allow the transmission of up to 150 kW into the cavity. Two of these cavities have been ordered. The first was delivered to BNL on September 12 and fabrication of the second cavity will be completed in the next few months. The first cavity has undergone extensive testing in the NSLS RF test room. The cavity has been baked and achieved a pressure of  $1 \times 10^{-9}$  Torr while being powered to 60 kW. The new window has been tested at power levels of 120-150 kW. All higher harmonic modes have been damped to levels lower than the other RF cavities using antennae. Because of RF heating of a stainless steel spool piece, it will be replaced with a copper spool piece in February. The RF resonant frequency was successfully corrected by replacing the large Helicoflex vacuum seal with a larger cross-section seal. In preparation for the Spring shutdown, a 1.5 foot deep hole was cut through the X-Ray Ring shield wall for the installation of a transmission line for the new cavity. A new hole was required because the RF1 and RF2 cavities are being split to provide room for the possible future installation of another IVUN in this straight section (X9).

Another major task for the Spring shutdown will be the installation of the new X17 superconducting wiggler. The wiggler is being tested in a horizontal cryostat at Oxford Instruments the first week of February. NSLS engineers will be present at these performance tests which consist of demonstrating required magnetic field specifications, training within a prescribed number of quenches, appropriate helium consumption, quench protection and recovery systems. The wiggler will then be shipped in late February or early March. Acceptance tests will take place in Building 902 where the goal is to ramp the wiggler to full current without quenching. In preparation for the new wiggler, a loading dock was installed outside the present location of the X17 refrigerator for the delivery of 500 liter dewars. Double doors were installed. Duct work was removed to make room for wiggler power supplies. Before the Spring shutdown, a raised steel floor will be installed for the power supplies and water cooling will be hooked up to these supplies.

In addition to replacing the X17 superconducting wiggler, the X17 straight section chamber downstream of the wiggler will be replaced. A water-to-vacuum leak developed in the cooling channel in the horizontal "speed bump", which protects X17 front end components from excessive thermal loading. The leak first started November 21, 1997, a few days before the power outage, and climbed into the  $5 \times 10^{-7}$  Torr region in December. When the water pumps were turned back on at the end of December, a large leak opened up. In order to resume operations on schedule in January, the chamber was baked out and a pump installed on the water channel to keep it evacuated during ring operation. The wiggler will not operate until a new chamber is installed in April 1998.

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date :



February 23, 1998

---

# VUV Ring Report

[Stephen L Kramer](#)

*VUV Ring Manager*

The VUV Ring was running at record breaking levels prior to the winter 1997-1998 shutdown. The lifetime was exceeding 340 minutes at the 500 mA level, yielding an average operating current over a 24 hour period of 568 mA. This was achieved despite vacuum leaks in a bellows and a front-end mask. These were temporarily sealed with epoxy until the shutdown, when they were repaired permanently. The 4th harmonic RF system was providing longer and more stable bunch lengths, also contributing to this increased lifetime. Similarly shorter injection times and improved beam stability during operations was achieved as a result of continued improvement in monitoring and correcting problems as they occur.

The winter shutdown was primarily scheduled to improve the shielding of the VUV Ring. During injection into the ring, the radiation resulting from injection losses required the users to stay away from their beamline front end components and caused enough radiation to the Phase II offices of the C wing that the rooms required posting of the potential levels that could occur. The shielding installed during the winter shutdown should reduce the radiation levels on the experimental floor for the beamlines U1 through U6, as well as to the offices in the C wing. This was done using heavy concrete (iron ore loaded) blocks under and around the lead shielding. This will further attenuate the gamma ray levels and reduce the neutron levels resulting from gamma ray interactions in the shielding material. The existing lead shielding was improved in thickness and in the completeness of its coverage. The shield wall supports were also improved, reducing the potential risk from falling lead bricks.

The initial results of this shielding indicated an improvement in the radiation levels on the experimental floor and to some of the offices. However, the return from a major shutdown like this winter's has a much higher injection frequency and therefore more radiation exposure, making it difficult to evaluate the real reduction in radiation exposure compared to normal operations. The real effect of the radiation attenuation resulting from this work will continually be studied and will be reported at a later time. Planning is underway to continue this improvement in shielding around the rest of the VUV Ring for the winter 1998-1999 shutdown. Once real progress has been made on reducing the radiation levels around the entire ring, a continuation of the studies on the Top-Off Method of Injection (TOMI) will begin with the goal of proving the usefulness of this method for the users in terms of increased photon beam stability and brightness. The reason the VUV Ring doesn't presently run with a beam brightness up to a factor of ten higher, is due to the reduction in lifetime it would cause. However, lifetime becomes a minor issue if TOMI can be perfected to such a point that it becomes the method of operation for the ring. In fact TOMI is the present method of injection but with injection currents of typically 500 mA every 4.5 to 5 hours, rather than 20 to 50 mA every 10 to 15 minutes.

During the shutdown, the access to the ring was used to make big progress in replacing the signal cables on the beam position monitor system. The old cables were the result of an evolution of the system in the past and were a source intermittent problems. The new cabling has less attenuation and fewer connectors



and should improve the reliability and repeatability of the orbit measuring system.

The turn-on of the VUV Ring, after the shutdown, had some difficulties resulting from scheduling problems arising from the electrical power problems that occurred prior to the shutdown last November (see [X-Ray Ring Report](#), in this issue). Despite those delays stored beam was achieved quickly and normal operating currents were achieved several days prior to scheduled operations. The return to pre-shutdown beam lifetime has been slow and appears to be due to poor vacuum in the U5 undulator and super period three, both were opened to air during the shutdown. The undulator vacuum chamber doesn't get much exposure to synchrotron light and therefore is subject to less beam induced gas desorption. To increase the rate of this desorption two additional injections and higher injection currents (up to 1 Ampere) have been instituted during the night shifts. This will continue until the vacuum levels and lifetime improve to within 10% of pre-shutdown values. Following the initial conditioning of the ring, the U12IR mirror was inserted and conditioned and alignment and commissioning of that new beamline is proceeding well. This should result in a major increase to the science performed on the VUV Ring in 1998 and beyond.

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 23, 1998

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# CALL FOR GENERAL USER PROPOSALS

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

**Monday, June 1, 1998 for scheduling September - December 1998**

## Prior to Submitting a Proposal

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

## Preparing Your Proposal

The same form is used for new proposals and for beam time requests against existing proposals. Follow the instructions on the proposal information sheet. All information must be typed or printed legibly. Be sure all of the required sections are completed and submitted at the same time. **MAIL OR FAX ONE COPY** of the proposal form, and any attachments to the NSLS User Administration Office. Only **one copy** is required - do not mail a hard copy or fax a second if you have already faxed one. **NEW: Do not send a Safety Approval Form.**

## Proposal Deadline

The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Monday, June 1 in order to be considered for the May - August cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on June 1. We encourage submitting new proposals by mail prior to the deadline. Beam time requests for active proposals will be accepted after the deadline, but will be allocated beam time only after requests received on time have been allocated. Late requests are not eligible for a rating upgrade if beam time could not be allocated to them.

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



## Additional Information and Forms

Blank proposal forms and instructions, a guide to the NSLS beamlines, and more information about the General User Program are available on the World Wide Web at [www.nsls.bnl.gov](http://www.nsls.bnl.gov), or by contacting E. Pinkston or L. Rogers at 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.

### PLEASE NOTE THAT SAFETY APPROVAL FORMS . . .

- ◆ Are now valid for one (1) year from date of approval.
- ◆ Must be submitted at least 1 week before the expected start date for your experiment.

If you are submitting a NEW proposal, do NOT submit a SAF - wait until you receive notification that you have been allocated beam time. Your allocation letter will contain a reminder for you to submit your Safety Approval Form before your experiment starts.

If you are submitting a BEAM TIME REQUEST against an existing proposal, you may already have a valid Safety Approval Form in place. Remember, they are valid for a year. If your previous form has expired or this proposal has not yet received any beamtime, then you will have to submit a Safety Approval Form before you experiment starts.

**If you need to find out about your SAF's status, contact: Andrew Ackerman, 516-344-5431, [ackerman@bnl.gov](mailto:ackerman@bnl.gov)**

[More information about NSLS Safety Approval Form policies.](#)

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### [March 1998 Table of Contents](#)

---

[NSLS Home Page](#)..... [BNL Home Page](#)

*The [NSLS User Administration Office](#) maintains this document.*

*[DISCLAIMERS](#) : Revised Date : February 28, 1998.*

# MARCH 1997 NSLS NEWSLETTER

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## IMPORTANT UPCOMING DATES

May 15, 1998

Deadline for submissions, July 1998 Newsletter

May 19 - 21, 1998

[NSLS Annual Users' Meeting and Workshops](#)

June 1, 1998 (because May 31 falls on a weekend)

[Deadline for General User Proposals](#)

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[NSLS Home Page](#)..... [BNL Home Page](#)..... [March 1998 Table of Contents](#)

The [NSLS User Administration Office](#) maintains this document. [DISCLAIMERS](#) : Revised Date : February 23, 1998

---