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Beamline X1B: Fraunhofer diffraction rings generated from a 20 μm pinhole by 215 eV (5.77 nm) photons. While lasers can be used to produce coherent radiation in the visible and near UV range, no source of intrinsically coherent radiation is currently available in the x-ray range. However, using a monochromator and a pinhole arrangement, synchrotron radiation can be made coherent. This is demonstrated by the existence of the diffraction rings in the image. The availability of coherent radiation in the soft x-ray region makes new experiments possible, which rely on interference effects. For example, speckle patterns can be generated and analyzed in the space and time domain, in order to obtain information beyond the statistical average on the morphology and dynamics of the sample. The image was recorded using a 2D position sensitive detector for soft x-rays with a pixel resolution of 20 μm x 80 μm and is shown on a logarithmic color scale.

S. Eisebitt, A. Karl, R. Scherer, W. Eberhardt (Forschungszentrum Jülich), M. Adamczyk, T. Tiedje (U. of British Columbia), and C. Pistonesi (U. Nacional del Sur)

ONE

INTRODUCTION

NSLS

INTRODUCTION

Michael Hart
NSLS Chairman

In FY 1998, following the 50th Anniversary Year of Brookhaven National Laboratory, Brookhaven Science Associates became the new Managers of BNL. The new start is an appropriate time to take stock of past achievements and to renew or confirm future goals.

During the 1998 NSLS Annual Users' Meeting (described in Part 3 of this Activity Report), the DOE Laboratory Operations Board, Chaired by the Under Secretary for Energy, Ernest Moniz met at BNL. By chance all the NSLS Chairmen except Martin Blume

The NSLS Annual Users' Meeting and the Laboratory Operations Board meeting held at BNL the same week made possible this unusual gathering of current and former administrators of BNL's National Synchrotron Light Source (NSLS). (from left) **Michael Knotek**, NSLS Chair 1985-1989, and now program advisor for Science & Technology in the Office of the Under Secretary of Energy; current NSLS Chair **Michael Hart**, who has held the post since 1995; current Deputy NSLS Chair **Samuel Krinsky**, who served as Acting Chair from 1989-1990; **Denis McWhan**, NSLS Chair 1990-1995, and now BNL's Associate Director for Basic Energy Sciences; **Arie Van Steenbergen**, who headed the NSLS Construction Project 1977-1982, and who is now actively retired from BNL; and **John McTague**, NSLS Chair 1982-1984, and now Vice President, Technical Affairs, Ford Motor Company. Not shown is **Martin Blume**, who was Acting NSLS Chair 1984-1985 and is now Editor-in-Chief of the American Physical Society.

(acting NSLS Chair 84 – 85) were present as recorded in the picture. Under their leadership the NSLS has improved dramatically:

- The VUV Ring current has increased from 100 mA in October 1982 to nearly 1 A today. For the following few years 10 Ahrs of current were delivered most weeks - NSLS now exceeds that every day.

- When the first experiments were performed on the X-ray ring during FY1985 the electron energy was 2 GeV and the current up to 100 mA – the X-Ray Ring now runs routinely at 2.5 GeV and at 2.8 GeV with up to 350 mA of current, with a very much longer beam half-life and improved reliability.

- Starting in FY 1984 the proposal for the Phase II upgrade, mainly for a building extension and a suite of insertion devices and their associated beamlines, was pursued – the promises were delivered in full so that for some years now the NSLS has been running with two undulators in the VUV Ring and three wigglers and an undulator in the X-Ray Ring. In addition two novel insertion devices have been commissioned in the X13 straight.

- At the start of FY 1998 the NSLS welcomed its 7000th user – attracted by the opportunity for pursuing research with high quality beams, guaranteed not to be interrupted by “delivery failures”, and welcomed by an efficient and caring user office and first class teams of PRT and NSLS staff.

R & D have lead to the possibility of running the X-Ray Ring at the higher energy of 2.8 GeV. **Figure 1** shows the first user beam, which was provided thereafter for half of the running time in FY 1998. In combination with the development of narrow gap undulators this mode opens the possibility of new undulators which could produce hard X-rays in the fundamental, perhaps up to 10 keV.

On 27 September 1998, a low horizontal emittance lattice became operational at 2.584 GeV. This results in approximately a 50% decrease in the horizontal beam-size on dipole bending magnet beamlines, and somewhat less of a decrease on the insertion device lines. The beam lifetime is not degraded by the low emittance lattice. This represents an important achievement, enhancing for all users the x-ray ring brightness. The reduced horizontal emittance electron beam will produce brighter x-ray beams for all the beamlines, both bending magnets and insertion devices, adding to other recent increases in the X-Ray ring brightness. During FY 1999 users will gain

Figure 1: The first week of user beam time at 2.800 GeV.

Figure 2. The source profile with “normal” and reduced emittance magnet lattice.

experience of the new running mode and plans are in place to do the same at 2.8 GeV during further studies sessions. Independent evidence of the reduced emittance is shown in **Figure 2**. This is a pinhole camera scan showing the X-ray beam profile, obtained on the diagnostic beamline X28.

Finally, work has begun to update and refine the proposal of the Phase III upgrade endorsed by the Birgeneau panel and BESAC last year. With the whole NSLS facility in teenage years and with many demonstrated enhancements available, the time has come to herald in the next stage of life at the Light Source. ■

NSLS

USERS' EXECUTIVE COMMITTEE

John B. Parise
SUNY @ Stony Brook
UEC Chair

The UEC represents the interests of the users to NSLS and BNL Administrations as well as to the community outside BNL. Those interests include continued access to reliable X-ray and UV beams and to a working environment supportive of their research. Almost 2400 users carried out experiments at the NSLS during 1998. "Users" are defined as researchers who actually showed up at the User Administration Office and were given access to the experimental floor to perform experiments on a beamline. With over 97% reliability on both X-ray and UV storage rings, most users are satisfied that the NSLS can provide what they need to fulfil their research obligations. More and more of these users however, are regarding the NSLS as an extension of their home laboratory and this raises issues of access to resources and infrastructure. In order to transmit those interests as "cleanly" as possible, the UEC has studiously cultivated its independence from the NSLS Administration, while closely coordinating efforts with it to improve services for the user. The UEC provides independent advice with minimal influence from the Laboratory or Light Source.

There are several avenues for users to express opinions and suggestions, on subjects as varied as the availability of consumables for experiments to the impact of changing the characteristics of the storage rings. Examples include prompting the NSLS to send out regular news announcements to keep the user community informed about the general goings on. A problem of security for privately ordered specialty gas bottles was brought to the attention of the UEC and a commitment to provide a locked and controlled space under management of the operations coordinator was made by the NSLS. Several Special Interest Groups (SpIGs) serve specific experiments or techniques and information on these can be found at the following web address - <http://www.nsls.bnl.gov/UserGrp/spiginfo.html>. The SpIG-representatives, as members of the UEC, can transmit a user's concerns. These representatives were elected in an electronic ballot for the first time in 1999 thanks to the efforts of Malcolm Capel (BNL - Biology) and Carl Zimba (NIST). Quarterly town meetings are held in the NSLS

seminar room and this provides another opportunity for users to voice users' concerns. While most of the attendees at these meetings are frequent users, local beamline contacts and NSLS staff, I urge everyone who can make these meetings to do so or to email concerns to any UEC member or SpIG representative. Again, details are posted on the NSLS web site. With the NSLS and BNL staff in attendance, there are opportunities for immediate discussion and feedback. The UEC then meets with NSLS and BNL staff and management to discuss relevant issues after the Town Meeting.

The main topics at recent UEC meetings have been the fate of the weekend food service and participation in a Community Advisory Committee (CAC) formed to advise the laboratory on its operations and their potential impact on the broader Long Island community. The users attending the town meetings were able to assure BNL Management of their strong desire to have access to food on the BNL campus during weekends, and as a result of this input Management established expanded food service hours on both Saturdays and Sundays at the Center. As for maintaining a reasoned voice in the CAC, the UEC is grateful to Jean Jordan-Sweet (IBM) for advancing our interests from the early planning stages of the advisory group. Peter Stephens (SUNY @ Stony Brook, past UEC-chair) has recently joined her as an alternate UEC-representative. The relationship between BNL and the community is a delicate one. It is one with the potential to help or harm the interests of NSLS users. We all hope the independent voice of the UEC will be heeded in this forum.

Many users operate research programs at more than one light source, and the four DOE-funded synchrotrons (NSLS, APS, SSRL and ALS) have similar interests in terms of maintaining viable and expanding research programs. In recognition of their common interests in synchrotron radiation research, the chairs of the User groups at NSLS, APS and SSRL co-signed a letter urging the implementation of a BESAC-report recommendation for increased funding for synchrotron research. The letter read, in part: "Our communities are growing rapidly, and demand for beam time continues to increase at each

facility. . .we'd like to offer the assistance of our community by providing full support of DOE efforts to acquire the necessary additional funds to fully implement the Birgeneau-Shen recommendations and continue support for other important areas in the ER program." In addition to such joint letters, further user group coordination on issues such as funding and joint meetings of the light source chairs are in the best interests of the whole synchrotron community.

The Annual Users' Meeting, funded largely out of registration and vendor fees, is the most visible function organized by the UEC. Along with celebrating scientific accomplishments, the meeting serves to bring changes and proposed changes, which have the potential to alter the physical and intellectual environment in which the users operate, to their attention. This meeting is an opportunity for the UEC to devise workshops, invite overview talks from leaders in the arenas of science and science administration, and to set a tone for the year to come by electing representatives to join the UEC. The 1998 meeting was a great success thanks mainly to the efforts of workshop organizers, and particularly John Hill who attracted one of the best slate of speakers on record for the one-day Users' meeting. Excellent presentations emphasized science, the future of the NSLS, and DOE funding initiatives. The need to improve infrastructure and user support were common themes of the talks by

John Marburger, Director of the BNL, Michael Hart, Chairman of the NSLS, Robert Marianelli, Director of the Division of Chemical Sciences for the Office of Basic Energy Sciences (BES) and Dr. Ernest J. Moniz, Under Secretary of Energy at the US Department of Energy. A complete report appears in Section 3 of this Activity Report. Apart from the annual meeting, the UEC from time to time uses its funds to further the science agenda and advertise the capabilities of the Light Source. To this end the UEC was among the sponsors of the Montauk conference on Inelastic X-ray Scattering in November 1998 and helped with other activities which raised the profile of the NSLS at other conferences.

I am grateful to all the members of the UEC (see photograph with this article), to the User Administration Office and to the staff of the NSLS for all their efforts over the past year. Some, such as the restoration of power during the unscheduled shutdown in December 1997, were Herculean efforts. More typically they involve the steady production of high quality beams. They also include patient implementation of DOE directives, and mollifying users who run afoul of these, the planning of meetings, the compilation of this Activity Report, and many other efforts impacting every user. Whether they are aware of them or not, the user community depends on these efforts to produce the science presented in this annual report. I am grateful to you all. ■

Users' Executive Committee and
Special Interest Group Representatives

Seated: Malcolm Capel (BNL-Biology), Lisa Kelly (U. of Maryland); Michael Dudley (SUNY @ Stony Brook); John Parise (SUNY @ Stony Brook); Jean Jordan-Sweet (IBM); Luz Martinez-Miranda (U. of Maryland); Carol Hirschmugl (U. of Wisconsin); Mark Chance (Albert Einstein College of Medicine).
Standing: Eva Rothman (NSLS); George Cody (Carnegie Inst. of Wash.); Joel Brock (Cornell U.); Steve Whisnant (U. of S. Carolina); Paul Stevens (Exxon); Gwyn Williams (NSLS); Larry Carr (NSLS); Barbara Illman (U. of Wisconsin/USDA Forest Products Lab).

Beamline X4A: The HIV lipid envelope, which is derived from the membrane of the host cell, hides most of the infectious virus particle from immune surveillance. Only two viral proteins protrude beyond the protective lipid bilayer, the exterior envelope glycoprotein, gp120, and its membrane-spanning partner, gp41. Of these, gp120 appears to occlude much of the gp41 ectodomain and is the target of virtually all neutralizing antibodies. Because of the pivotal role of gp120 in receptor binding and interactions with neutralizing antibodies, we undertook an investigation of its atomic structure. The figure shows the structure of the gp120 ternary complex in its biological context. The center of the figure depicts the gp120 ternary complex in ribbon representation, with core gp120 (red) caught in the act of binding to the CD4 receptor (yellow) by the neutralizing antibody, 17b (heavy chain, blue; light chain purple). The pivotal Phe 43 residue CD4 is also shown, at the nexus of the gp120-CD4 interface. The target cell membrane (cyan), domains 3 and 4 of CD4 (yellow ovals), chemokine receptor (green), viral spike and virion (pink) are also shown for reference.

P. D. Kwong (Columbia U.), R. Wyatt and J. Sodroski (Dana-Farber Cancer Institute), and W.A. Hendrickson (Columbia U. and the Howard Hughes Medical Institute)

TWO

SCIENCE HIGHLIGHTS

SCIENCE HIGHLIGHTS

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POWDER DIFFRACTION

Structure Solution from Powder Diffraction at the NSLS

Peter W. Stephens (SUNY Stony Brook, Physics & Astronomy Department)
David E. Cox (BNL Physics Department)

INTRODUCTION

Over the last few years, there has been a rapid growth in the development and application of methods for the *ab initio* solution of unknown crystal structures from powder diffraction data. Although conventional laboratory techniques have been successful in solving many heavy-atom inorganic structures, the most exciting recent advances have centered around the use of synchrotron x-ray powder data for the solution of molecular and organic structures, and framework structures such as zeolites. For structure solution problems, synchrotron radiation offers two outstanding advantages: the very high instrumental resolution that can be achieved with the use of a crystal analyzer mounted in the diffracted beam (about an order-of-magnitude better than that of a conventional diffractometer), and the feasibility of using samples loaded in thin-walled capillaries (which can be up to 2 mm in diameter depending on the sample transparency) without any loss in resolution. Other significant advantages are accurate peak positions, improved peak-to-background discrimination, wavelength tunability, well-characterized peak shapes, and the ease with which data can be collected at low temperatures in order to minimize thermal disorder.

Most *ab initio* powder studies at the National Synchrotron Light Source (NSLS) are carried out at the bending-magnet beam lines X3B1 and X7A, which have a long and productive history in this field. In a typical experiment, a double-crystal Si or Ge (111) monochromator is used in conjunction with a flat Si or Ge analyzer crystal, usually in the wavelength range 0.7-1.3 Å. The capillary sample is rotated or rocked during data collection (which in favorable circumstances might extend to a d-spacing of 0.8 Å), and the raw data converted to a diffraction histogram. The standard procedure at this point is to: (1) index the unit cell using one of several powerful auto-indexing programs, (2) extract the structure factors, (3) solve at least part of the structure using traditional methods, and (4) locate the rest of the atoms by a combination of Rietveld refinement and difference Fourier synthesis.

At stage (3) the problem is basically reduced to a single crystal structure determination with a limited and noisy data set, and can be tackled by the application of traditional Patterson and direct methods. The powder technique has been successfully exploited for a number of structures containing 30 to 50 atoms in the asymmetric unit, and in the future may perhaps be extended to 70-100 atom problems. Some examples are described below.

Figure 1: Structure of Uranyl Methylphosphonate, from Reference 1, determined by powder diffraction data.

Although the powerful multiple anomalous diffraction techniques increasingly being used for protein crystallography cannot be exploited in the same way for powder data, anomalous scattering can nevertheless be applied in principle to the solution of unknown structures through the generation of partial Patterson maps of the anomalous scatterer, and a test case is described below.

The most recent developments in the application of powder techniques have focused on more novel approaches to the solution of organic compounds, molecular crystals and other light-atom structures such as zeolites. These approaches include Monte Carlo procedures, simulated annealing techniques, global optimization based on a genetic algorithm, entropy

maximization and maximum likelihood, Fourier recycling with a specialized topology search specific to zeolites, and the use of periodic nodal surfaces. Several of these methods operate in direct rather than reciprocal or Patterson space, and rely on random displacements of structural fragments to generate an acceptable fit to the diffraction pattern, thereby avoiding the necessity of extracting structure factors in step (2) above.

A one-day workshop, "Using Powder Data to Solve Crystal Structures," reviewing this area of crystallography was held at the NSLS 1998 Annual Users' Meeting. There were eighty participants, testifying to the exceptionally high level of interest in this topic.

Figure 2: MEM vs. Fourier maps of the Sr partial Patterson function in SrSO_4 , determined by anomalous scattering.

TRADITIONAL METHODS

Some of the traditional methods used to phase single-crystal diffraction patterns have been adapted to powder data sets, and are in productive use. Structure solutions from powder data are never as routine as single-crystal experiments, because the collapse of the diffraction pattern into one dimension generally requires close human supervision and evaluation of the quality of the raw data, determination of the unit cell, extraction of integrated intensities, etc. Patterson methods are very powerful for cases in which there are a few heavy atoms. One recent example is the structure of uranyl methylphosphonate,

$\text{UO}_2(\text{O}_3\text{PCH}_3)_2$, illustrated in Figure 1.^[1] This layered compound is of interest because metal phosphonate systems can be synthesized with a variety of functional groups, and are able to self-assemble into lattices with interesting potential materials properties, in this case, luminescence. The approximate structure was solved from laboratory x-ray data using Patterson methods, and completed by successive Fourier difference calculations, but synchrotron data were necessary to obtain a stable refinement.

Direct methods constitute the current workhorse technique for phasing single crystal diffraction patterns, based on statistical relationships of the phases and the intensities of combinations of three or more diffraction peaks. Powder data would seem to be ill-equipped to meet the condition of the number of independent observations much greater than the number of atom coordinates for any but the simplest problems, but the combination of high resolution experiments with advances in software has increased the power of this technique. Several recent examples of newly-solved structures, drawn from this year's experimental abstracts, include Rb_3PbCl_5 , the charge-transfer complex $[\text{Ni}(\text{dmit})_2]^{2-} \text{DPD-Me}^{2+}$, and the alkali salicylates $\text{C}_7\text{H}_5\text{O}_3\text{Rb}$ and $\text{C}_7\text{H}_5\text{O}_3\text{K}$.

In contrast to macromolecular crystallography, anomalous scattering techniques have not yet been used for *ab initio* structure solution from powder data. The key to the use of anomalous dispersion in phasing single crystal diffraction patterns lies in the fact that it produces a different intensity for the two members of Bijvoet pairs hkl and $\bar{h}\bar{k}\bar{l}$. The technique is not directly transferable to powder diffraction, because all such pairs of reflections are superimposed, but it is nevertheless possible in principle to determine the difference Patterson density of the anomalously scattering atom from two measurements, close to and far from the absorption edge. The application of this technique to the test case of SrSO_4 is illustrated in Figure 2, which shows the partial Patterson density of Sr.^[2] A notable feature of this work is the obvious superiority of maximum entropy methods over conventional Fourier synthesis in the reconstruction of the density maps.

Figure 3: Crystal structure of RbC_{60} and $\text{Na}_2\text{RbC}_{60}$ polymeric ground states. From Reference 3.

SIMULATED ANNEALING TECHNIQUES FOR MOLECULAR SOLIDS

In the cases of organic solids, one can usually make a fairly good guess of the molecular structure from chemical principles (bond lengths and angles) and so there are only a few degrees of freedom in determining how the molecules might pack into a lattice. Organic molecules typically occupy the general position of a lattice, and so a structure is specified by six real numbers to locate the position and orientation of the molecule's center, plus however many internal degrees of freedom are described by torsion angles. In these techniques, the agreement between a calculated diffraction pattern and the observed data is regarded as an energy to be minimized, and exhaustive searches or simulated annealing algorithms are applied to determine the best fit between model and data. There are several interesting new results from recent experiments at the NSLS.

The developing story of polymerization of C_{60} has been full of surprises. In 1994, experiments at the NSLS showed that RbC_{60} and KC_{60} had different low-temperature states depending on their thermal history. If cooled slowly, they polymerized spontaneously into the ground state structure illustrated in Figure 3a, with two single bonds (a [2+2] cycloaddition) linking adjacent fullerenes into chains. If quenched, the fullerenes paired off into dimers; surprisingly, it was found that the dimers were linked by a single carbon-carbon bond, different than the polymer. More recently, it was found that if the fullerene charge state is increased to 3-, in Na_2RbC_{60} or Na_2KC_{60} , it has a polymeric ground state (Figure 3b) connected by the same single bond as the singly charged fullerenes in quenched $Rb_2(C_{60})_2$ and $K_2(C_{60})_2$.^[3] These results imply that the charge state has a crucial effect on determining the available routes of interfullerene bonding. In this work, the fullerenes were previously known to be centered at special positions of the crystal lattice, but the

orientation had to be determined in order to find the plausible bonding geometry. The high angle part of the diffraction pattern suffered from such severe peak overlap that no progress could be made with extracted intensities, but the problem could be attacked by comparing the entire calculated diffraction profile. This would only work for a molecule that largely retains its rigid identity in different crystal structures, but the successful refinements prove that this premise is correct.

A very different example comes from the chemical industry. Amoco technology produces over four million tons annually of purified terephthalic acid (1,4-benzenedicarboxylic acid). Understanding the natures of terephthalate complexes of catalyst and corrosion metals will lead to process insights and improvements. Nothing, however, is known about the solid state structures of such complexes, because they are intractable solids, available only as powders. Despite the importance of this problem, there has been little progress from applying conventional crystallographic techniques to the powder patterns of transition metal terephthalates. There are a great number of possible metal-carboxylate binding modes, and so it is

worthwhile to develop skills and techniques for solving the structures of such coordination compounds, and to gain insight into how terephthalate anions pack in the solid state. Toward that end, J.A. Kaduk at Amoco has studied a number of terephthalate salts of non-transition metal cations. Combinations of Monte Carlo simulated annealing, molecular mechanics, and crystallographic techniques have proven successful in obtaining the structures of such salts. The structures of dilithium, disodium, and diammonium terephthalates have been solved *ab initio* using synchrotron powder data and simulated annealing software from Molecular Simulations Inc., and can be compared to the structure of dipotassium terephthalate, determined using conventional single-crystal techniques. The structures of the three alkali salts are shown in Figure 4, which illustrates large differences between the structures of these closely related compounds. For example, the terephthalates in the Li, K, and ammonium salts are nearly planar, but the carboxylates are rotated approximately 20° out of the ring plane in disodium terephthalate. These four structures provide enough of a "database" to begin to understand the

Figure 5: An assortment of molecules whose crystal structures have been determined by *ab initio* powder diffraction techniques. Circular arrows indicate molecular torsion angles that must be determined experimentally.

energetics of these structures. Such understanding is proving useful in determining the structures of more process-related salts.

Simulated annealing techniques, both as developed by users and in commercial software, and with varying degrees of input from calculations of the conformational and packing energetics, have been applied to first determine the solutions of a large number of different molecular solids. The range of complexity is illustrated in the molecular diagrams in Figure 5. More details on all of these structures are available in the experimental abstracts from beamlines X3B1 and X7A.

It is worth pointing out that synchrotron data is often crucial for solving organic structures, not only because it yields better structure factors, but because it permits determination of the correct unit cells and space groups. The systematic errors inherent in a laboratory Bragg-Brentano experiment, particularly peak shifts due to the penetration of x-rays into weakly absorbing samples, make it difficult to index the laboratory patterns. One often thinks of the determination of the unit cell as mechanical, but it is crucial to obtaining the correct structure. Furthermore, the high angular resolution and high signal to background ratio of data sets usually collected with synchrotron radiation are of great importance in the determination of systematic absences and therefore of the correct space group.

FRAMEWORK COMPOUNDS

Framework compounds such as zeolites are very widely used as catalysts, adsorbents and ion-exchangers in industrial processes. The catalytic activity and selectivity of zeolites depend upon active sites which are accessed by reactant organic molecules through pores of about the same size as the molecules. Understanding zeolite structures is thus of utmost importance in optimizing the properties of these materials for industrial use. Since most zeolites are synthesized via the use of organo-cation templates under hydrothermal conditions which typically yield crystals less than 5 microns in size, powder techniques are vital in structural studies, and many such structures have been successfully solved by industrial and academic groups in the past decade. The techniques to solve zeolite structures from powder data are somewhat different than molecular structures. Instead of a distinct molecule which can be moved around in the unit cell (by computer), one has a highly connected framework, with a large number of tetrahedrally coordinated (Si,Al) atoms. It is therefore necessary to generate and test a number of different framework topologies against the experimental data, and several recently developed algorithms have extended the utility of these techniques.

A striking recent example is the structure of a new small-pore zeolite, ERS-7, with the approximate

Figure 6: The 17-sided 'picnic-basket' cage element in the structure of the new zeolite ERS-7, determined by powder diffraction.

composition $\text{Na}_x\text{R}_y(\text{Si}_{1-z}\text{Al}_z)\text{O}_2$, where R is the template ion, N,N-dimethylpiperidinium and $x \sim 0.03$, $y \sim 0.09$, and $z \sim 0.1$. The correct structure was identified as one of 373 unique framework topologies generated by a simulated annealing technique using MSI software, and was verified by Rietveld refinement and lattice energy minimization calculations.^[4] The orthorhombic framework has twenty atoms in the asymmetric unit, and consists of 17-sided cages with a "picnic-basket" shape connected by an eight-membered ring system such that the ring channels run through the "handles" of the picnic basket, depicted in Figure 6.

Many groups are actively seeking new zeolites with even larger pores, consisting of rings of twelve or more tetrahedral atoms, for catalysis or absorption on large organic molecules. Materials with a high-silica content are particularly desirable because of their high acidity and thermal stability. The structure of a novel compound with a 14-membered ring, CIT-5, was recently determined at the NSLS by an iterative process of model building, distance-least-squares refinement of the model parameters, and comparison of the simulated and experimental patterns until the closest match was found^[5]. Rietveld refinement was carried out on this structure, which is orthorhombic with 5 Si and 8 O atoms in the asymmetric unit, and is composed of one-dimensional extra-large pores of nearly circular cross-section (Figure 7).

Another interesting example is the *ab initio* solution of the structure of two closely-related large-pore high-silica molecular sieve materials, SSZ-44 and SSZ-35. The first was solved by application of the Fourier recycling and topology search (FOCUS) method,^[6] followed by Rietveld refinement. The structure is monoclinic with 8 Si and 17 O atoms in the asymmetric unit, and consists of unusual one-dimensional straight channels containing pore openings that oscillate between rings of ten and eighteen tetrahedral atoms in layer-like units^[7]. An interesting feature of this structure is that the powder diffraction experiments revealed that SSZ-44 and SSZ-35 are very similar, with ABAB and ABCABC stacking, respectively, of layers along the pore direction. It was previously known that SSZ-44 and SSZ-35 had similar catalytic properties, and this new structural information shows that they are the pure end-member polymorphs of a new fault series, which allow investigation of the relationship between the organic structure-directing agent and the stacking fault structure of the zeolite framework.

Figure 7: Structure of CIT-5 viewed down the [010] axis, showing the large pores circumscribed by 14-membered rings of tetrahedral Si atoms.

MARGINAL MATERIALS

Du Pont Corporate Research and Development is working on new families of catalysts for Freon® replacements, and this has prompted work in various aluminum fluoride preparations. Compounds which are industrially useful catalysts often give very poor diffraction patterns which make them difficult to attack. One well-publicized example was the DuPont powder challenge. Data sets collected at two NSLS beamlines of a material alleged to be HalF_4 proved to be intractable, and so Richard Harlow issued a \$1000 cash challenge to the crystallographic community for the first acceptable solution from the data plotted in Figure 8.^[8] Relative to other work described here, this data is of extremely poor quality, having broad, overlapped peaks, with a limited range of d -spacings. Several entries (including the proposer) correctly found part of the structure, layers of

Figure 8: Diffraction data for alleged HAIF_4 . Upper trace, in a capillary to minimize preferred orientation, at beamline X3B1; lower trace, on a flat plate to maximize signal to noise ratio, at beamline X7A.

edge- and corner-sharing AlF_6 octahedra, but the credit for a complete solution goes to Leonid Solovyov and Sergei Kirik from the Institute of Chemistry and Chemical Engineering, Russian Academy of Sciences in Krasnoyarsk, Russia. By chemical tests on samples provided by DuPont, they suspected the presence of organic material, and so converged to the correct solution, with pyridinium counterions. This brings us back to a

theme that runs through much of the work reviewed here: chemical information beyond the diffraction data is often crucial to solve a structural problem, but that information had better be correct! It also shows that if one is sufficiently motivated, one can solve significant problems from rather poor samples, and that we can expect to see considerable further progress in the utility of *ab initio* powder techniques to interesting problems. ■

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Rapid At-Wavelength Inspection of EUV Mask Blanks by Photoresist Transfer

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Extreme-ultraviolet (EUV) lithography is an advanced form of optical projection lithography being developed by a consortium of integrated circuit manufacturers to print features $\sim 0.1 \mu\text{m}$ ^[1]. EUV lithography will likely employ a reflective mask consisting of a patterned absorber on a multilayer coated substrate (mask blank) that reflects a narrow band of EUV wavelengths near 13.4 nm. Such masks have the advantage of being thick and dimensionally stable, however, the use of such masks presents some challenges. Because it is currently impossible to repair certain types of defects in a mask blank, it will likely be necessary to begin with a defect free mask blanks. An effort is being made to produce defect free blanks^[2]. Blanks with as few as three defects, as determined by optical means, have been produced.

Unlike other existing and likely future technologies, optical inspection may not be sufficient to find all defects in an EUV mask. While optical inspection tools can be used to find certain defects (such as particles and scratches) on such a mask, subtle anomalies in the reflective

multilayer coating can produce dramatic changes in the real and imaginary components of the reflectivity at the EUV wavelength but have no apparent effect on visible light. Indeed, such defects have already been observed in EUV imaging experiments^[3]. Because of this, at-wavelength techniques, i. e. techniques using light of the wavelength at which the mask blanks are to be used, must be used to locate and mark defect sites. However, because optical inspection tools are so fast and efficient, it would be very economical to read out these marked defects optically. Therefore, we have developed a new EUV mask inspection technique in which defects are marked by a brief flood exposure to EUV radiation (from a synchrotron or laser-plasma point source) and then examined optically^[4].

In the simplest experimental arrangement for the new inspection technique, illustrated in **Figure A-1**, an EUV sensitive resist is deposited directly on the multilayer-coated mask blank. During a flood exposure, incoming EUV radiation passes through the thin layer of resist, reflects off the multilayer coating and then passes

Figure A-1. Diagram illustrating the photoresist transfer process. a) A photoresist is applied directly to an EUV reflective multilayer coated substrate that is subsequently flood exposed with EUV radiation. A reflectivity defect results in less exposure to the resist above the defect. b) After partial development of a positive tone resist a bump remains above the defect.

back through the resist once again. A local decrease in reflectivity will reduce the resist exposure above defect sites. Under these conditions, partial development of the resist will show reflectance defects as mounds (pits) in a positive (negative) tone resist. Once the EUV flood exposure has created features in the resist immediately above defect sites, these mounds or pits can be detected by an optical scanning instrument, allowing rapid flagging of EUV defects.

The experiment was performed on an EUV mask blank, a 2" diameter Mo/Si multilayer-coated silicon wafer with a peak reflectance of 62% at a wavelength of 13.4 nm. To demonstrate the detection of known defects, reflectivity defects (of approximately 12% less reflectivity than normal) were created in the mask blank using a focused ion beam of Ga⁺. Before exposure, the multilayer-coated EUV mask blank was spin-coated with an 800 Angstrom thickness of ZEP resist and baked at 190°C for 1 hour. The exposure was performed with 13.4 nm radiation from the 3rd harmonic of the U13 undulator. Following EUV exposure, the sample was spin-developed in xylenes and rinsed in isopropyl alcohol.

AFM images of an area of an EUV mask blank containing a programmed defect both before and after EUV exposure and development are shown in **Figures A-2a and A-2b**. In **Figure A-2a**, an AFM image of an area containing a bare programmed defect, there is a rectangular shaped depression in the surface of the multilayer coating in the implanted region approximately 10 Å deep. In **Figure A-2b**, an AFM image of the same area after EUV exposure and development, there is no longer a small depression; instead, there is a mound in the photoresist at the site of the defect approximately 40-50 Å high.

Current research efforts include the use of a higher contrast resist to create taller resist features. The taller features should be easier to detect, hence improving the sensitivity of our technique. Also currently being researched is an alternative technique which greatly reduces the risk of contaminating the mask blank. In this method, the resist is applied to a EUV-transmitting membrane which is placed in near contact with the mask blank during flood exposure. ■

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Atom-Specific Chemistry in Halogen Reactions with III-V Semiconductors

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Gaseous processes, such as plasma or reactive ion beam etching, are routinely used to fabricate structures for microelectronic devices. As device dimensions shrink and three-dimensional topography becomes more complex, however, these methods need to be better characterized and controlled. This requires that the gas-surface interactions be understood at the most fundamental level. Silicon etching is, by far, the system that has been the most widely studied, yet significant questions still remain. The situation for compound semiconductor materials is much worse^[1]. Despite their importance for photonic and electro-optic devices, factors affecting their processability by gaseous methods are poorly understood.

We have been using a number of surface-sensitive techniques, most notably synchrotron-based soft x-ray photoelectron spectroscopy (SXPS), to investigate the interaction of halogens with III-V semiconductor surfaces. Our group and others have found that not only are these reactions technically relevant, but there is also a rich chemistry involved. For example, ordered halogen overlayers form under certain conditions, while etching occurs under other conditions^[2]. Also, after large exposures at room temperature, surfaces are often covered with a thick halogenated reaction layer^[3].

Another useful by-product of our work has been the identification of methods whereby particular surface structures can be prepared in a simple manner via dry

chemical methods, i.e., without the need for a complex and expensive procedure such as molecular beam epitaxy (MBE). For example, we showed how to prepare high-quality GaAs(110) surfaces by reaction with Cl₂ at elevated temperature^[4], and how to prepare a group V-terminated (001) surface by saturating a group III-terminated surface with I₂ and then annealing^[2,5,6]. These methods of surface preparation can now be developed for use in other applications.

Most of our work has been with the (001) surface. This surface can be prepared to be either group III- or group V-terminated, so that the relationship between the surface termination and the reaction pathway can be directly determined. Note that this is also the surface employed for the fabrication of devices. We have used low energy electron diffraction (LEED) to measure the ordering and SXPS to determine the bonding configuration on the surface.

One of the remarkable observations is that, at room temperature, halogen adsorption on certain III-V semiconductor surfaces forms ordered overlayers, while other surfaces become disordered. The disorder is presumably the precursor to etching. Sometimes these complementary reaction pathways are obtained from initial surface conditions that are not too different from each other. **Figure A-3** summarizes whether particular (001) surfaces become ordered or disordered following a moderate exposure to I₂ or Cl₂, as determined by LEED.

<u>Material</u>	<u>I₂</u>		<u>Cl₂</u>	
	<u>Group III-Terminated</u>	<u>Group V-Terminated</u>	<u>Group III-Terminated</u>	<u>Group V-Terminated</u>
InAs	Order	Disorder	Disorder	Disorder
InSb	Disorder	Disorder	Disorder	Disorder
GaAs	Order	Order	Disorder/ Order	Disorder

Figure A-3. The experimentally determined reaction pathways of various III-V (001) surfaces when exposed to I₂ and Cl₂.

As can be seen, chlorine will almost always induce disorder (given sufficient exposure), while iodine sometimes orders and sometimes disorders.

There are a few factors that appear to control most of the observed behavior. For one, the various surfaces have different relative amounts of group III vs. group V atoms directly exposed to the incoming reactants. Note that, although a surface may be terminated with one element, there are most often vacancies in the first layer which expose some of the second layer atoms. Next, there is always a preference for reaction with group III, rather than group V, elements. On the reconstructed surface, group III elements have empty orbitals that protrude into the vacuum, while group V elements have filled lone pairs. The closed-shell halogen molecular orbitals are attracted to the empty surface orbitals, while they are repelled by the filled surface orbitals. Thus, the bonding of halogens is more likely to occur at group III surface sites than at group V sites. The degree of preference varies from system to system, however. Finally, an inability to form higher halides can passivate surfaces with respect to further reaction following the formation of a single monolayer of adsorbates.

For I_2 reacted with InAs(001), there is a big difference in the reaction pathway for the two terminations^[2]. On the In-terminated surface, a well-ordered (1×1) structure forms in which all of the iodine is attached to In, mostly in the form of the monoiodide, i.e., InI. The As-terminated surface becomes disordered, however, and iodine attaches to both In and As. SXPS spectra collected from I_2 -reacted InAs(001) are shown in Figure A-4.

These observations can be explained by assuming that iodine initially bonds to the group III In atoms, whether they are in the first or second layer^[2]. On the In-terminated surface, iodine attaches to the

Figure A-4. High-resolution SXPS In 4d and As 3d spectra collected from In- and As-terminated InAs(001) after exposure to I_2 .

Figure A-5. Schematic illustration of the reaction mechanism for I_2 with In- and As-terminated InAs(001).

outermost In atoms forming an ordered overlayer of monoiodides. This surface passivates, as the formation of diiodides is limited. On the As-terminated surface, on the other hand, iodine initially bonds to a second layer In atom. Upon bonding, charge is transferred away from the filled orbitals associated with surface As. This makes the first layer As atoms reactive to additional incoming I_2 . When iodine has bonded to both In and As, the In-As bonds break and the surface eventually becomes disordered. This mechanism is illustrated in **Figure A-5**.

On some surfaces, however, the preference for bonding to the group III element overwhelms all other factors. An example of this is given by the reaction of I_2 with InSb(001). As seen in **Figure A-6**, InI_2 is the major surface product, and the two terminations produce nearly identical spectra following reaction.

Cl_2 reactions appear to not be limited by either the preference for bonding to a group III atom, or by the stability of monohalides. Instead, it appears that chlorine can easily bond to either the group III or group V atom, and can easily form higher halides^[7]. This is seen in **Figure A-7**, which shows spectra collected from three InAs(001) surfaces following Cl_2 reaction. InCl is the primary species formed on all three surfaces, although some InCl_2 , InCl_3 , AsCl and AsCl_2 also form (AsCl_3 is volatile). On the initially As-terminated surface, relatively more of the higher In chlorides and As chlorides are produced. On the sputtered surface, however, relatively more In chlorides form, possibly because As was preferentially removed by the sputtering. Thus, although the relative amounts of the chlorides produced depend on the initial surface stoichiometry, the basic reaction pathway is essentially

independent of termination. The same type of behavior is seen for Cl₂ reaction with GaAs(001) ^[8].

In summary, we have used synchrotron radiation and other surface-sensitive probes to enable a fundamental understanding of halogen reactions with III-V semiconductor surfaces. We found that the surface stoichiometry, and the relative ease of formation of the

higher halides determine the reaction pathway. These experiments (1) provide good basic physical and chemical insight into surface reactions, (2) will enable the development of a more rational approach to dry processing procedures, and (3) provide the understanding necessary for chemically producing surface structures with atomic precision. This last point will become even more crucial as the size scales on which we work get smaller. ■

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Solving the Structure of a Histone Acetyltransferase: Pushing the Limits of MAD

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Chromatin is a complex between the genetic material DNA and some highly charged proteins called histones. It is built up from repeating units called nucleosomes, which under the electron microscope appear like beads on a string of DNA. Within each nucleosome the DNA is wrapped nearly twice around a core structure composed of two copies each of the core histones H2A, H2B, H3 and H4. Beyond this fundamental level, nucleosomes form complex interactions with each other and with other chromatin components to form chromosomes.

Organizing DNA this way has two major advantages. Firstly it means that the large amount of information contained within the genomes of higher organisms can be packaged into a nucleus. For example, the DNA in the chromosomes of a human genome, if laid out end to end, would measure roughly two metres long but has to be contained within a nucleus that is only about 10-20 μm in diameter. Secondly it is a powerful way of regulating how the information within a genome is expressed.

The first step in gene expression involves the formation of mRNA by a complex machinery of proteins that interact with DNA during the process of transcription. Eventually the mRNA serves as a template for the production of specific proteins. By changing the structure of chromatin so that it is more or less accessible to components of the transcription machinery, cells can regulate which genes are expressed and which are not. Changes in the pattern of genes that are expressed drive the differentiation of different cell types during the development of an organism. After development is complete, maintaining these patterns helps to define the behaviour of the various cell types found within a complex organism. Failures in this complex control can result in developmental disorders, cancer, or other diseases.

One way that cells regulate the structure of chromatin is by modifying specific lysine residues of the

histones by attaching an acetyl group. Each core histone is made up of a globular region that is involved in interaction with the other histones and with DNA, and more flexible, highly charged tails at each end of the globular region. The N-terminal tails in particular contain many positively charged arginine and lysine residues. Many of the lysine residues have been found to be acetylated under different conditions, and it has been known for a long time that the level of acetylation of histones in different regions of chromatin correlates well with the activity of the chromatin in terms of gene expression. For example, genes that are active often are associated with chromatin in which the histones are highly acetylated, while inactive genes are associated with chromatin made up from less acetylated histones. Despite this correlation it still is not known for certain how histone acetylation alters the structure of chromatin. One possibility is that acetylation reduces the positive charge of the histone tails, making them less able to bind DNA where they could interfere with binding of various transcription factors. A second is that it alters the way in which the nucleosomes interact with each other so that chromatin becomes generally more open to the large protein complexes involved in transcription. Finally, it may act as a signal or flag, similar to phosphorylation of proteins during signal transduction, so that it alters the interaction of other proteins with nucleosomes.

Histone acetylation is controlled by specific histone acetyltransferase (HAT) enzymes and histone deacetylase enzymes. Many of the genes that encode these enzymes have been identified within the past few years, which has enabled a more detailed examination of the way in which they function. One difference between HAT enzymes that has emerged from biochemical studies relates to their specificity toward histones. Some have very broad specificity, being able to acetylate lysine residues in any of the four core histones, while others discriminate more highly, showing a marked preference for modifying one

or two lysine residues within one or sometimes two of the histones. Some HATs also can acetylate non-histone substrates such as proteins involved in the transcription machinery itself.

There are examples of chromosome translocations that fuse a HAT gene with another gene. These translocations often are associated with leukemia, indicating that uncontrolled acetylation could be a major factor in promoting cancer formation. Therefore it is important to understand how these enzymes work, in particular how they achieve specificity for a particular target protein. From comparisons of protein sequences it also has been shown that many of the known HATs belong to a large superfamily of enzymes that acetylate a variety of different types of substrate, including antibiotic compounds and factors involved in regulating circadian rhythm. There is therefore more widespread interest in this class of enzymes because of their roles in gene regulation, bacterial antibiotic resistance, and control of day/night cycles.

In collaboration with Rolf Sternglanz from SUNY Stonybrook we set out to determine the structure of the enzyme Hat1, a HAT gene from baker's yeast. Hat1 was the first HAT gene to be identified, both by Sternglanz and separately by Dan Gottschling's group at the

University of Chicago. We initially tried to crystallize the full length 374 residue protein, and eventually ended up with some large crystals, but they took several months to grow and diffracted X-rays only to medium resolution. To fix the problem we used an approach that is quite often adopted by structural biologists. We used proteases to probe the Hat1 protein for flexible regions that might be interfering with the formation of crystals. However we wanted to be careful not to destroy the catalytic activity of the protein, so we combined these proteolytic assays with HAT activity assays to be sure that we retained an active fragment. From this analysis we were able to determine that 54 residues at the C-terminal end of the protein were probably more flexible, and that we could remove them without reducing the activity of the protein. When we made this new protein (residues 1-320) we quickly obtained crystals that diffracted to high resolution ($\sim 2\text{\AA}$ at Beamline X12C of the NSLS) giving us the opportunity to solve the structure.

However there was still one potential obstacle in our way, which was how to solve the phase problem. Two methods are available for determining phases for a new structure. The first is multiple isomorphous replacement (MIR) which involves soaking crystals in compounds of various heavy metals and comparing the diffraction

Structure of the Hat1-Acetyl CoenzymeA complex

Figure B-1. (left) A cartoon style representation of the Hat1-Acetyl CoA complex structure. The acetyl coenzymeA (acetyl CoA) is shown in a stick representation and marks the active site of the enzyme. The two topological folding domains of the protein are marked.

(right) A model for how Hat1 might bind a substrate histone molecule. The model was developed on the basis of our structure in conjunction with biochemical data and helps to explain why Hat1 specifically

Model of a histone peptide bound to Hat1

modifies a particular lysine side chain in histone H4. The panel shows a close up of a surface representation of Hat1 in the vicinity of the active site, highlighting the features of a possible peptide binding channel that complement the structural and chemical features of the histone peptide (shown in a stick representation). This stereochemical complementarity helps to explain why Hat1 prefers to modify lysine residue 12 of histone H4 and not other lysine side chains.

patterns from these "derivative" crystals to those of "native" untreated crystals to yield phase information. One drawback of this approach is that attaching the heavy metal to the protein must not severely disrupt the crystal lattice, otherwise it is impossible to compare the "derivative" and "native" diffraction patterns. This often means that a large number of heavy metal compounds have to be screened to find a reasonable number of suitable derivatives. An alternative approach is to incorporate an atom into the protein that scatters X-rays anomalously. X-Ray diffraction patterns then are collected using X-rays at different wavelengths, typically two wavelengths around the X-ray absorption edge of the atom and another at a remote wavelength. The information from these data sets collected at different wavelengths can be treated in a fashion analogous to the "native" and "derivative" data sets of MIR to calculate phases. This multiple wavelength anomalous diffraction (MAD) method is becoming an increasingly popular way to solve protein structures. One reason for this is that it requires only a single crystal of the protein and so the problem of non-isomorphism that often makes MIR difficult does not exist. However, it does require that data can be collected at a tuneable X-ray source such as only exists at synchrotron sources like the NSLS.

Our lab has solved several structures using MAD but at first this did not seem to be the best approach to take for Hat1. One factor influencing the success of MAD is being able to incorporate sufficient anomalously scattering atoms into the protein. An ideal method to do this is to take advantage of protein chemistry and prepare protein in which the sulphur atom in each methionine residue is replaced by selenium. This can be done fairly easily by making protein in bacteria and growing the bacteria on a medium that contains selenomethionine instead of methionine. It usually takes a ratio of 1 methionine for every 100 or so protein residues to give enough anomalous signal to successfully determine a MAD structure. However, Hat1 has only 2 methionines for 320 residues, putting us well below the typical 1 in 100 mark. Therefore we made several attempts to prepare derivative crystals to try solving the structure by MIR, but in most cases the crystals either did not diffract as strongly or were clearly non-isomorphous with native crystals. We therefore reconsidered using the MAD approach (and given the type of protein we were working on there were lots of jokes about "MAD HATters"). We went ahead and prepared Selenomethionine-Hat1 crystals but to try to increase the chance of success we attempted a variation on the MAD approach. We prepared crystals of a methyl-mercury derivative of Selenomethionine-Hat1, which although not isomorphous with underivatized protein, did contain more anomalously

scattering atoms. We then planned to collect a four wavelength MAD data set, collecting data sets at the absorption edges of the Selenium and mercury atoms (2 for Se, 1 for Hg) plus a remote wavelength.

We took these crystals to the NSLS in Feb. 1998 and collected a four wavelength MAD data set using a crystal of methylmercury-selenomethionine-Hat1 plus a "typical" three wavelength MAD data set using a crystal of plain selenomethionine-Hat1. We collected the data on beamline X12C using a CCD detector which gave excellent sensitivity in measurements of X-ray data. Being able to measure accurately small differences in the X-ray data is important for the MAD technique. As things turned out we were able to solve the structure using the "typical" three wavelength MAD data set (based on Se atoms alone). In part we were fortunate that both methionine residues in Hat1 are buried in the core of the protein and are well ordered in the structure. The second factor in our favour was that we were able to collect high quality data sets using the CCD detector and measure the very small differences produced the two selenium atoms. This is, to our knowledge, the lowest selenium content (1 per 160 residues) ever used to solve a structure, but we were still able to produce high quality electron density maps, making interpretation much easier. We later used the four wavelength data set to calculate a map but there were no clearly discernable improvements over the map using phases from selenium alone. Another innovation that was available to us was the program SOLVE, which automatically locates heavy atom positions and calculates phases. This enabled us to calculate an electron density map within only three to four hours of completing the data collection. This almost instant feedback gave us a better assessment of the level of success of our experiment allowing us to make better use of our data collection time and return to Utah, sleepy but happy MAD hatters.

The structure of the Hat1-acetyl CoenzymeA complex has given us a wealth of information about how this enzyme, and other related histone acetyltransferases such as Gcn5, function. Using the structure we have been able to propose a model for how Hat1 binds to a particular histone and places a specific lysine residue within the active site for modification. The structure will play a key role in future structural and biochemical studies aimed at understanding this important group of enzymes. ■

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Chemical Differences in Subchondral Osteoarthritic Bone Observed with Synchrotron Infrared Microspectroscopy

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Infrared spectroscopy at the NSLS has grown substantially over the last several years as increased use of the existing beamlines has led to construction of new facilities. The use of microscopy to study biological structure is one of the main goals of the new station at U2B, which is operated by the Albert Einstein College of Medicine as an NIH Resource Center. The study of bone structure and function is one of the main projects of the Center, and this article describes some of the progress that has been made so far in understanding bone disease

using synchrotron methods of the NSLS.

Osteoarthritis is the leading cause of disability among people over 65 years old and affects approximately 40 million people in the United States^[1]. It is generally considered a disease of the cartilage. However, it has recently been suggested that the health and integrity of the articular cartilage is dependent upon the mechanical properties of the underlying subchondral bone (Figure B-2), where stiffened subchondral bone may lead to the breakdown of articular cartilage^[2].

Figure B-3. (Clockwise from top left) Frequency distributions for phosphate/protein, carbonate/protein, crystallinity, and carbonate/phosphate ratios from 5 histologically normal monkey samples (10 infrared maps) as compared to and 2 monkeys with severe osteoarthritis (7 infrared maps). Mineral/protein ratios and carbonate content are significantly higher in osteoarthritic bone than normal bone. Average crystal size/perfection is broadly distributed and not significantly different between normal and osteoarthritic monkeys. Data are expressed as ratios to account for small variations in sample thicknesses.

Osteoarthritic joints display significant thickening of the subchondral bone. To date, the basis for subchondral bone thickening is unknown. But, an extensive study of cynomolgus monkeys has demonstrated that this thickening precedes the development of articular lesions^[3]. In addition, there appears to be a threshold subchondral bone thickness, below which articular cartilage lesions are extremely rare. Because of the inherent difficulty in studying the *early* stages of the disease in humans, the use of an appropriate animal model is important. We hope that using the cynomolgus monkey model to examine all ages and stages of the disease will help shed light on the comparable human condition.

The mechanical properties of subchondral bone are dependent upon its chemical structure. Modifications in the chemical composition of bone can affect properties such as its strength, rigidity, density, and flexibility. Thus, we hypothesize that the bone matrix composition and/or bone mineral content and crystallinity are modified *in situ* in a specific manner as a function of subchondral bone thickness.

We are addressing this hypothesis by examining the chemical composition of subchondral bone using synchrotron infrared microspectroscopy. Infrared spectroscopy is an analytical technique that is sensitive to the chemical components in bone; a spectrum of infrared light is absorbed differently depending on the protein (primarily collagen) and mineral (hydroxyapatite) content and composition. More specifically, this technique can be used to determine (1) protein structure and concentration, and (2) mineral concentration, crystallinity, and content (e.g. phosphate, acid phosphate, carbonate). The infrared crystallinity results are well-correlated to hydroxyapatite crystal size and perfection as determined by x-ray powder diffraction. If infrared light is put through a microscope (equipped with special infrared optics), then infrared spectra can be collected on micron-sized regions of bone *in situ* and compared to visible images of the same region. Thus, we can examine the chemical composition of subchondral bone as a function of (a) age, (b) position in the subchondral plate, and (c) thickness of the subchondral plate and compare

these results as a function of severity of the disease. Inherently, the long wavelengths of infrared light limit the spatial resolution achievable with this technique. Our preliminary results show that substantial changes in chemical composition occur within 20 μm of new bone growth^[4,5]. This type of spatial resolution can only be achieved with a synchrotron infrared source. The National Synchrotron Light Source at Brookhaven National Laboratory provides a source of broadband infrared light that is 1000 times brighter than a conventional infrared source. This permitting rapid data collection at the highest spatial resolution possible (3-5 μm).^[6]

Using synchrotron infrared microspectroscopy, we have compared the chemical composition of subchondral bone *in situ* in histologically normal versus osteoarthritic monkey tibias. Specifically, we have analyzed the protein, phosphate, and carbonate concentrations and bone crystallinity throughout the subchondral bone as a function of disease severity. **Figure B-2** illustrates mineral/protein infrared maps from the subchondral bone of normal (top) and osteoarthritic (bottom) monkeys. As can be seen, both the phosphate/protein and carbonate/protein ratios are higher in the osteoarthritic bone. The results from this single sample were confirmed by examining five radiographically normal monkeys and two monkeys with severe osteoarthritis (as defined by considerable fibrillation and cleaving of the joint cartilage.)

This analysis totals approximately 3000 infrared spectra for each disease state. Frequency distributions were calculated for normal bone versus osteoarthritic bone and can be seen in **Figure B-3**. Variance analysis (ANOVA) demonstrates that these both the phosphate/protein and carbonate/protein distributions are significantly different for normal versus osteoarthritic bone, where the osteoarthritic bone is more mineralized than the normal bone.

Analysis of the carbonate/phosphate ratio (**Figure B-3**) also demonstrates that the carbonate content is significantly higher in osteoarthritic bone than normal bone. High carbonate content often correlates with low average crystal size, since carbonate substitution into the hydroxyapatite lattice is usually limited to the surface of hydroxyapatite crystals. However, we find that the average crystal size/perfection are broadly distributed in both normal and osteoarthritic bone, but are not significantly different between the normal and disease states.

These results support our hypothesis that chemical composition changes occur in the subchondral bone in osteoarthritis. Since subchondral bone composition affects both the mechanical and physiological properties of bone, differences between histologically normal versus osteoarthritic monkeys may provide insight into the chemical basis for subchondral bone thickening and articular cartilage breakdown in osteoarthritis. ■

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Crystal Structure of a Hepatitis Delta Virus Ribozyme Reveals a Buried Active Site Cleft Reminiscent Protein Enzymes

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The hepatitis delta virus (HDV) ribozyme is the only catalytic RNA known to be required for the viability of a human pathogen. This self-cleaving RNA is remarkable for its high stability, its limited requirement for divalent cations, and its fast rate. The HDV ribozyme is fully active in conditions, such as 8 molar urea, under which most RNAs and proteins are denatured and strongly inactivated. Because of the negatively charged phosphodiester backbone of RNA, most catalytic RNAs depend on divalent cations to fold into their active conformations. The HDV ribozyme is the fastest natural self-cleaving RNA that has been characterized. Even in very low concentrations of a variety of divalent cations, it

can cleave itself at $\sim 1/\text{second}$, which is two orders of magnitude faster than a typical self-cleaving RNA, and approaches the rate of some protein enzymes^[1].

In order to gain structural insight into the unique catalytic prowess of this RNA, we had to obtain well-diffracting crystals. Good crystals of tightly folded RNAs are notoriously difficult to obtain, presumably because the surface of these molecules is dominated by a periodic array of phosphates. This might allow neighboring molecules to pack subtly out of register in the crystal, resulting in poor order. To overcome this problem, we engineered a solvent-exposed, functionally and structurally dispensable stem-loop of the HDV ribozyme into a high-affinity binding site for the small, basic RNA-binding domain (RBD) of the U1A protein. We expected the complex between the 72 nucleotide RNA and the U1A protein RBD to produce better crystals than the "naked" RNA, because the complex not only has a negatively and positively charged ends, but has the wide variety of surface functional groups of the protein available for making crystal contacts.

Our best crystals of an HDV ribozyme-U1A RBD complex diffracted X-rays to beyond 2.2 Å resolution. We solved the structure by crystallizing RNA-protein complexes in which the methionines of the protein moiety had been biosynthetically substituted with selenomethionine. We then carried out a multiwavelength anomalous diffraction (MAD) experiment around the selenium absorption edge at NSLS Beamline X4A, with help from Drs. C. Ogata and D. Cook. The phases thus obtained produced a good electron density map, and the resulting atomic model has now been refined against diffraction data extending to 2.3 Å resolution^[2]. Use of the RNA-binding protein "crystallization module" might be a powerful general technique, not only for obtaining high quality crystals of large RNAs, but also for determining their structures by MAD.

Our crystal structure revealed that the HDV ribozyme adopts a compact, highly convoluted fold in which five helical segments are linked by five strand crossovers; a connectivity that can be described as a "nested double pseudoknot" (Figure B-4). This fold buries the

Figure B-4. Ribbon and stick representation of the HDV ribozyme-U1A RBD complex structure. The protein moiety lies at the bottom of the figure, far away from the active site residues that surround the 5' terminus of the RNA. The five helical or base-paired segments are labeled P1 through P4 and P1.1.

Figure B-5. Solvent accessible surface representation approximately in the same orientation as Figure B-4. The atoms of the 5'-most ribose are shown as red spheres. The active site lies in the cleft behind this sugar.

active site at the bottom of a deep, solvent-inaccessible cleft, in which various base and backbone functional groups appear to be poised to achieve catalysis (Figure B-5). Interpretation of previously published biochemical results from other workers in light of the structure suggests that the complex fold of the HDV ribozyme is at least in part responsible for the remarkable stability of this RNA. No tightly bound metal ions were found in the electron density maps. This is consistent with published biochemical data, and indicates that nature can achieve intimate packing of negatively charged RNA without recourse to tight metal ion chelation^[3].

The HDV ribozyme is the first catalytic RNA whose ground state structure demonstrates an active site pre-organized for catalysis. The three-dimensional structure of this remarkable ribozyme constitutes the starting point for further biochemical, biophysical and pharmacological investigations into its mode of action both *in vitro* and in the life cycle of the virus in the human liver. ■

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How Sos Activates Ras: The Crystal Structure of the Ras-Sos Complex at 2.8 Å Resolution

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The Ras proteins are structurally conserved guanine nucleotide binding proteins that are involved in intracellular pathways controlling cell proliferation and differentiation^[1,2]. When bound to GTP, Ras is “on” and is able to transmit the cellular signal initiated by ligand binding to receptor tyrosine kinases. When bound to GDP, Ras is “off” and is unable to propagate the cellular signal. The intrinsic rates of GTP hydrolysis and nucleotide dissociation from Ras are low, therefore, two classes of proteins regulate the nucleotide-bound state of Ras: GTPase activating proteins catalyze the hydrolysis of GTP to GDP, turning Ras “off”, while guanine nucleotide exchange factors facilitate the exchange of GDP for GTP, switching Ras “on”. Although many

biochemical studies have been done on Ras exchange factors, the mechanism for exchange was largely unknown due to the lack of structural information.

The X-ray crystal structure of Ras complexed to the exchange factor Son of Sevenless (Sos) was recently determined (**Figure B-6**) using multiple isomorphous replacement methods. Data used to solve the structure were collected at beamline X25 at the National Synchrotron Light Source at Brookhaven National Laboratories using the Brandeis B4 2x2 module CCD detector. The structure provides the first glimpse of how Ras exchange factors function to (1) remove nucleotide from Ras and (2) allow nucleotide to rebind to Ras.

The structure reveals that Sos interacts extensively

Figure B-6. The complex of H-Ras with the exchange factor region of human Sos1. The N-terminal domain of Sos (residues 568 to 741) is blue; the C-terminal catalytic domain (residues 752 to 1044) is green; the Switch 1 and Switch 2 segments and the P-loop region of Ras (as defined in the text) are orange and red, respectively. Conserved regions (SCRs) among Ras family exchange factors shaded cyan^[2,3]. Disordered residues of Sos are shown as dotted lines.

with Ras to exclude nucleotide and magnesium ion and stabilizes Ras in the nucleotide-free state. Residues 25-40 of Ras (the Switch 1 region), which normally form part of the nucleotide binding pocket, are completely removed from the nucleotide binding site. Conformational changes in residues 57-75 of Ras (the Switch 2 region) result the occlusion of the magnesium binding site of Ras by Ala 59 (Figure B-7). Sos also introduces a hydrophobic residue (Leu 938) into the magnesium binding pocket, further changing the electrostatic environment to one unsuitable for a divalent ion. Residues which normally

coordinate the nucleotide (Lys 16 and Gly 60) are in alternate hydrogen bonding interactions and Sos introduces an acidic residue (Glu 942) into the α -phosphate binding site, further disrupting the binding site of the nucleotide. Importantly, the complex leaves the guanine base and ribose binding sites unobstructed, thus providing a mechanism for the rebinding of nucleotides to Ras to complete the exchange reaction.

This structure was recently published in *Nature*: P. A. Boriack-Sjodin, S.M. Margarit, D. Bar Sagi, and J. Kuriyan, "Structural basis for the activation of Ras by Sos", *Nature* 394, 337-343 (1998). ■

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Figure B-7. A schematic of the differences in the Switch 2 regions of a) Ras-GTP analogue (5P21)^[4] and b) Ras-Sos. Selected polar interactions are shown as dashed lines, hydrophobic interactions are shown as solid arcs.

Structure of the HIV gp120 Envelope Glycoprotein in Complex with CD4 and a Neutralizing Human Antibody

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As the 20th century draws to a close, the human immunodeficiency virus (HIV) continues to spread, having recently surpassed both malaria and tuberculosis as the leading infectious disease killer. Since 1983, when HIV was first discovered, the UNAIDS organization estimates that over 40 million people have been infected of which almost 15 million have died. Despite global awareness and recent therapeutic advances, the only hope for containment in many countries lies in the development of a vaccine. In spite of enormous effort, all such vaccine candidates have thus far met with failure.

HIV is an enveloped virus, which establishes a persistent infection in its human hosts. Although neutralizing antibodies are generated, HIV eludes the immune system. This ability of HIV to evade the immune system is the basis of both the well noted vaccine failures as well as of the ability of HIV to establish a persistent infection. Over a period of years, the constant battle with the virus impairs the immune system enough to allow opportunistic infections to kill the human host.

The HIV lipid envelope, which is derived from the membrane of the host cell, hides most of the infectious virus particle from immune surveillance. Only two viral proteins protrude beyond the protective lipid bilayer, the exterior envelope glycoprotein, gp120, and its membrane-spanning partner, gp41. Of these, gp120 appears to occlude much of the gp41 ectodomain and is the target of virtually all neutralizing antibodies.

The exposure of gp120 on the virion surface is a functional consequence of its central role in virus-cell entry. The gp120 glycoprotein binds sequentially to the CD4 glycoprotein and a chemokine receptor on the surface of specific cells of the immune system, and then in concert with gp41 orchestrates a fusion of the viral and cellular membranes. This fusion is critical for the introduction of HIV's genetic material into the target cell.

Because of the pivotal role of gp120 in receptor binding and interactions with neutralizing antibodies, we undertook an investigation of its atomic structure.

The size of gp120 as well as its extensive glycosylation and expression in eukaryotic cells limited

the choice of high resolution structural techniques to X-ray crystallography. X-ray crystallography in turn depends on obtaining well ordered crystals of the molecule of interest. For gp120, this proved to be extraordinarily difficult.

It was apparent from functional and sequence analyses that many of the same mechanisms that HIV uses to avoid specific protein:protein contacts in eluding the immune system, would also stymie attempts at forcing gp120 into the specific protein:protein lattice interactions required to produce a well ordered crystal.

To surmount these difficulties, we used a procedure, which we term variational crystallization, that focuses on protein modification to enhance the overall probability of producing crystals. Variational crystallization takes advantage of the ability of molecular biology to provide almost limitless variation in the protein of interest. To guide our efforts, we also derived the theoretical underpinnings for an approximate probability theory, based on the comparison of crystallization probabilities before and after a modifying procedure^[1].

This combination of probability analysis and variational crystallization produced very small crystals from four different gp120 crystallization variants, in six different crystallization conditions. Because the larger crystals necessary for high resolution structural analysis are often difficult and time-consuming to grow, we used the NSLS beamline X4A, to shortcut the process. The intense X4A x-rays, several orders of magnitude stronger than standard laboratory sources, enabled us to directly assay the suitability of the very small initial crystals for structural analysis. Of the six, only one proved sufficiently well-ordered for further study. This crystallization variant was composed of a ternary complex of core gp120 (gp120 with deletions at the N and C termini as well as at two internal variable loops), the N terminal two domains of CD4, and the antigen binding fragment of the neutralizing human antibody, 17b.

Small needle-shaped crystals, 5-10 μm in cross-section, could readily be grown. These were used to screen for appropriate cryocrystallography conditions and heavy atom derivatives. Despite several months of concentrated

effort, only five larger crystals, with cross-sections of $\sim 50 \mu\text{M}$, could be grown. Nonetheless, use of the NSLS X4A beamline made possible the collection of a native and three derivative data sets. The structure was solved to $2.5 \text{ \AA}$, a resolution sufficient to decipher the gp120 atomic structure (Figure B-8).

Analysis of this structure revealed the detailed features of a cavity-laden CD4-gp120 interface, highlighted by an extraordinary $150 \text{ \AA}^3$ cavity which reached from the pivotal Phe 43 residue of CD4 into the heart of gp120. Also revealed were a conserved binding site for the chemokine receptor, evidence for a conformational change upon CD4 binding, and the nature of a CD4-induced antibody epitope^[2]. In addition, epitope maps in conjunction with the crystal analysis defined the antigenic structure of gp120 and specific mechanisms of immune evasion^[3]. These included multiply redundant mechanisms of steric hindrance and conformational occlusion as well as a completely unanticipated, immunologically "silent" face masked by carbohydrate.

Whereas previous attempts to devise an HIV vaccine relied on indirect hints and clues, our results visualize gp120 at atomic resolution. Understanding the structural basis for the remarkable ability of HIV to elude

the immune response should assist in the design of a vaccine. Our results thus provide not only a framework

for understanding the biology of HIV entry into cells, but also a guide in attempts to intervene. ■

Figure B-8. Structure of the gp120 ternary complex shown in its biological context. The center of the figure depicts the gp120 ternary complex in ribbon representation, with core gp120 (red) caught in the act of binding to the CD4 receptor (yellow) by the neutralizing antibody, 17b (heavy chain, blue; light chain purple). The pivotal Phe 43 residue CD4 is also shown, at the nexus of the gp120-CD4 interface. The target cell membrane (cyan), domains 3 and 4 of CD4 (yellow ovals), chemokine receptor (green), viral spike and virion (pink) are also shown for reference.

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Structural Basis for the Interaction of Ras with RalGDS

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Ras is central within a web of signaling circuits, thus understanding the basis for Ras control is key to unraveling the principles of signal transduction. Indeed, *ras* is the most prevalent of all the oncogenes isolated from human tumors. Mutationally activated Ras plays a role in 90% of pancreatic cancers, 50% of colon cancers, and 25% of lung cancers.

Ras and a large family of related GTPases function as molecular switches that control a variety of signaling pathways that regulate cell growth, motility, intracellular transport, and differentiation. Interaction of the GTPase with its effectors is dependent on binding of GTP. Binding of GTP to human Ras results in major conformational changes in two highly conserved regions: switch I and switch II.

The Ras protein signals to a number of distinct pathways by interacting with diverse downstream effectors. Among the effectors of Ras are the Raf kinase and RalGDS, a guanine nucleotide dissociation stimulator specific for Ral (Ras like GTPase). Ral may regulate Cdc42, which plays a role in the actin cytoskeleton organization. Ral also signals to Phospholipase D pathway. Despite the absence of significant sequence similarities, both effectors bind directly to Ras, but with different specificities. The solution of the Ras/effector complex would lay a foundation for understanding Ras signaling, and thus the role of malfunctioning Ras in oncogenesis.

The X-ray data taken at the BNL beam line X12B and X25 enabled us to solve the structure of the complex between Ras(E31K) and the Ras-interacting domain (RID) of RalGDS in the presence of bound non-hydrolyzable GTP to a resolution of 2.1 Å⁽¹⁾. The small crystals of the complex diffracts only weakly at home source, which is not sufficient to determine the precise side chain interactions in the complex. Access to the high beam intensity of beam line X25 was mandatory in order

to determine its structure to such a high resolution.

This structure reveals that the β -sheet of the RID joins the switch I region of Ras to form an extended anti-parallel β -sheet with a topology similar to that found in the Ras homologue Rap-Raf complex. In addition, the overall structure of RalGDS-RID is similar to that of the Ras-binding domain (RBD) of Raf and ubiquitin. However, the side chain interactions at the joining junctions of the two interacting systems, the electrostatic complementarity at the interface, and the relative orientation of the two binding domains are distinctly different. Furthermore, in the case of the Ras-RID complex a second RID molecule also interacts with a different part of the Ras molecule, the switch II region. These findings account for the cross-talk between Ras and Ral pathways. Additionally, comparison with the Rap1A/Raf-RBD structure enabled us, for the first time, to explain the multiplicity and specificity of Ras signaling at an atomic level. It also helps to distill the common structural motif underlying protein-protein interaction in the signal transduction process.

The mechanism by which RalGDS is activated by Ras is implied by the observation that the interaction with Ras perturbs the structure of RalGDS-RID, but not Ras itself. The free RalGDS-RID was solved by the MAD method with data taken at beam line X4A using a selenomethionine mutant protein crystal⁽²⁾. This conformational change might activate the catalytic domain of RalGDS structurally, which in turn would activate Ral.

The structural characterization of the Ras/effector interface will assist in the rational design of small drug molecules capable of disrupting this interface, thereby halting the oncogenic effect of the constitutively activated Ras. Furthermore, the discovery of this interaction motif will aid in recognizing other pairs of signal transduction proteins that may interact via these mechanisms. ■

Figure B-9. Ribbon representation of the complex between RalGDS-RID and Ras(E31K).

(a) Front view of the complex;

(b) a different view of the complex.

Ras molecules are in purple (Ras1) and brown (Ras2), and RID molecules are in cyan (RID1) and green (RID2). The switch I region (residues 30-37) is colored yellow, while the switch II region (residues 60-76) is colored blue. Non-hydrolyzable GTP (GMPPNP) and magnesium are drawn in ball-and-stick representation.

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Structure of the Large Ribosomal Subunit from *H. marismortui* at 5.5 Å Resolution

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The ribosome plays a pivotal role in the process of protein synthesis. Ribosomes mediate the interaction between mRNA and tRNA, and catalyze peptide-bond formation. Protein synthesis is one of the most studied cellular processes. Intense research over the past 30 years led to many important discoveries regarding sequences of ribosomal components, folding of the rRNAs, spatial proximity of ribosomal proteins, and overall shape of the ribosome^[1, 2, 3, 4]. Structural information about the nature of protein-nucleic acid interactions in the ribosomes and the structure of the entire organelle is a prerequisite for a mechanistic analysis. Undoubtedly this structure also will reveal fundamental architectural principles that govern the assembly and maintain the stability of all ribonucleoproteins. The progress on the research of ribosomes is reviewed periodically in book form^[5, 6, 7, 8]. The general interest has shifted from investigations of the biochemical properties of the ribosomes to high-resolution studies of their structure and function.

Prokaryotic ribosomes (70S) are composed of two subunits and have a molecular weight of roughly 2.5×10^6 . Each of the subunits is a complex of ribosomal RNA (rRNA) and proteins. The small subunit (30S) contains a single 16S rRNA molecule and 21 different proteins, while the large subunit (50S) contains two rRNA molecules and 32 different proteins^[9]. The 50S subunit has a molecular weight of about 1.5×10^6 Daltons and catalyzes peptide bond formation, possibly functioning as a ribozyme^[10]. Eukaryotic ribosomes are similar structurally, but they are significantly bigger and more complicated^[11]. Although the research on which we are embarked focuses on the large subunit from an archaebacterial ribosome, there is no reason to believe that bacterial and eukaryotic ribosomes are fundamentally different^[12], and many of the conclusions are likely to be transferable.

We have generated X-ray crystallographic electron density maps of the large ribosomal subunit from *Haloarcula marismortui* at various resolutions up to 5.5 Å, having measured data from crystals that diffract to 3 Å resolution. Initial steps in the solving of this structure demanded a marriage of two techniques, electron microscopy and X-ray diffraction. We were able to place a 20 Å resolution, three-dimensional model of the 50S

subunit determined by EM methods into the crystal unit cell. To accomplish this required that we had measured the lowest-angle X-ray diffraction data from the crystals, in the 80-20 Å range, which often are ignored by crystallographers. This provided initial phases for low resolution reflections. Using these EM-derived phases, we were able to use difference-Fourier electron-density maps to locate the high-occupancy sites in heavy atom-derivatized crystals. This allowed us to initiate the process of heavy-atom refinement, leading to our first interpretable electron density map of the 50S subunit. The map at 9 Å resolution revealed long, continuous, but branched features whose shape, diameter and right-handed twist are consistent with segments of double helical RNA that crisscross the subunit^[13].

High-resolution structure determination of the large ribosomal subunit will be initiated using crystallographically determined phases calculated at 5.5 Å resolution by multiple isomorphous replacement and anomalous scattering (MIRAS) methods. Electron-density maps calculated using these phases begin to unveil the molecular architecture that underlies this large, ribonucleoprotein complex. First glimpses of the 7.0 Å resolution map show ribbons of electron density corresponding to the RNA backbone, punctuated at regular intervals by bumps arising from the phosphates (**Figure B-10**). Some of the proteins can be distinguished clearly from RNA at this resolution. Thus, we believe that a critical step has been taken on the path leading to an electron-density map of the large ribosomal subunit that can be interpreted in atomic detail.

X-ray crystallographic data collection for this project represents a significant technical challenge which, it appears, can be met by the facilities at NSLS. All the data collection to date has been done at NSLS beamlines X12C, X12B, and X25. For this purpose, numerous adjustments and modifications have been provided by the helpful staff at these beamlines. Initial heavy-atom data were taken at beamline X12C with the Brandeis four-module CCD-based detector. Using a MAR300 imaging-plate detector on beam line X12B we have been able to collect excellent 3.8 Å resolution data. We hope that in the future, when some additional modifications will be made, it will be possible to collect data to the diffraction limit of the crystals. ■

Figure B-10: Results from the study of the 50S ribosomal subunit from *Haloarcula marismortui*. Preliminary electron-density maps at 7Å resolution are shown and compared to the previously published 9Å resolution map^[13].

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Chemistry of SO₂ and Organosulfur Molecules on Metal and Oxide Surfaces: Unraveling the Mysteries behind Catalyst Poisoning and Desulfurization

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Sulfur dioxide is one of the major air pollutants released to the atmosphere as a result of volcanic activity and the combustion of fuels in power plants, factories, houses and transportation. On the other hand, organosulfur molecules are common impurities in oil-derived feedstocks and poison many metal/oxide catalysts used in commercial processes. Millions of dollars are lost every year due to the negative effects of sulfur poisoning. In the Catalysis Group at the Chemistry Department of Brookhaven National Laboratory, experiments are being carried out to obtain a fundamental understanding of

the effects of sulfur on the structural, electronic and chemical properties of metal and oxide surfaces. This type of knowledge is necessary when designing catalysts that have a high efficiency for the destruction or removal of sulfur-containing molecules (DeSO_x and hydrodesulfurization processes), or when choosing catalysts that have a low sensitivity to sulfur poisoning. The research projects make extensive use of the unique facilities available at the NSLS for surface and materials characterization.

Figure C-1. S 2p photoelectron spectra taken after annealing a Ru(001) surface saturated with SO₂ from 100 to 160, 260 and 350 K.

The chemistry of sulfur dioxide on Pt(111),^[1] Rh(111),^[2] Ru(001),^[3] and Mo(110)^[4] was examined at temperatures between 100 and 500 K. In experiments performed at beamline U7A, several sulfur species with distinct electronic properties were identified (for example, see Figure C-1). On all the metals, SO₂ dissociates (SO_{2,a} → S_a + 2O_a) or transforms into SO₃ and SO₄ species. No evidence was found for the existence of SO as a stable product. The reactivity of the metals increases following the sequence: Mo < Ru < Rh < Pt. It was found that adsorption geometries in which SO₂ is di-coordinated via O,O or S,O are the most probable precursors for dissociation.

Metal-metal bonding can have a drastic impact on the chemical affinity of a metal for SO₂ and other sulfur-containing molecules.^[1,2,5,6] A Sn/Pt(111) alloy is much less reactive towards SO₂ (Figure C-2), H₂S, S₂, and thiophene than polycrystalline tin or clean Pt(111).^[1] The electronic perturbations that accompany the formation of Pt-Sn bonds reduce the electron-donor ability of Pt and Sn, and both metals are not able to respond in an effective way to the presence of molecules like SO₂ or H₂S.^[1,5] Among the common site blockers (Cu, Ag, Au, Al, Zn, Sn), tin is the best choice for reducing the sensitivity of Pt reforming catalysts to sulfur poisoning.^[1,5] In a similar way, Pd/Rh automotive exhaust catalysts can have a good catalytic activity and be more tolerant to the presence of sulfur-containing molecules in the gasoline than pure Pd catalysts.^[2] A Pd monolayer supported on Rh(111) is less chemically active than pure Pd or Rh(111) for the dissociation of SO₂ or thiophene.^[2] A very large drop in the reactivity of Pd can be expected when this metal is bonded to s,p or early-transition metals.^[2] All these results together indicate that bimetallic bonding can be a useful "tool" for increasing the sulfur tolerance of Pt and Pd based catalysts.^[1,2,5] On the other hand, in hydrodesulfurization processes metal-metal bonding can be useful for enhancing the activity of Mo-based catalysts in the removal or transformation of organosulfur molecules.^[6]

Since most poisoned catalyst are expensive, there is a need to reactivate them. Reaction with oxygen (S_{solid} + O_{2,gas} → SO_{2,gas}) is a promising route for regenerating pure oxide catalysts. The process is carried out under pressures of oxygen (0.2-2 atm) that are not compatible with most of the techniques currently used in surface science. One can study it using x-ray absorption near-edge spectroscopy (XANES) and x-ray diffraction (XRD).^[8,9] Investigations at beamline X7B have established the feasibility of conducting subminute,

Figure C-2. (top) Structural geometry for a $(\sqrt{3} \times \sqrt{3})R30^\circ\text{-Sn/Pt(111)}$ surface alloy. The dark and white circles represent Sn and Pt atoms, respectively. The Sn atoms are present only in the top layer and protrude 0.22 Å from the plane of Pt atoms (from ref. [7]).

(bottom) Total sulfur uptake for the adsorption of SO₂ on polycrystalline tin, Pt(111) and a $(\sqrt{3} \times \sqrt{3})R30^\circ\text{-Sn/Pt(111)}$ alloy.

Figure C-3. Time-resolved x-ray powder diffraction patterns for the regeneration of sulfided NiMoO_4 . The sample was sent in an open capillary exposed to air and heated from 25 to 650 °C. Heating rate = 1.2 °C/min.

time-resolved XRD experiments under a wide variety of temperatures and pressure conditions ($-190\text{ °C} < T < 900\text{ °C}$; $P \leq 45\text{ atm}$).^[10] This unique approach has been used to study the regeneration of poisoned oxide catalysts.^[8,9] A typical case is shown in Figure C-3. After exposing a nickel molybdate (NiMoO_4) to H_2S , one sees the formation of metal sulfides and sulfates that change the x-ray diffraction pattern of the system.^[8,9] In the presence of O_2 this "mixture" is stable up to 250 °C when reaction begins.^[9] As the temperature is raised several

intermediates are observed, and at $\sim 400\text{ °C}$ the diffraction lines for pure NiMoO_4 appear. At 480 °C only the XRD pattern of NiMoO_4 is seen.^[8] Results of time-resolved XRD and XANES indicate that reaction with oxygen at high temperature is very effective for removing sulfur from poisoned NiMoO_4 , ZnO , NiO and MoO_3 catalysts. ■

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Using X-Ray "Vision" to Understand Self-Assembly

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INTRODUCTION

The concept of "self-assembly" (SA) is derived from biological systems and can be defined as a process by which molecules having many internal degrees of freedom are able to form ordered supermolecular structures utilizing weak interactions such as van der Waals forces or hydrogen bonding^[1]. Monolayers of alkyl-thiols grown on Au(111) surfaces are special among the many self-assembling systems because this system is widely considered as prototypical and has many potential technical

applications. In this system the gold-sulfur bond is strong enough to anchor the molecules to the surface, yet at the same time this interaction leaves sufficient lateral mobility to allow the inter-chain van der Waals interaction to drive the system toward the formation of a stable well-ordered monolayer. Here we review some of the phenomena that we have uncovered using x-ray scattering techniques to study the growth of decanethiol (C10) on Au(111) surfaces from the vapor phase (for details of sample preparation, etc., see references [2, 3, 4]).

Figure C-4. The coverage-temperature (Q,T) phase diagram of C10/Au(111) derived by GIXD (reprinted from Schreiber et al., 1998). The phases include the striped (S), the intermediate (IP), the C(4x2) (C) and the melted (L) phases, respectively (a schematic of each phase is also shown). The vertical lines indicate the nominal coverages of the striped phase (0.27ML), the nucleation of the C(4x2) phase (0.47ML), and saturation of the C(4x2) phase (1ML).

PHASE BEHAVIOR OF SAMS

The first complexity that must be overcome is to understand the different equilibrium structures that are present during growth. Early growth studies have interpreted SAM growth as a continuous process by which the alkyl chains within the monolayer collectively transform from a disordered to an ordered configuration^[5]. Instead, through a combination of X-ray, He atom diffraction and scanning tunneling microscopy, it has been shown that there are a number of distinct phases present during the growth process^[3,6]. In addition to the densely packed "standing up" C(4x2) phase at saturation coverage ($\Theta = 1 \text{ ML} = 4.6 \times 10^{14} \text{ molecules/cm}^2$), at low coverages a lying down "striped" phase ($\Theta = 0.27 \text{ ML}$) has been observed. By mapping out the temperature and coverage dependence of these two ordered phases using grazing incidence x-ray diffraction (GIXD), we have delineated^[3] the equilibrium phase behavior of this system (shown in Figure C-4). In addition to these two ordered phases there are at least two other *disordered* phases. There is an "intermediate" phase which is found at coverages intermediate between the C(4x2) and the striped phases (for $T < 15^\circ\text{C}$, with a nominal coverage of $\Theta = 0.47 \text{ ML}$), and a melted phase which apparently exists at all coverages for sufficiently elevated temperatures. Thus, at

equilibrium, the growth proceeds primarily through the *coexistence* of these distinct phases^[3].

A remarkable feature is that while a complete monolayer melts at $\sim 90^\circ\text{C}$, at $\Theta \sim 0.5 \text{ ML}$ the SAM melts at $\sim 15^\circ\text{C}$ (even though both phases are chemisorbed!). This dependence of T_m with Θ is one of the essential keys to understanding the SAM's ability to assemble into highly ordered structures^[3]. When growing in the presence of the melted phase ($T > 15^\circ\text{C}$) the order in the SAM (as measured by the average domain size) is substrate limited which is suggestive of significant diffusivity and Ostwald ripening of the growing islands. At lower temperatures ($< 15^\circ\text{C}$) the final SAM is significantly less ordered and the domain size evolution during growth is consistent with a fixed number of islands suggesting a loss of diffusivity at these temperatures^[3].

THE HEADGROUP-SUBSTRATE INTERFACE STRUCTURE AT THE BURIED INTERFACE

How can we understand the combination of high thermal stability coupled with an ability to assemble at, and even *below*, room temperature? This issue is closely related to the nature of the S-Au bond. While the vast majority of studies on these systems assume that the sulfur

Figure C-5. (A) A map of the two sulfur head-group positions at heights of $z_1 = 2.21 \pm 0.05 \text{ \AA}$ (green), and $z_3 = 2.97 \pm 0.05 \text{ \AA}$ (blue) with respect to the underlying substrate lattice as derived from the XSW data for $\Theta = 1.0 \text{ ML}$. The large circles denote the Au substrate lattice sites. (B) A schematic of the derived "sulfur-dimer" structure, in which only one of the two head-groups interacts directly with the substrate (green circle). The hydrocarbon chains are schematically shown as "R" (adapted from Fenter et al., 1998).

headgroup bonds to the Au surface in an hexagonal $\sqrt{3}\times\sqrt{3}$ 30° mesh with a S-S spacing of 5 Å^[7], our 2D crystallography results found that while the chain-chain spacing was indeed ~5 Å, the sulfur headgroups were instead arranged as sulfur dimers with a S-S spacing of ~2.2 Å^[8].

To resolve the ensuing controversy, we performed X-ray standing wave (XSW) measurements to directly probe the S/Au interface structure for this system^[2]. XSW is one of the few techniques which can directly and quantitatively probe a buried interface structure containing a low Z element such as sulfur by probing its XSW induced photoelectron yield near a substrate Bragg condition. The results (summarized in Figure C-5) reveal a highly detailed picture of the S-Au interface structure and directly contradict the widely assumed hexagonal S

mesh. We find that the two distinct S headgroup locations within the unit mesh each have a distinct height and lateral position^[2]. By combining these results with the information derived with GIXD^[8], we find that the S dimer is bound to the Au substrate through only one of the two S atoms with the presumed S-S bond being inclined by $19^\circ \pm 2^\circ$ with respect surface plane. The derived Au-S-S angle is consistent with the tetrahedral angle and therefore provides a natural context in which to understand the S-Au interface structure. The existence of a sulfur *dimer* species (which presumably interacts more weakly with a gold surface than a sulfur monomer) provides a simple yet plausible explanation for the observation of both a melted phase and significant surface diffusivity for these chemisorbed species at temperatures as low as 15°C.

Figure C-6. The C(4x2) growth rate is plotted as a function of impingement rate for two representative temperatures, and as a function of temperature for two impingement rates (reprinted from Eberhardt et al., 1998). At 40°C the dependence is linear, but at 5° C the growth rate increases *quadratically* with the impingement rate. Also shown is a schematic of the uni- and bi-molecular adsorption processes.

ADSORPTION MECHANISMS IN SELF-ASSEMBLY

In this system, SA proceeds in a two-step fashion^[2,3], of which the second step is associated with the formation of the C(4x2) phase and the final "assembly" of the monolayer. We have made use of the simple proportionality between the GIXD integrated intensity and the coverage of a particular phase (the C(4x2) phase) to follow the growth kinetics *in situ* and in real-time, as a function of the thiol impingement rate and the substrate temperature^[4]. The data (Figure C-6) clearly show that there are two distinct adsorption processes, each having a distinct dependence upon temperature and pressure. From the decrease of the growth rate vs. increasing temperature in both processes, we can directly conclude that the molecules adsorb as a physisorbed precursor phase and thermalize before either chemisorbing or desorbing from the surface (i.e., physisorption mediated chemisorption).

A unique feature of these data is that while the high temperature process varies *linearly* with the impingement rate, the low temperature process varies *quadratically* with this quantity^[4]. While a linear variation is ubiquitous in surface adsorption and can be understood phenomenologically as the result of a uni-molecular process (i.e., each molecule adsorbs or desorbs independently of other molecules), the quadratic variation can be understood as an associative adsorption process, whereby the rate-limiting step is a *bimolecular reaction* in

the physisorption well:

where "p" and "c" denote physisorbed and chemisorbed species, respectively. This bimolecular adsorption process is very unusual and may ultimately be the result of the peculiar adsorption energetics of this system^[9].

CONCLUSIONS

X-ray scattering techniques have allowed us to unravel many of the molecular-level aspects of SA of C10 on Au(111). We conclude that the growth of this system can be understood in terms of its phase behavior, bonding, and adsorption mechanisms, and the complexity of this system is *not* a direct result of the molecular structure (e.g., as a result of chain entanglement), although chain entanglement is expected to be important for longer aliphatic molecules (e.g., polymer brushes). In work presently in progress^[9], we are testing our level of understanding of the SA process by probing the behavior of molecules that have two headgroups (e.g., hexanedithiol) and by investigating the influence of the molecular backbone rigidity. ■

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Ionic States of Small Molecules Investigated by Vibrationally Resolved Core-level Photoelectron Spectroscopy

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Upon photoionization of a small molecule in the gas-phase, the excitation of one or several vibrational degrees of freedom is common. This occurs because the potential energy curve of the molecular ionic state is different from that of the neutral ground state. Since the photoionization process corresponds to a *sudden* change between the two states, the atomic core has no time to adiabatically respond to the change in conditions and is thus left in a state with potential energy. The ground state configurations of small molecules are usually well known and it is therefore possible to draw conclusions as to the nature of the excited state. This is especially true for the case of inner-shell ionization. However, the ability to record vibrationally resolved electron spectra at photon

energies sufficient to ionize an inner shell requires very good monochromatization of the incident light. In the last few years we have recorded core-level photoelectron spectra of several closed shell molecules at the X1B beamline^[1-3].

A good example of the determination of the ionic geometry in a closed-shell molecule is provided by the C 1s ionization spectrum of ethane (C_2H_6). Two vibrational progressions are visible in the C 1s photoelectron spectrum of C_2H_6 (Figure C-7). The first one, with vibrational energies around 400 meV, is typical for the photoionization of all small hydrocarbons and pertains to the totally symmetric C-H stretching vibrations. In terms of molecular geometries, excitation

Figure C-7. C 1s photoelectron spectra of ethane and deuterated ethane measured with 309 eV photon energy at beamline X1B. The results of a least-squares fit assuming the excitation of only two normal vibrational modes have been included. The dashed lines represent the individual vibrational states, while the full line through the data points represents the fit result after summation and convolution with the instrument profiles.

Figure C-8. Core level photoelectron spectrum of gaseous NO recorded at beamline X1B. Preliminary results from a least-squares fitting procedure are shown as dotted lines.

of this mode means that the C-H bond length shortens upon removal of the C 1s core electron. The second mode, with vibrational energies around 180 meV, was at first assigned to a totally symmetric C-C stretching vibration by several groups^[4]. However, calculations of the vibrational excitation probabilities have yielded the first evidence that another mode, namely an umbrella-like bending vibration of the hydrogens attached to the site of the core-hole, might play a stronger role than the movement of the two methyl groups against each other^[5]. By studying isotopically substituted ethane, C_2D_6 , in parallel with C_2H_6 , we have found that the frequency shift observed for the mode in question matches that of a C-H bending vibration, but not that of a CH_3-CH_3 stretching vibration. That is, the molecule changes its

C-C-H bond angle along with a change in C-H bond length. Quantitatively, vibrational energies of 176 ± 5 meV were found for this bending mode for ethane and 130 ± 10 meV for deuterated ethane. The C-H stretch was found to be 407 ± 5 meV and the C-D stretch to be 291 ± 10 meV. The changes in C-H bond length and C-C-H bond angle are 0.036 Å and 2.1° , respectively, assuming a delocalized core hole^[6].

Molecular Species with an unpaired electron determine an important part of the chemistry of the atmosphere despite their generally low concentration. As a step towards the spectroscopic characterisation of these systems, we have recently measured inner-shell photoelectron spectra of the open-shell molecules O_2 and NO. Upon removal of an inner-shell electron, both of these molecules, will, in principle, show a photoelectron line split into two components by the exchange interaction between the unpaired electrons. The high-resolution photoelectron spectra of these systems can therefore give information on the influence of this spin-dependent force on the binding state of the molecular ions in play.

Turning to open shell molecules, we note that in the ground state configuration NO has an unpaired electron in the 2π orbital. Together with the spin 1/2 of an inner-shell hole, the molecular states $^1\Pi$ and $^3\Pi$ are thus formed. These states can be easily distinguished after ionization of an N 1s electron: our value for the exchange splitting is 1.40(1) eV (Figure C-8). Both states show a similar vibrational progression, in which excitations of up to two vibrational quanta can be seen. The vibrational energy from a preliminary analysis is around 240 meV for both spin states. This is similar to the value of 241 meV found for the N_2 $N1s \rightarrow \pi^*$ excited neutral state^[3] and is much smaller than the value of 314(4) meV measured for inner-shell ionized N_2 . A close similarity between NO and π^* -excited N_2 is expected from the Z+1 model. Less evident are the differences in exchange splitting, which can be seen by comparing the N 1s ionization spectrum with the O 1s spectrum. The triplet and singlet components of NO O1s⁻¹ overlap each other and the singlet peak can be seen as a shoulder about 0.48 eV away from the larger triplet peak. This splitting is, to our knowledge, resolved here for the first time and was earlier predicted to have a smaller value by theory:

The authors of [7] give values of 1.48 eV at the N-edge and 70 meV at the O-edge. A plausible explanation in chemical terms seems to be possible in this case: As oxygen has a higher electronegativity than nitrogen, the charge density of the bonding orbitals is stronger on the O side. Therefore, the anti-bonding lone π^* electron preferentially adheres to the N side and causes a stronger exchange interaction with a lone core electron at the same site. However relaxation, i.e. the reaction of the molecular charge cloud to the core hole creation, seems to substantially influence the charge distribution. Otherwise, the failure of theory to reproduce the splitting quantitatively would not be explainable.

The inner-shell photoelectron spectrum of O_2 shows an easily observable separation into the $O\ 1s^{-1}\ ^2\Sigma$ and $^4\Sigma$ states (Figure C-9). An analysis of both lines reveals further differences. Although vibrational

components cannot be resolved for both of these lines, a significantly larger width of the $^2\Sigma$ component compared to the $^4\Sigma$ is clearly observed. A least squares fit results in a profile for the $^4\Sigma$ line which hardly shows any vibrational excitation. Interestingly, the $^2\Sigma$ line has vibrational components with a significantly larger spacing, 170(20) meV compared to 110(20) meV for the $^4\Sigma$ and larger excitation probability as well. One is therefore led to conclude that an ion with a stronger force constant and smaller bond-length is formed in the case of doublet ionized O_2^+ compared to quartet O_2^+ . This change in molecular potential is solely brought about by the different coupling between the lone inner-shell electron and the high-spin coupled $1\pi_g^2$ open valence shell. The high brightness of the X1B undulator has proved to be crucial in measuring high resolution core-level spectra of small molecules with high sensitivity. ■

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Figure C-9. $O\ 1s$ photoelectron spectrum of oxygen recorded at beamline X1B. Open dots are the data points, the solid line is the result of a least-squares fit for the total profile, while the dotted lines are results for the constituent components before convolution with the apparatus profile function.

X-ray Scattering from a Soluble Monolayer at the Water-Hexane Interface

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Measurements of the microscopic structure of liquid interfaces or surfactant assemblies at these interfaces have utilized primarily one of two geometries: (1) macroscopic, single liquid-*vapor* interfaces at which the tail groups of surfactants are exposed to the vapor, and (2) multiple, microscopic liquid-liquid interfaces existing within surfactant solutions. A macroscopic single liquid-vapor interface is oriented by gravity and, in turn, orients surfactants supported on its surface. This allows for detailed structural measurements using, for example, either x-ray or neutron reflectivity or x-ray grazing incidence diffraction. However, surfactants at liquid-liquid interfaces in solution are in a chemical environment typically used for either man-made or biological applications. Recent efforts have combined the advantages of these two geometries in a third geometry, a macroscopic single liquid-liquid interface^[1-5]. In particular, our ability to flatten otherwise very curved interfaces allows x-ray surface scattering techniques to be used in the study of a wide range of liquid-liquid interfacial phenomena.

Here, we describe an x-ray surface scattering study of the structure of a monolayer of soluble surfactants at the water-hexane interface. The few available thermodynamic or spectroscopic measurements indicate that soluble and insoluble amphiphilic monolayers at water-oil interfaces are more loosely packed than the corresponding monolayers at the water-vapor interface^[6,7,8]. In particular, monolayers formed from surfactants soluble in the oil phase are expected to be disordered and in a liquid or gas phase at liquid-liquid interfaces^[8]. Contrary to this expectation, we find that soluble monolayers can form solid monolayer phases at the water-oil interface. In addition, the system described below exhibits a solid to gas phase monolayer transition at this interface. Comparison to a similar transition at the liquid-vapor interface indicates that the solvent acts to disorder the solid monolayer at the liquid-liquid interface at a lower temperature.

Monolayers of $F(CF_2)_{10}(CH_2)_2OH$ (denoted here as $FC_{12}OH$) self-assemble from a solution (2×10^{-3} mol/kg) in hexane onto the solution-water interface^[9, 10].

Figure C-10. X-ray reflectivity from the water-(hexane solution of $F(CF_2)_{10}(CH_2)_2OH$) interface at $T=32^\circ\text{C}$, triangles; $T=48^\circ\text{C}$, circles; pure water-hexane interface at $T=32^\circ\text{C}$, open squares. Inset is a cartoon of the solid surfactant monolayer that forms at $T=32^\circ\text{C}$. Also indicated are the x-rays reflecting off the top and bottom of the monolayer. The constructive interference of these x-rays is responsible for the increase in reflectivity.

Figure C-11. Surface coverage of the lower temperature solid monolayer phase on a heating-cooling cycle through the solid-gas monolayer transition of $\text{F}(\text{CF}_2)_{10}(\text{CH}_2)_2\text{OH}$ at the water-(hexane solution) interface.

Figure C-10 illustrates x-ray reflectivity measurements at $T=32.00 (\pm 0.03)^\circ\text{C}$ from the monolayer at the water-(hexane solution) interface (plotted as a function of the wave vector transfer normal to the interface,

$Q_z = (4\pi/\lambda)\sin(\alpha)$, where $\lambda = 0.0825 \pm 0.0002$ nm is the x-ray wavelength for these measurements and α is the reflection angle measured from the plane of the interface). Also shown are measurements from the pure water-hexane interface at the same temperature and measurements from the water-(hexane solution) interface at $T=48.00^\circ\text{C}$. The enhanced reflectivity at higher Q_z from the water-(hexane solution) interface at $T=32.00^\circ\text{C}$ is due to a constructive interference of x-rays reflected from the top of the fluorinated monolayer with x-rays reflected from the bottom of the monolayer. The reflectivity from the water-(hexane solution) interface at $T=48.00^\circ\text{C}$ is nearly identical to the measurements from the pure water-hexane interface and indicates that most of the FC_{12}OH molecules have desorbed from the interface at this temperature^[11].

These data can be analyzed by using a general expression, derived from the first Born approximation for x-ray scattering, that relates the reflectivity to the electron density gradient normal to the interface, $\frac{d\langle\rho_e(z)\rangle}{dz}$ (averaged over the interfacial plane), and written as ^[12]

$$\frac{R(Q_z)}{R_F(Q_z)} \cong \left| \frac{1}{\Delta\rho_{e,bulk}} \frac{dz \frac{d\langle\rho_e(z)\rangle}{dz}}{\exp(iQ_z z)} \right|^2 \quad (1)$$

where $\Delta\rho_{e,bulk} = \rho_{e,bulk,lower} - \rho_{e,bulk,upper}$ and $R_F(Q_z)$ is the Fresnel reflectivity predicted for an ideal, smooth and flat interface^[13]. The layer of FC_{12}OH is modeled simply as a thin slab of higher electron density sandwiched between two bulk liquids. The interfaces of the top and bottom of this slab are roughened by thermal capillary waves, characterized by the roughness parameter, σ . The reflectivity calculated from Eq.(1) using the slab model for the electron density is fit to the data to yield values for the three fitting parameters: the slab thickness $L = 1.24 \pm 0.03$ nm, $\sigma = 0.36 \pm 0.02$ nm, and the fluorinated slab electron density (normalized to $\rho_{bulk,water}$) $\rho_f = 1.90 \pm 0.04$ (Figure C-10) ^[11].

Our measurement of the electron density of the monolayer at $T=32.00^\circ\text{C}$, $\rho_f = 1.90 \pm 0.04$, corresponds to a mass density of 2.19 ± 0.05 g/cm³. This agrees with the density of bulk solid fluoroalkane phases (e.g., for $n\text{-C}_{20}\text{F}_{42}$) which have a density of either 2.23 g/cm³ for the monoclinic crystal phase or 2.16 g/cm³ for the rhombohedral rotator solid phase ^[14]. These measurements exclude the possibility that the FC_{12}OH monolayer at the water-hexane interface is in a liquid monolayer phase (bulk liquid fluoroalkanes have a mass density of approximately 1.7 g/cm³) ^[14-16].

A similar analysis of the data from the water-(hexane solution) interface at $T=48.00^\circ\text{C}$ indicates that a conservative upper limit to the surface coverage of FC_{12}OH is approximately 1.5%^[11]. The transition from the solid FC_{12}OH monolayer to this gaseous monolayer at higher temperature can be studied by measuring reflectivity as a function of temperature. Figure C-11 illustrates the first heating-cooling cycle through this transition. We believe that the transition region represents an interface with coexisting solid and gas monolayer phases. This is consistent with enhanced off-specular diffuse scattering that we measured in this transition region (not shown)^[17]. Accordingly, the reflectivity has been interpreted to yield the surface coverage of the lower temperature solid phase monolayer. Preliminary data indicate that the hysteresis in Figure C-11 is reduced with further heating-cooling cycles through the transition temperature.

An important feature of studies of the liquid-liquid interface is the ability to characterize the role of the upper phase on the structural order within, for example, monolayers at the interface. To gain some insight into

the role of the upper-phase hexane on the ordering of the low temperature FC_{12}OH solid phase monolayers, we studied a similar transition in FC_{12}OH monolayers supported on the water-vapor interface. The FC_{12}OH monolayer was spread in the crystalline island phase at an inverse density of $0.4\text{ nm}^2/\text{molecule}$. Since the crystalline unit cell size is $0.29\text{ nm}^2/\text{molecule}$ there is excess area at the interface that allows the monolayer to undergo a transition to a liquid or gaseous disordered phase. X-ray grazing incidence diffraction measurements (not shown) indicate that a solid to gas (or, possibly liquid) monolayer phase transition occurs between 58.8°C and 62.09°C , slightly more than 20°C higher than the solid-gas transition in the FC_{12}OH monolayer at the water-(hexane solution) interface^[11]. Therefore, the presence of the hexane solvent acts to disorder the solid monolayer at a lower temperature. ■

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GEOLOGICAL SCIENCES

Chemical Analysis of Impact Material on a Dust Collector Flown on the MIR Space Station

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Low density silica aerogel collectors have been flown on earth-orbiting satellites to collect samples of earth-orbital debris and interplanetary dust in order to characterize the flux and types of small particles in the near-earth environment. This effort has three objectives:

- 1) to determine the relative contributions of man-made orbital debris and interplanetary dust to the flux,
- 2) the chemical analysis of the man-made debris to infer the sources of the most abundant types of particles which may be hazardous to earth-orbiting spacecraft, and,
- 3) the chemical analysis of interplanetary dust particles which have never resided in the stratosphere as a monitor of possible stratospheric contamination of the interplanetary dust particles collected by NASA from the earth's stratosphere.

High-velocity (km/sec) particles penetrate the aerogel, leaving a cone-shaped damage track, sometimes having a surviving particle at the end (as shown in **Figure G-1**). In 20 mg/cc density aerogel a hypervelocity particle will typically produce a track whose length is about 1000 times the diameter of the particle. Thus, a 10 micrometer diameter particle will stop in a distance of about 1 centimeter. Depending on the entry angle of the particle relative to the aerogel

surface, a 10 micrometer diameter particle may stop as deep as 1 centimeter below the surface, with a typical depth of ~0.5 centimeters.

Previously, chemical characterization of these

• Figure G-1. Image of a high-velocity particle captured in low-density silica aerogel.

captured particles has required their removal from the aerogel. The penetration depth of the analysis electron beam in instruments such as electron microprobes is inadequate for the analysis of particles buried millimeters to centimeters beneath the surface of the aerogel. However, high-energy x-rays are not readily absorbed, and the inherent limitation on in-situ analysis of particles beneath the aerogel surface results from the absorption of the fluorescence x-rays. We have developed an *in situ* analysis technique^[1], performing x-ray fluorescence elemental analysis using the X-Ray Microprobe on Beamline X26A of the National Synchrotron Light Source, to characterize the heavy elements in particles at depths of up to several centimeters in low density aerogels. The aerogel is composed mostly of Si, but trace amounts of Ti, Fe, Cu, and Zn are also present. Thus, the composition of particles or their residue is inferred by observing significant excesses in these elements over the signal from adjacent regions of the aerogel.

The X-Ray Microprobe on Beamline X26A (and previously on Beamline X26C) has been used for the chemical analysis of interplanetary dust particles collected by NASA stratospheric sampling aircraft for the past decade^[2]. The chemical compositions of more than 200 interplanetary dust particles have been measured, and these provide a basis for comparison with particles captured in earth orbit. The most abundant type of interplanetary dust collected from the Earth's stratosphere is similar in chemical composition to the chondritic meteorites. Chondritic material has an Fe/Ni ratio of about 17, quite distinct from the much higher Fe/Ni ratio in terrestrial surface rocks. We use this Fe/Ni ratio to identify likely interplanetary dust particles. This identification is not perfect, since stainless steel orbital debris could also have high Ni. Some types of orbital debris, such as Ti-rich paint flakes, can also be identified on the basis of their chemical composition.

Thus far, we have examined five pieces of aerogel, each having a surface area of about 1" by 2", exposed for 18 months on the MIR Space Station. Two contained a series of parallel tracks, several having particles at the end, indicating a "swarm" of particles struck the aerogel simultaneously. All 14 of the tracks in these two swarms

Figure G-2. X-ray fluorescence spectrum of a particle captured in aerogel exposed for 18 months on the MIR Space Station. This particle contains Fe, Ni, Cr and possibly Mn, in roughly chondritic proportions, indicating it is an interplanetary dust particle rather than orbital debris.

contained Fe residue, either distributed along the track or in particles at the end of the track. In three cases we also detected Ni in the residue or particles, with an Fe/Ni ratio similar to the chondritic meteorites. The largest of these particles, several micrometers in size, had detectable Fe, Ni, Cr, and possibly Mn, present in roughly chondritic proportions (**Figure G-2**). This composition provides an almost certain identification of this particle, and the other particles in this swarm, as extraterrestrial. Further analysis of these swarm particles is in progress.

We also analyzed an apparent particle at the end of a track not associated with a swarm. No elements were detected in this particle. Because this particle was several millimeters below the aerogel surface, Ca was the lightest element we would expect to detect. The apparent particle might simply be compacted aerogel at the end of the track, or it might be composed predominately of Si (which cannot be detected because of the Si signal from the aerogel) or of elements lighter than Ca.

Other impacting material produces shallow pits in the aerogel, sometimes with debris from the impacting particle on the bottom or walls of the pit. The residue in these pits was also characterized using the X-Ray Microprobe. We examined three aerogel pieces which contained pits. One pit showed K and Ca in the bottom, consistent with the impact of human waste. Examination of two other pits showed no element enhancements,

suggesting that either the impactor consisted predominately of low-Z material (lower than Al), which we cannot detect with the X-Ray Microprobe, or silicon, or that little residue from the impacting particle was retained in the these pits.

This *in situ* analysis of particles captured in aerogel also serves to develop and demonstrate analytical techniques that will be important for NASA's upcoming STARDUST mission. STARDUST, which is scheduled for launching in February 1999, carries a large aerogel collector that will fly through the dust cloud of Comet Wild-2, with a relative velocity of about 6 km/sec,

collecting more than 1000 cometary dust particles larger than 10 micrometers in size. The rear surface of the aerogel will be exposed during the cruise-phase of the STARDUST mission in an effort to collect hundreds of 0.5 micrometer size interstellar dust grains. The STARDUST aerogel collector is scheduled to return to Earth in 2006. Because the removal of individual particles from the aerogel would be tedious, and would leave open the possibility of contamination during extraction, *in-situ* particle characterization, using and X-ray Microprobe, is likely to be an important part of the preliminary characterization of these particles. ■

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Figure G-3. IDP (~10 mm) collected from the stratosphere showing the aggregated, porous texture (NASA/JPL/Caltech).

Comparative Rheology of Mantle Phases

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The mineralogy of the Earth's mantle changes with depth owing to pressure induced phase transformations. Olivine, pyroxene and garnet are expected to be the stable minerals in the upper 400 km of the mantle; garnet, wadsleyite and ringwoodite likely dominate the region from 400 - 700 km depth; and perovskite and ferropericlase are the stable phases at depths greater than 700 km. Rheological properties of the relevant minerals are important for defining dynamic earth phenomena such as mantle convection and the origin of deep earthquakes. Using a newly developed in-situ high pressure x-ray diffraction technique at X17B^[1,2], we have measured rheological properties of the predominant mantle phases at elevated pressure (to 20 GPa) and temperature (to 1300K).

Stress held in the powdered sample is measured as a function of pressure, temperature and time by monitoring the x-ray diffraction line profile. The line broadening is

generally caused by either elastic strain or small grain size^[3,4]

$$B^2 = [K(1/2)hc / (P \sin \theta)]^2 + [2\epsilon E]^2$$

where B represents the line breadth (refers to full width at half maximum of a gaussian profile in our experiments), K is the Scherrer constant, h is Planck's constant, c is the velocity of light, P is the average crystallite size, 2θ is the fixed scattering angle, ϵ is the upper limit of strain (refers to elastic shear strain in our experiments), and E is the x-ray photon energy. Product of the aggregate Young's modulus of the mineral and the measured strain derived from above expression yields the deviatoric stress supported by the sample.

The experiments are conducted in the multi-anvil high pressure apparatus, SAM85^[5]. Deviatoric stress is generated through the interaction of individual grains of the sample. Stress relaxation measurements are

accomplished by observing the decay of stress-induced diffraction-peak broadening as a function of time and temperature. The minerals we have studied are olivine, wadsleyite, ringwoodite, garnet, perovskite and periclase. Hydrous phases of olivine, wadsleyite, ringwoodite are also studied to investigate the water weakening on the rheological properties of minerals in the mantle. The experiments are carried out by first compressing the sample at room temperature, then stepping up and holding the temperature at desired interval for the stress relaxation measurements.

Figure G-4 illustrates the experimental result on anhydrous and hydrous olivine. The deviatoric stress in both anhydrous and hydrous phases increases with applied pressure at room temperature until the room-temperature yield strength is reached. The stress in the hydrous phase saturates at a lower level than that in the anhydrous phase, which shows that the hydrous phase is weaker at room temperature. At an applied pressure of 10 GPa the yield strength of the hydrous α is about 28% less than that of the anhydrous phase. When the samples are heated up to 400°C, thermal yielding results in significant stress drop in the both phases. The hydrous α shows again weaker behavior than the anhydrous phase, and the yield strength of the hydrous α drops by 62% while that of

the anhydrous phase drops by 39%. Upon heating to 600°C, the yield strength of the anhydrous α drops further to about the same strength level of hydrous phase, and the hydrous phase does not show any obvious decrease in yield strength. The anhydrous olivine shows stress relaxation as a function of time at temperatures of 400°C and 600°C. Yield strength data from single crystal San Carlos olivine at room pressure by Evans and Goetze^[6] are superposed on the figure showing that our data agree well with theirs at low temperature.

In contrast to olivine, its higher pressure - deeper depth phases, wadsleyite and ringwoodite, are much stronger than olivine and similar to each other. Furthermore, water only slightly weakens wadsleyite and ringwoodite while weakens olivine dramatically. **Figure G-5** shows the result of wadsleyite.

The rheological property of garnet is comparable to wadsleyite and ringwoodite, and periclase is as weak as olivine. Standing out of all these phases, perovskite is by far the strongest phase. In the range of investigation, temperature doesn't result in any significant stress drop as seen in the other phases. It only changes the relaxation rate. The stress at 800°C remains as high as 75% of that at room temperature at 20 GPa (**Figure G-6**).

Figure G-7 shows a summary of the stress capacity of the predominant mantle phases at the indicated temperatures. The temperature-depth profile of this figure is similar to that expected in a subduction zone where cold material is sinking and where deep earthquakes occur (to depths of 700 km). The general conclusion is that olivine and periclase are by far the weakest phases, with majorite, wadsleyite, and ringwoodite of comparable, but intermediate strength, and perovskite is significantly the strongest of all. The experiments represent the first high

temperature measurement of the rheological properties of most of these materials. The results suggest that the bimodal distribution of earthquake activity with depth and the cessation of earthquakes at 700 km is simply a reflection of the strength dependence and stress relaxation character of the stable phases and thus does not require different triggering mechanism for earthquakes above and below 400 km. The experimental observations are also consistent with a significant viscosity increase at the upper mantle-lower mantle boundary. ■

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Figure G-7. Shear strength of predominant mantle phases at indicated temperature. The depth represents where the minerals are stable in the earth. Ol: olivine, β: wadsleyite, γ: ringwoodite, Mj/Py: majorite pyrope (1:1) garnet, Pv: perovskite.

Iron in Martian Meteorites: Microanalyses of $\text{Fe}^{3+}/\Sigma\text{Fe}$ by Synchrotron MicroXANES (SmX) as Indicators of Variable Oxygen Fugacity

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Igneous rocks from Mars are believed to be sampled by SNC or martian meteorites. Martian magmatism is inferred to have produced basaltic and more evolved magmas from a peridotitic mantle^[1,2]. These rare meteorites cannot be realistically sampled for destructive measurements of $\text{Fe}^{3+}/\Sigma\text{Fe}(\text{Fe}^{2+}+\text{Fe}^{3+})$ which is used as a proxy for the oxygen budget of the planet, so that nondestructive microanalysis is essential for improved understanding of this important parameter. Variation of oxygen fugacity ($f\text{O}_2$), causes compositional diversity among terrestrial magmas^[3,4] and the ratio $\text{Fe}^{3+}/(\text{Fe}^{2+}+\text{Fe}^{3+})$ is used as a monitor of oxygen fugacity, particularly when applied to coexisting minerals. Ultimately $\text{Fe}^{3+}/(\text{Fe}^{2+}+\text{Fe}^{3+})$ provides a fundamental constraint on the abundance oxygen, this most abundant element (volumetrically) in any crust and mantle of all the rocky planets. The few reliable measurements of $\text{Fe}^{3+}/(\text{Fe}^{2+}+\text{Fe}^{3+})$ in Martian meteorites were done on bulk samples and are difficult to apply to the crystallization history of the rocks because weathering effects complicate the results^[5]. Partitioning data for Fe^{2+} and Fe^{3+} among the coexisting minerals are needed to determine fugacity sensitive magmatic equilibria for Martian rocks. Here we provide *in situ* microanalyses of $\text{Fe}^{3+}/(\text{Fe}^{2+}+\text{Fe}^{3+})$ using the XANES microprobe at X26A for minerals in eight meteorites that may be used to estimate oxygen fugacity in Martian crustal rocks. The data are comparable with those from terrestrial rocks buffered at the quartz-magnetite-fayalite and nickel-nickel oxide reactions and are distinct from products of asteroidal magmatism buffered by iron-wüstite reaction^[6,7,8]. The results indicate that the crust of Mars is relatively oxidized and comparable with that of the Earth. The results reflect Martian magmatism, shock effects and probably hydrothermal activity.

To document oxygen fugacity related equilibria among coexisting minerals, $\text{Fe}^{3+}/\Sigma\text{Fe}$ data for each mineral being considered must be acquired at petrologically constrained locations on mineral grains. Ninety three spectra of the Fe K-absorption edge (Figure G-8) were acquired using Synchrotron microXANES (SmX) spectroscopy for 12x15 micrometer areas on coexisting

olivine, pyroxene, plagioclase/maskelynite, oxides, and carbonate in petrographic thin sections of eight Martian meteorites: ALH77005, ALH84001, Chassigny, EET79001, LEW88516, Nakhla, Shergotty and Zagami. The spectra were processed to extract quantitative estimates of $\text{Fe}^{3+}/\Sigma\text{Fe}$. Sm measurements were made using the X-ray fluorescence microprobe facility (beamline X26A) at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratory. The spatial resolution

of SmX permits microanalyses to be made avoiding oxide inclusions in many minerals that confound bulk analytical determinations of $\text{Fe}^{3+}/\Sigma\text{Fe}$. The energy shifts of a pre-edge feature in the Fe K-absorption spectra are a function of oxidation state^[9] and were calibrated against the $\text{Fe}^{3+}/\Sigma\text{Fe}$ values in standard minerals^[10]. SmX spectra of a broad range of rock-forming minerals from terrestrial rocks that were independently analyzed by either Mössbauer or wet chemical techniques^[11,12] were also used to test the consistence of the results. The grain size of these meteorites is in the sub-millimeter range, so the spatial resolution of SmX permits variation of $\text{Fe}^{3+}/\Sigma\text{Fe}$ to be detected within mineral grains.

Results: Every mineral shows variation of $\text{Fe}^{3+}/\Sigma\text{Fe}$ from meteorite to meteorite and within a single meteorite. $\text{Fe}^{3+}/\Sigma\text{Fe}$ is lowest in olivine and highest in feldspathic phases (Figure G-9 - Table)

OLIVINE

Meteorites ALH77005, LEW88516 and Chassigny all contain significant olivine. (Our EET79001 is from a basaltic lithology B with no olivine). Olivine is generally not a host for Fe^{3+} so $\text{Fe}^{3+}/\Sigma\text{Fe}$ values of 0 are normal. Colorless olivine in these meteorites contains $\text{Fe}^{3+}/\Sigma\text{Fe}$ less 0.02. Olivine in ALH77005 and LEW88516 is both colorless and brown as a result of shock induced modifications^[2]. Brown olivine has values of $\text{Fe}^{3+}/\Sigma\text{Fe}$ up to 0.12 in ALH77005 and 0.26 in LEW88516 in which very deeply colored healed fractures through the grains have the highest values. Mössbauer analysis 13 of $\text{Fe}^{3+}/\Sigma\text{Fe}$ in bulk olivine from ALH77005 gave 0.045, and probably represents the average of the microscale variability seen. In Chassigny, olivine $\text{Fe}^{3+}/\Sigma\text{Fe}$ varies from 0 to 0.16 but does not correlate with color variations. The range observed is consistent with recent TEM observations of exsolved lamellae containing iron oxides in Chassigny olivine^[14].

PYROXENE

Pyroxene is present in all the Martian meteorites and is the most useful mineral for examining the variation of $\text{Fe}^{3+}/\Sigma\text{Fe}$ with fO_2 . In general, pyroxene in the Iherzolitic meteorites has lower $\text{Fe}^{3+}/\Sigma\text{Fe}$ than in the basaltic meteorites (Figure G-9). $\text{Fe}^{3+}/\Sigma\text{Fe}$ in two pyroxene species in ALH77005 varies (0.09-0.3) and may correlate with variations of Ti, Al and Cr and the pyroxene species (pigeonite or augite), but insufficient $\text{Fe}^{3+}/\Sigma\text{Fe}$ analyses are currently available to confirm these correlations. The pyroxenites examined, Nakhla (augite) and ALH84001 (orthopyroxene), have similar $\text{Fe}^{3+}/\Sigma\text{Fe}$ values (0.09 - 0.14) to the Iherzolitic samples. The observed values are, however, comparable with the lower part of the $\text{Fe}^{3+}/\Sigma\text{Fe}$ range measured in mantle peridotites

and pyroxenites on Earth^[7]. The observed values are typical for terrestrial orthopyroxenes (~0.1) and lower than those of clinopyroxene equilibrated at the quartz-magnetite-fayalite buffer curve.

In Nakhla, spectra of several grains and a traverse across a single grain of augite revealed homogeneous $\text{Fe}^{3+}/\Sigma\text{Fe}$ (0.09), similar to the bulk meteorite value^[15], except where the analysis included a micrometer scale oxide inclusion has higher $\text{Fe}^{3+}/\Sigma\text{Fe}$ (0.16). Analyses that overlapped grain boundaries in Nakhla showed elevated $\text{Fe}^{3+}/\Sigma\text{Fe}$ (0.30). The basaltic meteorites, Shergotty and Zagami, and the basaltic lithology B of EET79001 have high $\text{Fe}^{3+}/\Sigma\text{Fe}$ (0.2 - 0.3) consistent with their having crystallized at or above the QFM buffer^[16]. Variations of $\text{Fe}^{3+}/\Sigma\text{Fe}$ in both Zagami and Shergotty may reflect steric differences between coexisting pigeonite and augite in these meteorites, but may also suggest that pigeonite has variable $\text{Fe}^{3+}/\Sigma\text{Fe}$ in both meteorites. Eucritic pigeonite, from asteroidal basalts, is dominated by Fe^{2+} as expected from the inferred oxygen fugacity at or below the iron-wüstite buffer.

FELDSPAR

The Iherzolitic meteorites ALH77005 and LEW88516 and the basaltic lithology of EET79001 have relatively constant $\text{Fe}^{3+}/\Sigma\text{Fe}$ (~0.31) in feldspar. The basaltic meteorites, Shergotty and Zagami, however, are more oxidized with $\text{Fe}^{3+}/\Sigma\text{Fe}$ ~0.48. The increase of oxidation ratio in pyroxene from the Iherzolitic to basaltic meteorites is repeated in feldspar data. Compositional differences between the maskelynite in EET79001 lithology B and that of the basaltic meteorites suggest that the coupled substitution $\text{Na}+\text{Si}=\text{3D Ca}+\text{Al}$ may have altered the abundance of sites most compatible with Fe^{3+} (Al) and Fe^{2+} (Ca) in plagioclase and that the feldspar results may reflect crystal chemical controls in addition to oxygen fugacity effects. Insufficient spectra are presently available to test this possibility and to document compositional variation within individual meteorites.

OTHER MINERALS

Oxide spectra from Nakhla suggest $\text{Fe}^{3+}/\Sigma\text{Fe}$ values of 0.4 - 0.5 similar to the previous calculated results^[17]. The low intensity of the pre-edges of the Fe-XANES spectra suggest that the oxide minerals have little tetrahedrally coordinated Fe. The oxide minerals may be exsolved ilmenite-hematite solid solutions. Iddingsite spectra from Nakhla appear to be dominated by this oxide and give $\text{Fe}^{3+}/\Sigma\text{Fe}$ of 0.45. A preliminary calcite spectrum for ALH84001 suggests $\text{Fe}^{3+}/\Sigma\text{Fe}$ at 0.14. Discussion: There are three independent processes that may have controlled the $\text{Fe}^{3+}/\Sigma\text{Fe}$ of the Martian meteorites: (1) oxidation caused by magmatism in a relatively oxidized

magmatic system. (2) hydrothermal alteration and (3) shock induced effects. Detailed $\text{Fe}^{3+}/\Sigma\text{Fe}$ measurements on coexisting phases and correlation of the oxidation state results with other microanalytical and crystallographic data will be needed to distinguish among these processes.

The general trend of increasing $\text{Fe}^{3+}/\Sigma\text{Fe}$ from the peridotitic samples to the basaltic samples suggests that magmatism on Mars became more oxidized as it evolved (again comparable to much terrestrial magmatism) and is consistent with the presence on the modern Martian surface of abundant ferric oxide minerals^[18]. Highly fractionated silicates discovered by Pathfinder/Sojourner at Sagan Memorial Station on Mars 1 should have high Fe^{3+} in pyroxene and feldspar and we anticipate that SmX measurements on returned samples, when available, will confirm this prediction. It is unlikely that magmatism is the only contributor to the range of oxidation in the Martian suite. Shergotty and Zagami are young and may have erupted on a (partially) dehydrated planet. The mantle from which they were extracted may well have been wet and capable of oxidizing the magmas. Old meteorites such as ALH84001 sample lithologies may have formed when Mars had extant surface water and hence had a significantly wetter mantle. The role of water in the formation, dehydrogenation and metasomatism of peridotitic and pyroxenitic rocks in the terrestrial mantle is increasingly recognized^[19] and is an important target for Martian studies. The roles of both hydrothermal

activity and shock modification of $\text{Fe}^{3+}/\Sigma\text{Fe}$ may be important. Both ALH77005 and LEW88516 experienced major shock events that generated abundant defects in the olivine.

The textures observed in olivine of these lherzolites are consistent with post shock annealing seen in experimental charges^[20]. The brown color in this olivine may result from light scattering from the defects, oxidation of the defects, or both. The Martian atmosphere is oxygen poor ($p\text{O}_2 \sim 10^{-6}$) so any shock induced oxidation probably involved other oxygen bearing species - perhaps atmospheric CO_2 . Water present in the Martian crust when ALH77005 was shocked may provide an alternate oxidizing agent. The time of oxidation is, however, uncertain. The presence of significant amounts of water in the Martian crust make hydrothermal processing of some crustal rocks seem very likely. In ALH77005, crystalline feldspar rims replacing the shock produced maskelynite glass, strongly implies a period of extended subsolidus heating of this rock during which hydrothermal interactions might have occurred. In LEW88516, the presence of deeply colored, healed fractures in olivine requires more localized oxidation processes than in ALH77005 and suggests that the olivine was first shocked, and then oxidized during a hydrothermal recrystallization event caused by the flux of an oxidizing agent through cracks. The absence of tight temperature constraints and of data for the coexisting magmas and fluids that equilibrated with the Martian meteorites prevents the

Figure G-9. (Table) Synchrotron microXANES analyses of $\text{Fe}^{3+}/\Sigma\text{Fe}$ in minerals from Martian meteorites.

Meteorite	Olivine	Pyroxene	Feldspar	Oxide	Others
Shergotty	n.p.	0.28 ± 0.05	0.47 ± 0.10	na	
Zagami	n.p.	0.212 ± 0.08 (0.171 ± 0.04)	0.49 ± 0.08	na	
Nakhla	na	0.09 ± 0.01 clean ' $0.17 - 0.35$ ' cloudy	na	0.49	Iddingsite 0.42 ± 0.05
Chassigny	0.06 - 0.16	0.35	na	na	
ALH77005	0 - 0.05 clean 0.12 brown	$0.09 - 0.30$	0.32	na	
EET79001	na	$0.27 - 0.3$	0.32 ± 0.05	na	
LEW88516	0 - 0.03 clean 0.11 - 0.26 brown	na	0.30 ± 0.07	na	
ALH84001	na	0.14 ± 0.01	na	na	cc 0.14

n.p.=not present, na=not analyzed

determination of $\text{Fe}^{3+}/\Sigma\text{Fe}$ partition coefficients from the present results. However, the data confirm the general trends of partitioning for ferric and ferrous in terrestrial magmatic systems and appear to be consistent with oxygen fugacities ranging from below the buffer curve quartz-fayalite-magnetite to about that of the nickel-nickel oxide buffer. These data provide the first petrologically constrained sampling of $\text{Fe}^{3+}/\Sigma\text{Fe}$ from any meteorite parent body. They differ from bulk

measurements of $\text{Fe}^{3+}/\Sigma\text{Fe}$ as the difficulties caused by contaminants such as weathering products and micrometer scale inclusions are easily excluded. The results for the Martian suite are generally consistent with the formation of these rocks in a very "terrestrial", i.e. oxidized, setting. The range of $\text{Fe}^{3+}/\Sigma\text{Fe}$ seen is comparable to that seen in many mantle and eruptive rocks on Earth. Martian samples are much more oxidized than lunar, basaltic achondrite and most chondritic meteorites. ■

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First XAFS Measurements on Ferrous Chloride Hydrothermal Solutions using a Novel Diamond Anvil Cell

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The decrease in density, viscosity, and dielectric constant of water with temperature signals that appreciable changes take place in its structure under sub- and supercritical conditions. Very little is known about how such changes in the physical properties of water, in going from ambient to supercritical conditions, influence the nature of short-ranged interactions between solvated ions. Determination of the structure and speciation of complex ions in hydrothermal solutions under controlled thermodynamic conditions is an essential step in developing a theoretical basis of electrolytes beyond the Debye-Hückel theory. Systems such as iron(II) chloride aqueous solutions have a two-fold interest for us: 1) Current theories of the molecular electronic structure and metal-ligand interactions for first-row transition metal ions are among the most developed^[1] and thus offer an opportunity for development of an extensive knowledge base of such electrolytes, and, 2) they have direct relevance to studies in hydrothermal geochemistry^[2,3].

Conventional metal autoclaves have proven restrictive for studies on dissolved salts in supercritical water because of the severe corrosion problems caused by such fluids. The hydrothermal diamond anvil cell developed by Bassett *et al.*^[4] offers a much more suitable alternative because corrosion problems can be greatly minimized while control over pressure, temperature, and concentration can be substantially enhanced. However, x-ray absorption experiments have been limited to the higher photon energies (> 15 keV) because of the attenuation of the beam by the diamonds. We have developed a new diamond anvil cell suitable for

making x-ray absorption studies of first row transition metal ions in supercritical aqueous solutions. Each diamond has been laser drilled to within 150 micrometers of the anvil face, minimizing the loss of x-ray intensity due to absorption and scatter by diamond. We have for the first time measured Fe K-edge X-ray Absorption Fine Structure (XAFS) spectra from 2m Fe(II)Cl₂/NaCl/H₂O solutions, at temperatures ranging from 25-500°C and pressures estimated up to 200 MPa, using the microprobe capabilities of beam line X26A. The capabilities of the

new diamond anvil cell were demonstrated more fully in a previous XAFS study made on zinc chloride aqueous solutions, at beam line B-2 of the Cornell High Energy Synchrotron Source (CHESS), at temperatures ranging from 25-650°C and pressures up to 800 MPa. The solution samples were placed inside a volume defined by a 0.5 mm hole, of a 0.13 mm thick rhenium gasket, and the opposing diamond anvil culet faces pressing against the hole. The measurements were made in transmission mode, using conventional gas ionization chambers. The incident x-ray beam was focused using the Kirkpatrick/Baez mirrors to approximately 13 micrometer spot size. Selective tilting of the cell by 0.1-0.2 degrees relative to the x-ray beam caused the diffraction peaks resulting from the diamonds to shift in photon energy between individual spectra. In this way, spectral refinement can be accomplished by replacement of a region contaminated by a peak in one scan by an uncontaminated region from another scan.

Studies of this system were largely motivated by our earlier studies on the structure of iron(II) complexes in saline fluid inclusions in quartz found in a pegmatite dyke intruding into granulites of the Saxon Granulite Massif, Germany¹⁵¹. Results from fitting of the Fe K-edge spectra measured at different temperatures, where the pressure varied according to the equilibrium liquid-vapor coexistence, indicate that the iron(II) ion is predominantly

octahedrally hydrated from 25°C to 100°C. From 200°C to 500°C, there appears to be a stable mixture of chloro iron(II) complexes in the fluid, with the average ligand coordination number being 3.5. The results from a preliminary analysis of the XAFS spectra measured from 2m Fe(II)Cl₂/11wt% NaCl/H₂O solutions under similar pressure and temperature conditions are in qualitative agreement with our results for the fluid inclusions. This is despite the fact that the iron concentration in the fluid inclusions is considerably lower (up to several thousand ppm) than in the solutions. In addition, the Cl⁻ concentration of the liquid phase of each inclusion undergoes a gradual increase with temperature due to the progressive dissolution of its constituent halite phase (NaCl), which is typically completely dissolved at about 260°C. There is a progressive and very similar slight change with temperature, from 200°C to 500°C, in the near edge region of the spectra measured from either the inclusions or the 2m Fe(II)Cl₂/11wt% NaCl/H₂O solution. This may result from the structural symmetry of the iron chloro complexes undergoing small changes with temperature. Our XAFS experiments on zinc and iron in solutions using the HDAC, including on the 2m Fe(II)Cl₂/11wt% NaCl/H₂O system, demonstrate that this is a very useful tool which can be used to constrain the thermodynamic variables and thereby help establish a base of knowledge to more fully understand, for

Figure G-11. Fe K-edge XAFS spectrum measured from a 2m Fe(II)Cl₂/11wt% NaCl/H₂O solution at 400°C and about 150 Mpa of pressure. The large peaks occur as a result of diffraction of the incident x-ray beam from the diamond anvils of the cell.

Figure G-12. The magnitude of the Fourier transforms (FT) of the extended XAFS data (blue line), isolated from the spectrum shown in Figure G-11, and of the fit (red line) of this data, made using FEFF6.

example, reaction kinetics in fluid-rock interactions. When used in conjunction with similar experiments carried out on naturally occurring fluid inclusions, the knowledge of speciation and structure of metal complexes

at elevated temperatures and pressures gained from such experiments should prove to be very useful for increasing our understanding of the transport and deposition of elements under ore-forming, hydrothermal conditions. ■

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MATERIALS SCIENCES

Quantifying Magnetic Domain Correlations in Multilayer Films

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ABSTRACT

The vertical correlation of magnetic domains in a Co/Cr/Co magnetic trilayer is statistically quantified as a function of applied magnetic field. These measurements used in conjunction with determinations of the individual layer magnetometry curves identifies the presence of both interlayer anti-ferromagnetic exchange coupling and ferromagnetic dipolar coupling for different regions within the trilayer.

The spin conductance of a magnetic heterostructure is controlled by the relative orientation of the magnetic moment directions of the component layers on a local scale (within a few spin mean-free-paths)^[1]. Any meaningful comparison between the measured and calculated spin-conductances for multilayer structures requires a quantitative description of the field dependence of the magnetic domain correlations taken vertically between the layers. The correlation between magnetic domains can be statistically quantified by measuring the magnetic field dependence of the X-ray resonance magnetic scattering (XRMS)^[2-5]. XRMS is the angle dependent specular reflectance of circular polarized soft x-rays whose energy is tuned to the absorption energy of a magnetic element present in the multilayer.

The specular scattered intensity of a resonant soft x-ray is a function of the incident angle of the soft x-ray, θ , and the magnetic configuration of the multilayer (which is dependent on the applied magnetic field history). For a single film with magnetic domains large compared to the photon coherence length, the reflected intensity, $I(\theta, B)$, is given by

$$I(\theta, B) = \sum_k I_k(\theta) x_k(B) \quad (1a)$$

where k denotes a particular magnetic domain type within the film, I_k the scattered intensity from that domain, and x_k is the fraction of the film in the k^{th} domain type. (Note that $\sum_k x_k(B) = 1$ at any field value.) For a film which has only two possible magnetic domains (left and right), eq. (1a) becomes

$$I(\theta, B) = I^{\rightarrow}(\theta) x^{\rightarrow}(B) + I^{\leftarrow}(\theta) x^{\leftarrow}(B) \quad (1b)$$

Eq. (1a) can also apply to a multilayer film, but now k denotes a particular configuration of the magnetic moment orientations for each magnetic layer taken vertically along the multilayer. I_k is the scattered intensity from that moment configuration and x_k is the fraction of the multilayer in that particular configuration.^[6] Similarly extending eq. (1b) to a magnetic film system consisting of two layers, each with two possible magnetic domain directions, the total scattered intensity becomes

$$I(\theta, B) = I^{\rightarrow\rightarrow} x^{\rightarrow\rightarrow} + I^{\rightarrow\leftarrow} x^{\rightarrow\leftarrow} + I^{\leftarrow\rightarrow} x^{\leftarrow\rightarrow} + I^{\leftarrow\leftarrow} x^{\leftarrow\leftarrow} \quad (2)$$

where $x^{\rightarrow\rightarrow}$ and $x^{\leftarrow\leftarrow}$ are the fractions of the film with the magnetic domains of the top and bottom film aligned with each other (correlated) and $x^{\rightarrow\leftarrow}$ and $x^{\leftarrow\rightarrow}$ are the fractions of the film anti-aligned to each other (anti-correlated). It is these four magnetic field dependent fractions which will ultimately express the vertical correlation of magnetic domains in the trilayer.

To experimentally realize only these 4 magnetic domain configurations, a magnetic structure with a strong uniaxial magnetic anisotropy is utilized. To accomplish this, a single crystal Co (50Å)/Cr (35Å)/Co (50Å) trilayer is deposited on an epitaxially grown ZnSe(001) substrate. To stabilize the bcc Co structure, a seed layer of 5 Å of bcc Fe is first deposited on the ZnSe. The specular reflected intensity is measured using a Si photodiode

located at an angle 2θ to the incident beam direction (q to the film plane). The 75% circular polarized soft x-ray is tuned to the Co L_3 edge (778 eV), corresponding to the maximum in the Co absorption curve.

To quantify the magnetic domain configuration fractions $x_k(B)$, the applied field dependence of the scattered intensity is measured. But, as indicated in Eq. (1a), the contributions to the total scattered intensity depend separately on the applied magnetic field and on the incidence angle. This latter angular dependence is demonstrated in the top panel of **Figure M-1**, where the log of the specular scattered intensities for negative helicity light and the resulting asymmetry $(I^+ - I^-)/(I^+ + I^-)$ are shown for the sample fully magnetized by an applied field of +120 Oe (I^+) and -120 Oe (I^-). Displayed with these angle dependent reflectance curves are a subset of the field

dependent reflected intensity curves recorded at the reported incidence angles (shown in the 8 curves at the bottom of **Figure M-1**). Due to the changing contribution of the scattering intensities $I_k(\theta)$ these curves are markedly different.

The obvious variation in these curves belies a hidden similarity. In eq. (2), the dependence on the applied magnetic field is contained only in the four configuration fractions, $x_k(B)$, whereas the angular dependence is derived solely from the four prefactor terms $I^{\rightarrow}(q)$, $I^{\leftarrow}(q)$, $I^{\rightarrow}(q)$, and $I^{\leftarrow}(q)$ which each remain constant for a fixed incident angle. Therefore, the large variations observed in the field scans taken at different incident angles, results only from a variation of these four multiplicative constants.

Actually only two of these prefactors are unknown. In the top panel of **Figure M-1**, we show the

Figure M-1.
(Top Panel)
Scattered intensity
for circular
polarized soft x-
rays near the Co
 L_3 edge (778 eV)
for trilayer fully
magnetized in
either direction.
Also shown is the
normalized
asymmetry.

(Bottom Panel)
Eight magnetic
field dependent
specular intensity
spectra acquired
at the indicated
incidence angle
for circular
polarized soft x-
rays near the Co
 L_3 edge (778 eV).

experimentally determined values for the angle dependence of the scattered intensity when the film is completely magnetized to saturation in the positive [$x^{\rightarrow}=1$ and $I^{\rightarrow}(\theta) = I^{\rightarrow}(\theta)$] or negative [$x^{\leftarrow}=1$ and $I^{\leftarrow}(\theta) = I^{\leftarrow}(\theta)$] field direction. Utilizing the many field dependent scans, an iterative, least-squares best-fit procedure can be applied to determine the two remaining unknown intensity factors, which are different constants for each angle, and the four configuration fractions, $x^{\rightarrow}(B)$, $x^{\leftarrow}(B)$, $x^{\rightarrow\leftarrow}(B)$, and $x^{\leftarrow\rightarrow}(B)$, which must be the same for ALL scans. In this way, the fraction of the film with a particular magnetic domain configuration can be uniquely determined.

The validity of this procedure can be checked by comparing measured magnetometry loops with calculated magnetometry loops derived from the four extracted domain configuration fractions by noting that the moment of the bottom layer, M_{BOTTOM} , is given by

$$M_{\text{BOTTOM}} = M_0(x_1^{\rightarrow} - x_1^{\leftarrow}) \quad (3)$$

where $x_1^{\rightarrow}$ ($x_1^{\leftarrow}$) is the fraction of the bottom magnetic layer with moment along (opposed to) the applied field direction. This can be rewritten in terms of the four configuration fractions by noting that $x_1^{\rightarrow} = x^{\rightarrow} + x^{\rightarrow\leftarrow}$ and $x_1^{\leftarrow} = x^{\leftarrow} + x^{\leftarrow\rightarrow}$ (and similarly for the top magnetic layer).

In the top panel of **Figure M-2** is shown both the normalized total moment hysteresis curve (measured by vibrating sample magnetometry) as well as the normalized hysteresis curve of just the bottom layer. We have directly measured the hysteretic behavior of the bottom film by measuring the element specific hysteresis curve in absorption of the strongly coupled Fe seed layer used as a template to establish the bcc growth of Co. Superimposed over these curves are the calculated hysteresis loops as determined from the four extracted configuration fractions and eq. (3). The agreement is nearly exact, giving confidence in the extraction of the film fractions.

For spin-conductance applications, it is the relative orientation of the magnetic moments within a domain which gives rise to the resistance variation (aligned for low resistance, anti-aligned for high resistance). Therefore, the correlation function representing the fraction of the film which is aligned, minus the fraction of the film anti-aligned, is a more useful quantity to examine. This quantity is plotted in the middle panel of **Figure M-2**.

From **Figure M-2**, it is clear that the Co/Cr/Co film never reaches a fully anti-aligned configuration. The field value where the correlation is most negative corresponds to the peak in the magneto-resistance since it is at this point that the two magnetic layers are most anti-aligned and the trilayer has the highest resistance.

This correlation function can be used in conjunction with measured transport curves to extract the coefficient of magnetoresistance^[7] or for a detailed comparison with theory. It can also be used to identify and quantify the interlayer coupling mechanisms present in this multilayer.

In the absence of any type of interactions between the magnetic layers, the purely random distribution of magnetic domains will still result in a statistical probability that two vertically offset regions of different magnetic layers within the multilayer are aligned with each other. For two non-interacting independent films, the predicted fraction of the film in a particular configuration, $X_{\text{NI}}(B)$, can be calculated as simply the product of the individual domain fractions of each layer (i.e., keeping with the previous notation $x_{\text{NI}}^{\rightarrow\leftarrow} = x_1^{\leftarrow} \times x_2^{\rightarrow}$, and similarly for each of the other configurations). Since, as shown in **Figure M-2**, the individual layer fractions can be extracted from either the magnetometry data or the measured two-layer configuration fractions themselves, a correlation function for two non-interacting films can be separately constructed for comparison with the derived correlation function of **Figure M-2**. By comparing the calculated non-interacting films correlation function to the extracted correlation function, we can imply the presence of coupling between the magnetic layers and even ascertain the sign of the coupling.

This comparison is performed in the bottom panel of **Figure M-2** where is shown the bottom film hysteresis curve, the extracted correlation function, and the difference between the extracted correlation function and the calculated correlation function for non-interacting films, each for only the increasing field leg of the hysteresis loop. This difference is just the remainder of the correlation function caused by coupling between the films^[8]. As the field is increased, the extracted correlation function is reduced while the bottom film hysteresis loop shows that the bottom film is still near saturation. Since the coupling-derived remainder of the correlation function remains zero, as it must if either film is at saturation, the initial reduction in the extracted correlation function would occur regardless of the presence or absence of any interaction between the films. As the field is increased further, the coupling-derived remainder of the correlation function remains near zero until a significant number of magnetic domains have begun to form in the bottom film. At this point the coupling-derived remainder of the correlation function becomes non-zero and positive, indicating that a significant fraction of the film is interacting and that the magnetic domains are preferentially aligned to those domains directly opposite them in the adjacent layer, probably from dipolar coupling. At higher applied fields, this predominant ferromagnetic interaction is replaced by an anti-

ferromagnetic coupling indicating that the majority of the sample is now dominated by an anti-ferromagnetic interlayer coupling mechanism.

Of the various formulations for describing the magnetic domains with this trilayer system, it is the final coupling-derived remainder of the correlation function associated with interacting films which probes the fundamentally significant behavior of the magnetic films by identifying and quantifying the type and strength of the interlayer coupling mechanisms. ■

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Figure M-2.
(Top Panel) Normalized magnetometry loops for the total moment and only the bottom film (the Fe hysteresis loop). Superimposed is the calculated hysteresis loops from the least-squares best-fit extraction of the configuration fractions (see text).

(Middle Panel) The extracted correlation function (as defined in the text) for increasing (solid) and decreasing (dashed) field.

(Bottom Panel) The bottom layer hysteresis behavior (solid line), the extracted correlation function (dashed line), and the coupling-derived remainder of the correlation function (dots) of the Co/Cr/Co trilayer as a function of increasing field.

Orientational Correlations in Liquid Crystalline Phases Revealed By Polarization-Analyzed Resonant X-Ray Scattering

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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 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 (**Figure M-3**). 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 to the molecules in alternate layers being tilted in opposite directions. Since this interlayer orientational order also applies to the local spontaneous polarization direction, the electro-optic response of the phases varies,

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 convention diffraction. Moreover, the polarization state of the diffracted x-rays can differ from that of the incident x-rays. Dmitrienko calculated the tensorial atomic scattering factor for a crystal with a 4_1 helical symmetry axis^[6]. We extended Dmitrienko's analysis to the 4-layer helical structure of the $\text{SmC}_{\text{Flz}}^*$ phase and found that his results also apply; namely, for a s-polarized incident x-ray beam, a structure with a 4_1 helical symmetry axis should produce s-polarized half-order peaks and p-polarized quarter-order peaks.

The existence of quarter and half order resonant peaks for the $\text{SmC}_{\text{Flz}}^*$ phase with the 4-layer helical pitch is shown in **Figure M-4**. Taking the scattering wavevector normal to the layers to be Q_z and the wavevector associated with the layering to be $Q_0 = 2\pi/d$, the scattered intensity is plotted as a function of both Q_z/Q_0 and as a function of the energy departure of the incident x-rays from the resonant energy, E_0 . It is evident that when $(E - E_0) > 20$ eV, only the integer order $Q_z/Q_0 = 2.0$ peak remains within the range of wavevectors probed.

To measure the polarization state of the scattered x-rays, we used a pyrolytic graphite (PG) analyzing crystal. At the sulfur resonant energy, the 2θ Bragg angle for PG is close to 90° so rotating the analyzing crystal about the

axis of the x-ray beam through the angle, χ_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 M-5** illustrates this for the incident beam. The incident beam was σ -polarized and was polarized perpendicular to the PG diffraction plane when $\chi_a = \pm 90^\circ$ giving maxima in the diffracted intensity. We applied a similar polarization analysis to the resonant peaks in the $\text{SmC}_{\text{FI2}}^*$ phase (lower three plots in **Figure M-5**) by rotating the analyzer crystal about the diffracted beam. For the half order peak, the diffracted beam was σ -polarized, same as the incident beam, giving χ_a maxima at $\pm 90^\circ$. On the other hand, for the two quarter order peaks, the diffracted beam was π -polarized so the maximum in χ_a occurs at 0° as predicted by the tensor analysis for the 4_1 helical symmetry.

In summary, we showed that polarization-analyzed resonant x-ray scattering provides a valuable and unique probe of the orientational correlations that occur in liquid crystalline systems. These orientation correlations can be studied from Angstrom to micron length scales. The observed polarizations agree with a clock-model of the SmC^* phases, and unambiguously rule out other proposals made to date for these structures. ■

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Figure M-5. *Upper Plot* Intensity diffracted by the PG polarization analyzer as it was rotated through an angle, χ_a , around the incident x-ray beam. This scan was taken at an energy where the PG 2 θ Bragg angle = 90° . *Lower Three Plots* These plots, taken at the sulfur K-edge energy, show the intensity diffracted by the PG polarization analyzer as it was rotated about the resonant diffracted x-ray beam for the quarter and half order peaks in **Figure M-4**.

Surface and Bulk Electronic Structure in Wide Band Gap Nitride Semiconductors

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INTRODUCTION

Wide band gap nitride semiconductors are the focus of intense scientific scrutiny due to their numerous potential applications in optoelectronic and high temperature devices.^[1] However, our understanding of the basic physics of these materials lags far behind our ability to make simple electronic devices from them. If wide band gap nitride semiconductors are to achieve their full technological potential then we must have as deep an understanding of their *fundamental* properties as exists for Si and III-V electronic materials. We have begun a program aimed at understanding both the basic electronic structure of a variety of wide band gap nitride semiconductors, and also the electronic structure of metal and non-metal overlayers on these nitrides. We use a unique combination of photon and electron spectroscopies in this effort, and our experiments are primarily performed on NSLS beamlines U4A and X1B.

EXPERIMENTAL TECHNIQUES

Four complimentary spectroscopic probes of electronic structure in solids are utilized in this program: angle resolved photoemission spectroscopy (ARP) and soft x-ray emission spectroscopy (SXE) being the primary tools, and soft x-ray absorption spectroscopy (SXA) and inverse photoemission spectroscopy (IPS) being secondary tools. ARP is a well established technique that allows us to measure the band dispersion of both bulk and surface states in solids. However, the short mean free path of electrons in solids leads to a very high surface sensitivity for ARP, which requires that the surface of the sample under consideration be atomically clean and well ordered. In contrast, as a photon in/photon out spectroscopy, SXE is a bulk probe. SXE measures the element- and site-specific density of states resolved into its orbital angular momentum components. A renaissance in SXE is presently underway due to two factors: i) the combined use of grazing incidence diffraction gratings to disperse the x-ray emission and a wide area x-ray detector to measure the emission, that can be moved under vacuum to remain at the focal point of the gratings for all energies, and ii) the use of high intensity monochromatic

synchrotron radiation as the excitation source.^[2-4] The former allows energy resolution comparable to that of conventional photoemission to be obtained, while the latter allows the selective excitation of individual core levels in multi-element systems. An added feature of our SXE spectrometer is the ability to be used as a high energy inverse photoemission spectrometer, since the physical process leading to inverse photoemission is similar to that leading to electron-excited SXE.^[5,6] In a collaboration with Dr. Peter Johnson from BNL Physics, we also have used his dedicated IPS instrument in this program. Finally, we also have used SXA spectroscopy, which gives complimentary information concerning the empty density of states.^[7]

RESULTS

Among the highlights of our nitride research program thus far are:

- The first *full* measurement of the bulk valence band dispersion of wurtzite GaN;
- The *definitive* observation and characterization of surface states on GaN(0001);
- The commissioning of a new high resolution x-ray emission spectrometer on X1B, and its use to measure the *elementally resolved* valence band electronic structure in GaN, AlN, $\text{Al}_x\text{Ga}_{1-x}\text{N}$, and $\text{GaN}_x\text{As}_{1-x}$;
- The direct measurement of shallow core level hybridization in semiconductors;
- The measurement of *elementally resolved* band gap evolution in $\text{Al}_x\text{Ga}_{1-x}\text{N}$;
- A measurement of the dispersion of conduction band states using inverse photoemission spectroscopy.

i) *Bulk Band Dispersion in GaN*: The full bulk band dispersion of the occupied valence states in n-type wurtzite GaN was measured for the first time.^[8,9] Figure 1 shows a series of photoemission spectra where states along the ΓM direction in the bulk Brillouin zone are being probed. The rich set of spectra in Figure M-6 are representative of those measured in other directions, and indicative of the high quality of the surfaces being studied. Since ARP is a surface sensitive technique, the bulk band

Figure M-6.

Figure M-7.

structure cannot be measured *unless* the surface is atomically clean and well-ordered.^[10,11] Achieving a reproducible method of cleaning the film surfaces was a prerequisite to all our ARP experiments. The method we used was repeated cycles of N_2 sputtering followed by annealing in UHV. Bermudez recently published an exhaustive comparison of the various methods of cleaning GaN surfaces, and found this to be one of the best approaches.^[12] The spectra in **Figure M-6** and those obtained for the other high symmetry directions of the zone can be used to generate the measured bulk band structure. This is shown in **Figure M-7**, with the data points corresponding to the experimentally measured structure.^[9] Also shown in **Figure M-7** as solid lines are the results of an LDA calculation by Rubio *et al.*^[13] Aside from the non-dispersive state at the top of the valence band, the agreement between this calculation and the measurement is good, in terms of both variation in band width and band velocities. The non-dispersive state near the valence band maximum that we have observed can be conclusively identified as a surface state, as is discussed below.

ii) *Bulk Density of States in GaN, AlN and Related Alloys:* The last two years saw the commissioning of our new high resolution x-ray emission spectrometer on X1B. Our first experiments involved studying the bulk electronic structure of GaN thin films.^[8,14] **Figure M-8a** shows both the SXE spectrum for the radiative decay of the valence band into a N $1s$ hole (N K emission), and the SXA spectrum for excitation of N $1s$ electrons into the empty conduction band (N K absorption). In the SXE spectrum, the N $1s$ core state is excited by 430 eV photons. Details of how the emission and absorption spectra are placed on a common binding energy scale relative to the VBM can be found elsewhere.^[14] The N K emission spectrum reflects the valence band N $2p$ PDOS. **Figure M-8b** shows both the SXE spectrum for the radiative decay of the states near and in the valence band into a Ga $2p_{3/2}$ hole (Ga L_a emission), and the SXA spectrum for excitation of Ga $2p_{3/2}$ electrons into the empty conduction band (Ga L absorption). Here, emission from N $2p$ states is dipole forbidden, and the spectrum reflects the Ga $4s$ PDOS of the valence band, and the Ga $3d$ shallow core state. Also shown in **Figure M-8** are the results of an LDA calculation of the PDOS for wurtzite GaN.^[15] Here we have

Figure M-8.

convoluted the theoretical DOS by Lorentzians to simulate the core-hole lifetime broadening and by Gaussians to simulate the instrumental broadening. The agreement between the calculated and measured PDOS for the valence and conduction bands is very good. The ability to study nitride alloy systems without worrying about cleaning their surfaces (and maintaining stoichiometry) is a particularly notable feature of SXE. Our results from the $\text{Al}_x\text{Ga}_{1-x}\text{N}$ alloy system are presented in Figure M-9.^[16,17] Here we have been able to measure the *elementally resolved* PDOS for the entire alloy system, thus allowing us to determine the evolution of the band gap as function of elemental concentration. Figure M-9 shows the dominant N 2*p* contribution to the valence band, i.e. these spectra result from the transition of N 2*p* valence electrons into N 1*s* holes. These spectra reveal that there is no bulk band gap bowing in this alloy system.

iii) Surface Electronic Structure of GaN:

As noted earlier, there is a state visible at the top of the valence band in the experimental band structure of Figure M-7 that does not correspond to any bulk state. Using the linearly polarized nature of synchrotron radiation, we have definitively identified this state as a surface state with sp_z character. The state is destroyed by adsorption of activated H_2 , and by exposure of the surface to O_2 or by ion bombardment. These measurements are performed on n-type GaN films grown by MBE. Recently, we have found that despite identical surface cleaning of the films, the electronic structure of GaN surfaces grown by MOCVD on SiC are

significantly different. (Fortunately, the *bulk* states measured by ARP are the same between the MOCVD and MBE samples.) Figure M-10 shows the experimentally measured surface band structure for MOCVD-grown GaN surfaces cleaned in *exactly* the same manner we cleaned the MBE films. For clarity, only the surface related states are plotted in Figure M-10, but the contrast with our results for MBE films presented in Figure M-7 is dramatic. Instead of a spatially localized state (and hence non-dispersive in k -space) at the valence band maximum, we find two highly dispersive states, one of which disperses throughout almost the entire valence band. The most obvious explanation for the different results from MBE and MOCVD films whose surfaces were cleaned in the identical manner is that the surfaces

Figure M-9.

Figure M-10.

have different terminations or polarities.^[9,12,18-21] Both types of surface were atomically clean (as measured using Auger electron spectroscopy) and displayed sharp (1 x 1) low energy electron diffraction (LEED) patterns indicating identical surface symmetries. However, it is likely that the termination of the surfaces is different.

iv) **Conduction Band Dispersion in GaN:** While soft x-ray absorption can measure the PDOS of the conduction band, inverse photoemission spectroscopy is required to measure the *dispersion* of the empty states. We began a collaboration with Peter Johnson from BNL Physics to use his high resolution inverse photoemission spectrometer.^[22] Our first experiments were on MBE-grown n-type GaN. We found that there are no

unoccupied surface states.^[23] Furthermore, we found that the conduction band states differ significantly from free electron states up to 20 eV above E_F . Since the free electron final state assumption is used to generate the measured band structure (Figure M-10) from the ARP spectra, this is an important observation. However, higher photon energies were used in the band mapping, so we are satisfied the free electron final state approximation is appropriate in this case.

We have embarked on a multi-technique study of the electronic structure of wide band gap nitride semiconductors that employs heavily the facilities of the NSLS. This research has lead to significant progress in understanding both the surface and bulk electronic structure in simple nitrides, and in more complex alloys. Future research will be directed towards the electronic structure of overlayers and interfaces in such systems.

This work was performed by the Electronic Structure Group, Department of Physics, Boston University: Yu-Chang Chao, Laurent Duda, Sarnjeet Dhesi, Cristian Stagaescu, James Downes, and Philip Ryan. The inverse photoemission experiments were performed in collaboration with Tonica Valla and Peter Johnson from the Department of Physics, Brookhaven National Laboratory. Some of the SXE experiments are performed in collaboration with Jinghua Guo and Joseph Nordgren from Uppsala University. Samples are provided by Theodore Moustakas from Boston University, Robert Davis from North Carolina State University, Thorvald Anderson from Chalmers University, and Jim Edgars from Kansas State University.

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X-ray Diffraction Studies on Levitated Liquids

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Liquid structure measurements are important in advancing condensed matter theory, in developing predictive models, and in establishing structure-property-process relationships in high temperature materials sciences. For example, the differences between the structures of the liquid and crystalline phases of the same composition can influence the nucleation kinetics and solidification paths.

The major experimental difficulties encountered in obtaining structural data on liquids at temperatures above about 1000K are, (i) reactions of the specimens with container walls, and (ii) influence of the containers on the structural measurements. Recently, a number of research groups^[1-4] have attempted to overcome these problems by employing levitation techniques in conjunction with X-ray scattering to study the structural properties of liquids. These groups have employed conical nozzle levitation (CNL)^[1,2] and electromagnetic levitation techniques^[3,4] to suspend 3-6 mm diameter droplets in X-ray synchrotron sources and have conducted X-ray scattering and EXAFS measurements to obtain structural data on liquids. Several key advantages of these methods include the elimination of container interactions and container-derived impurities, rapid access to high temperatures, localized heating conditions, and lastly access to the supercooled (metastable) liquid. We have used the CNL method extensively to investigate the structure of a number of liquids^[5-10] in their normal and supercooled states in a series of experiments conducted at the NSLS. These prior studies include a description of the techniques^[1], and structure measurements on liquid Si^[5], Al₂O₃^[6], boron^[7], Y₂O₃^[8], Co₈₀Pd₂₀^[9], Ni and Zr^[10].

This article highlights a few recent results obtained on levitated liquids that were obtained with the use of conical nozzle levitation (CNL). These experiments were conducted at the X25 beamline at the National Synchrotron Light Source (NSLS) at the Brookhaven National Laboratory. The CNL apparatus consisted of a gas flow system which delivered the levitation gas (inert gases like argon) into a plenum chamber from which the gas flowed out through a divergent nozzle. Small (2-4

mm diameter) spheres of the specimen materials could be levitated stably in the nozzle. The specimens were then be laser-heated, melted and levitated for long durations of up to several hours. The complete CNL system was integrated with a Huber model 6-circle, X-ray diffractometer.

The CNL system was enclosed in an environmentally-controlled chamber with suitable ports for laser heating, pyrometry, sample injection and retrieval, and pressure measurements. The upper part of the chamber was a 9 inch diameter sphere. Two beryllium windows were included in this section; the first was a small window for the incident X-ray beam which was placed at the end of a radial tube port. The second window was curved and subtended approximately 120° around the nominal specimen position at the center of the chamber. This window extended about 5° below the normal levitation height of the specimen to enable transmission of the direct X-ray beam. The specimens were heated with the aid of a 270 watt CO₂ laser. Temperature measurements were obtained with two pyrometers whose operating wavelengths were 0.65 μm and 1-2.5 μm, respectively. Precise sample positioning was achieved with a 3-axis motorized translator assembly and a phosphor screen to observe the shadow of the specimen in the X-ray beam. Specimen materials could be levitated, heated, melted and positioned stably over durations of up to 3 hours, which were sufficient for the X-ray diffraction studies.

Scattered X-rays were detected using a solid-state detector with 300 eV resolution over a 2θ angular range of 10-110°. Diffraction data on liquids were obtained over a momentum transfer ($Q = 4\pi\sin(\theta)/\lambda$) range of 1.2 - 13 Å⁻¹. The measured intensity, $I(2\theta, E)$, for an incident X-ray energy E is given by:

$$I(2\theta, E) = I_p(E) [A_{ss}(2\theta, E) \bullet P(2\theta) \bullet I_{coh}(Q) + I_{MS}^{coh}] \\ + I_p(E') [A_{ss}(2\theta, E') \bullet I_{inc}(Q') + I_{MS}^{inc}(Q)] \quad (1)$$

where:

$I_p(E)$ = intensity of the main beam at the energy E ,
 $I_x(Q)$ = the coherent or incoherent
 scattering intensity,
 $A_{s,s}(2\theta, E)$ = attenuation coefficient,
 $I_{MS}^*(Q)$ = the coherent or incoherent
 multiple scattering,
 $P(2\theta, E)$ = polarization factor
 $E' = E + \Delta = E/(1 - 0.00392E\sin^2(\theta))$, and
 $Q' = 1.014E \sin(\theta)$, where Q is
 measured in $\text{\AA}^{-1}$ and E in keV.

This equation was used as the basis of the correction procedure which employed a Monte Carlo simulation of the experimental geometry that was then back refined against the measured data. The corrections were implemented through a two-stage refinement procedure: the first stage refined instrumental parameters, including photon density profile and detector efficiency derived from a statistical average of all the data sets obtained in the experimental run cycle, and specimen parameters including sample position in the beam, sample radius, and density. In the second stage, individual runs were analyzed using fixed instrumental and variable sample parameters. Form factors were obtained from Baro, *et al.*^[11]; the temperature dependence of the form factors was obtained from Ref. 12.

The data analysis procedure directly yields the x-ray weighted average structure factor $S(Q)$. From the $S(Q)$, the radial distribution function, $G(r)$, is given by:

$$G(r) - 1 = \frac{1}{2\pi^2\rho_a} \int_0^{Q_{\max}} Q(S(Q) - 1) \sin \frac{Qr}{r} dQ \quad (2)$$

where ρ_a is the number of atoms per unit volume. This inverse Fourier transform method employs the maximum entropy criterion^[13] where $G(r)$ is constrained to one at large r . Values of the number density, ρ_a , was either estimated or obtained from literature data.

In this article, we will present structural results (obtained at the X25 beamline) on liquid silicon^[5] and boron^[7] in the normal and supercooled liquid states. **Figure M-11** shows the X-ray weighted average structure factors $S(Q)$ for liquid Si at temperatures of 1542, 1603, and 1829K. The melting point of Si is 1688K, so that the first and second measurements correspond to the supercooled liquid state. The $S(Q)$ shows a main peak with a pronounced shoulder on the high- Q side in addition to a second peak. **Figure M-12 (Table)** lists the locations of the peaks in the $S(Q)$ for liquid Si. The significant effect of supercooling is to move the first peak and shoulder to a Q value that is about 8% greater at 1542K than at 1829K. Pair correlations functions $g(r)$ were obtained from the $S(Q)$ using a maximum entropy method^[13] and are given in **Figure M-13**. Supercooling results in a significant sharpening of the first peak and a reduction in the coordination number from 6.4 to 5.6. A systematic decrease of 2% in the position of the first peak is observed with supercooling, as given in **Figure M-12**. The stable liquid shows a shoulder on the high- r side of the first peak, which splits into a double shoulder upon supercooling. The present results for liquid Si above the melting point were found to be in good agreement with the prior measurements of Waseda, *et al.*^[14] and in fair agreement with the *ab initio* MD simulations of Stich, *et al.*^[15]. The decrease in the first peak maximum and the first shell coordination with supercooling is consistent with the MD simulations of Angell and Borick^[16]. Further discussion of the structural measurements and a discussion of results obtained on liquid Si results can be found in a recent publication^[5].

Figure M-11. Total structure factor, $S(Q)$ for liquid Si at three specimen temperatures of 1542, 1603, and 1829K. The melting point of Si is 1688K. The $S(Q)$ curves have been shifted for clarity.

Figure M-12 (Table). Structural results for liquid Si and B; $S(Q)$, $g(r)$, and coordination (C_n)

		$S(Q)$			$g(r)$				
Temp	Q_1	hump	Q_2	r_1	sub1	sub2	r_2	C_n	
(K)	$\text{\AA}^{-1}$	$\text{\AA}^{-1}$	$\text{\AA}^{-1}$	$\text{\AA}$	$\text{\AA}$	$\text{\AA}$	$\text{\AA}$	atoms	
	± 0.03		± 0.03	± 0.03	± 0.03		± 0.05	± 0.5	
Silicon (m.p 1688K)									
Si	1542	2.65	3.65	5.65	2.41	3.28	3.98	5.22	5.6
Si	1603	2.70	3.60	5.70	2.42	3.27	3.95	5.22	5.6
Si	1829	2.45	3.55	5.45	2.46	3.45		5.20	6.4
Si [15]	1800	2.63	3.55	5.68	2.46	3.64		5.64	6.2
Boron (m.p. 2360K)									
B	2600	2.50		4.40	1.76			3.15	5.8
B	2400	2.50		4.45	1.78			3.16	5.8
B	2090	2.45		4.35	1.78			3.15	6.2
B [17]					1.71			3.18	6.0

The crystalline forms of boron embody some of the most complex crystal structures for a pure element. The α -rhombohedral, β -rhombohedral and tetragonal crystalline forms have a very large number of atoms per unit cell, and are dominated by the B_{12} icosahedron structural unit. Based on surface tension, entropy of melting and stability data, it was speculated that these icosahedrons may survive upon melting. Because of the very high reactivity of boron at high temperature, the liquid phase had never been studied prior to the present work which employed levitation to access the liquid phase. **Figure M-14** shows the pair correlation function, $g(r)$, for liquid boron at temperatures of 2600, 2400, and 2090K^[7]. The melting point of boron is 2360K. The positions of the peaks in the $g(r)$ are not significantly affected by temperature, although there is a sharpening of the second and third peaks upon supercooling and an increase in the first shell coordination (**Figure M-12**). The liquid $g(r)$ compares well with those predicted in a recent *ab initio* MD study^[17]. The liquid $g(r)$ is also similar to the $g(r)$ for the different crystalline forms, but the

absence of a low Q peak near $1 \text{ \AA}^{-1}$ in the $S(Q)$ [see ref. 7] does not provide conclusive proof of the existence of B_{12} icosahedral units. Nevertheless, the first direct measurement of the structure of liquid B shows that the short-range structure is remarkably similar to the crystalline and amorphous solid forms, with little change in bond lengths but a broadening of the first coordination shell, preserving the coordination number around a value of 6. The present study shows that boron is unique among covalently-bonded solids in undergoing an insulator to metal transition on melting with small volume expansion and little change in the short-range structure. ■

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Figure M-13. Radial distribution function, $g(r)$ for liquid Si at three specimen temperatures of 1542, 1603, and 1829K computed using the maximum-entropy Fourier transform method. The melting point of Si is 1688K. The $g(r)$ curves have been shifted for clarity.

Figure M-14. Radial distribution function, $g(r)$ for liquid B at three specimen temperatures of 2600, 2400, and 2090K computed using the maximum-entropy Fourier transform method. The melting point of B is 2360K. The $g(r)$ curves have been shifted for clarity.

Direct Observation of Charge and Orbital Ordering in Manganites

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Understanding the origins of high- T_c superconductivity and colossal magnetoresistance are among the most intensely studied issues of physics research today. Intriguingly, both phenomena appear in perovskite-type transition metal oxides, which share the common characteristic that the electrons are neither localized nor completely delocalized, but behave in an intermediate fashion. These materials are known as strongly correlated electron systems and it is believed that their unusual properties result from these correlations.

The doped manganites are one class of materials where such behavior can be investigated. The manganites are fundamentally interesting because the four main characteristics of strongly correlated systems, the spin, charge, orbital and lattice degrees of freedom, are all

present. In addition, members of this class exhibit colossal magnetoresistance phenomena, in which the measured magnetoresistance can change by 6-8 orders of magnitude in an applied magnetic field. This has attracted considerable attention because of possible applications in the magnetic recording industry and elsewhere.

Here, we briefly report our results on $\text{Pr}_{1-x}\text{Ca}_x\text{MnO}_3$ for $x=0.4$, and illustrate the use of resonant x-ray scattering techniques to probe the orbital and charge degrees of freedom directly. This is a new development in the field which holds enormous promise for helping to understand the unusual behavior exhibited by manganites. For $\text{Pr}_{0.6}\text{Ca}_{0.4}\text{MnO}_3$ the spin ordering occurs at 170 K, while the charge and orbital ordering occurs at a significantly higher temperature of about 240 K. In these experiments, we have been able to observe critical fluctuations of both the orbital and charge order at temperatures above the phase transition. We have discovered that, while the two order parameters have the same temperature dependence below the phase transition, they exhibit very different behaviors above. Specifically, the charge order fluctuations have a longer length scale than the orbital order fluctuations, for a given temperature. This in turn suggests that it is the charge order that is driving this phase transition, which is an important insight.

The charge ordering in these systems manifests itself in an alternating pattern of $\text{Mn}^{3+}/\text{Mn}^{4+}$ valences as shown in **Figure M-15**. Associated with this ordering pattern is a lattice distortion which has been studied in detail in related systems by neutron and electron diffraction^[1]. Direct information concerned with the valence of the Mn-atoms is gained by resonant X-ray diffraction via the anomalous dispersion of the Mn^{3+} and Mn^{4+} scattering factors. The slight difference in the Mn^{3+} and Mn^{4+} absorption energies leads to an anomaly in the scattered peak intensity at the charge order superlattice peak when the incident photon energy is tuned to the manganese K absorption edge^[2].

Orbital ordering in these systems refers to the long ranged orientation of the occupied Mn 3d orbitals in a periodic pattern. In a first approximation, the electronic configuration of Mn^{3+} in this compound is (t_{2g}^3, e_g^1) . The t_{2g} -electrons are localized, while the e_g -electrons are more

delocalized, and they occupy either $(3z^2-r^2)$ or (x^2-y^2) -type orbitals. It is the e_g orbitals, present only on the Mn^{3+} ions, which order periodically (Figure M-15). Very recently, it has been demonstrated that it is possible to probe this ordering of the orbitals by means of resonant X-ray scattering^[2,3]. The asphericity of the atomic electron density leads to the observation of forbidden reflections and a dramatic enhancement of the scattered intensity results when the incident photon energy is tuned to the Mn-resonance energy. Two characteristics of the orbital ordering superstructure reflection are that the polarization of the incident beam is rotated from σ to π and that the intensity depends on the azimuthal angle according to $I(\phi) \propto \sin^2(\phi)$. This latter behavior is in distinct contrast to the azimuthal dependence of a regular Bragg peak, which is independent of ϕ . These features of the scattering allow one to identify peaks arising from orbital order unambiguously^[2,3].

One of the most fascinating issues to be probed concerns the interaction between the charge and orbital ordering. In particular, it is not clear whether the two sublattices order independently, or if one drives the other. We have investigated the critical temperature range of the charge and orbitally ordered regime by means of resonant x-ray scattering, in order to gain insight into this question.

The experiments were performed at beamline X22C at the National Synchrotron Light Source. The beamline is equipped with a focusing toroidal mirror and a Ge(111) double monochromator with an energy resolution of about 5 eV. The polarization of the scattered beam was analyzed with a Cu(220) analyzer crystal. A single crystal of $Pr_{0.6}Ca_{0.4}MnO_3$ was cooled with a standard displex refrigerator.

Because the charge and orbital order have different periodicity, they give rise to superlattice peaks at different positions in reciprocal space, and the two types of order may be studied independently. The charge ordering was measured in a σ - σ scattering geometry at the $(0\ 3\ 0)$ superstructure reflection and the orbital order at the $(0\ 2.5\ 0)$ superstructure reflection in a σ - π geometry. In this notation the fundamental Bragg reflections occur at $(0\ 2n\ 0)$, where n is an integer. Figure M-16 shows the peak intensities and full width at half maximum of the charge and orbital ordering plotted versus temperature. The intensities show a sharp drop at $T=240$ K for both the charge- and the orbital ordering peaks, consistent with a first order transition. A few degrees above the transition temperature, fluctuations are observed at both the charge and orbital order positions. Remarkably, as seen from the peak width, shown in the bottom part of Figure M-16, the inverse correlation length for the orbitally ordered phase increases significantly faster with increasing

Figure M-16. Comparison of the peak intensity (top) and the peak width (bottom) of the charge peak $(0\ 3\ 0)$ (blue circles) measured in a σ - σ geometry and the orbital order peak $(0\ 2.5\ 0)$ (red squares) measured in a σ - π geometry.

temperature than that of the charge order. This behavior indicates that the charge order is better established at a given temperature than the orbital order, suggesting that a charge ordered phase is a prerequisite for orbital ordering. In particular, these results imply that it is the charge order which drives the phase transition. While more detailed investigations are still required, these results are extremely interesting and beautifully demonstrate the power of this new technique for the study of charge and orbital degrees of freedom, opening a new window on these intriguing and complex systems. ■

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NUCLEAR SCIENCE

The LEGS Collaboration - X5

The Laser Electron Gamma Source facility (LEGS) provides intense, polarized, monochromatic γ -ray beams by Compton backscattering laser light from relativistic electrons circulating in the X-Ray storage ring of the National Synchrotron Light Source at Brookhaven National Laboratory. Such a beam has a high degree of polarization (typically ~90%) with very low background and the energies of the photons are well determined by measuring the loss of energy of the struck electrons ($\pm 1\%$). Photon energies up to 333 MeV can be obtained with the present laser shining on 2.58 GeV electrons. With a new frequency-quadrupled laser that is now installed and 2.8 GeV stored electrons, photon energies up to 470 MeV have been obtained.

The LEGS facility and collaboration continue to be in a time of transition with activity on several fronts. The final paper describing the Compton and pion photoproduction experiments L7 and L8 is nearly complete. In addition, the first experimental determination of δ_π , the backward spin-polarizability, was extracted from this data and published last year. Analysis continues of the first data taken with the SASY (Spin ASYmmetry) detector array, experiment L10. In all instances, we have found good agreement between the experimental data and Monte Carlo simulation. Over the last year, the laser hutch has undergone a major renovation, and preparations are nearing completion for the first measurement of the GDH sum rule. Development work on the SPHICE (Strongly Polarized Hydrogen ICE) target is going quite well. The group is preparing to provide the first target as soon as the in-beam cryostat arrives from France and is installed. This should happen spring 1999. A multipole analysis of our L1 and L3 photodisintegration of deuterium data published this year, suggests that the inclusion of meson exchange currents in current models may be incomplete.

COMPTON SCATTERING FROM THE PROTON: L7 / L8

The first phase of LEGS culminated in a pair of experiments, L7 and L8, designed to measure the electric quadrupole component in the $N \rightarrow \Delta$ transition (EMR).

Concurrent measurements were made of the $p(\vec{\gamma}, \pi$ and $p(\vec{\gamma}, \gamma')$ channels. These data formed the basis for the first simultaneous multipole analysis of both Compton scattering and π -production. The use of the two new constraints from Compton scattering resulted in sizeable alterations to several multipoles and has provided a new high precision determination of the EMR, $-3.0\% \pm 0.3$ (stat+sys) ± 0.2 (model)^[1].

This simultaneous multipole analysis also led to the first experimental determination of the *backward spin-polarizability*, δ_π . The proton's lowest order scattering response is fixed by its static properties of mass, charge, magnetic moment and spin. The leading corrections to this *point* scattering arise from the dynamic rearrangement of constituent charges and spins within the proton, and are expressed in terms of *six polarizabilities*. Four of these are the result of the interaction of the photon with the constituent spins and thus are sensitive to the proton's spin structure. The first determination of a particular linear combination of these *spin polarizabilities* that characterizes backward scattering was extracted from the analysis of LEGS Compton scattering data. The value of $\delta_\pi = 27.7 \pm 2.3$ (stat+sys) $+2.8 / -2.4$ (model) is significantly different from current theoretical estimates, and indicates an appreciable contribution from the proton's spin-structure. Both of these results have gone to press in the last year^[2], and the final paper describing the full details of the pair of experiments will be submitted soon.

COMPTON SCATTERING FROM THE NEUTRON: L10

A measurement of Compton scattering and π^0 -production from quasi-free neutrons in a liquid deuterium target ran through July of 1997. The Compton data will provide a new extraction of the neutron polarizabilities. This requires accurate knowledge of the neutron photopion multipoles below 2π -threshold (309 MeV). Prior to this measurement, there was no π^0 -production data at all below 300 MeV.

This experiment was the first to use the SASY detector array. This large solid-angle calorimeter is

designed for efficient detection of neutral particles (γ , n , π^0). In the first phase, three major calorimetry subsystems were implemented. The XTAL Box, consisting of 396 optically isolated NaI crystals, covers the full azimuthal range for photons in the range $45^\circ < \theta < 135^\circ$, providing reasonable energy and position resolution. At forward angles, a 30 cm thick wall of plastic scintillators provides detection of the forward-going hadrons. The wall is roughly 40% efficient for the detection of neutrons. This plastic wall is backed by a 10 inch thick array of 176 lead glass erenkov detectors, providing forward angle coverage for photons from π^0 decay.

The analysis of this data is in progress. We find good agreement between the experimental data and results from Monte Carlo simulation of the array. Most notably, the experimental reconstruction of the π^0 invariant agrees quite well with Monte Carlo results for both the case of two photons in the XTAL Box and the case of one photon in the XTAL Box and one in the lead glass array. The separation of Compton and π^0 events is consistent with the separation achievable in Monte Carlo.

GDH AND FORWARD SPIN-POLARIZABILITY

The LEGS SPIN Collaboration (LSC) is preparing a series of double-polarization experiments (beam and target) designed to study the helicity structure of the nucleon. Among other things, we will evaluate large portions of the spin-polarizability and GDH (Gerasimov, Drell, Hearn) sum rules. This work requires the development of a new polarized HD ice target (SPHICE), an upgrade of the laser system to increase the energy range of LEGS, and additional detector components to be integrated with SASY.

SPHICE

There have been three major target production runs with the BNL dilution refrigerator and 17 Tesla magnet system presently stationed in Syracuse. Full size (3 cm dia. x 5 cm long) HD targets with over 50% H polarization have been produced and successfully extracted from the dilution refrigerator. After the first round of experiments has been completed, the SPHICE target factory (the dilution refrigerator and magnet system and its ancillary equipment) will be moved from Syracuse to BNL and recommissioned in the new lab adjacent to the LEGS beam line. This required some building modifications to safely accommodate all of the equipment.

The dilution refrigerator-and-superconducting magnet system has been designed with the potential of manufacturing three targets simultaneously, with a ~10% increase in elapsed time. Work will focus on developing

this capability and on optimizing deuteron polarization.

As many as four sets of three SPHICE targets could be produced annually, if sufficient cryogens are available. To facilitate this a helium liquefier has been obtained from the University of Virginia. The use of this liquefier, together with a recovery system for helium gas, will reduce cryogen costs in subsequent years by a factor of five.

There are four main advantages to the new HD targets that can potentially lead to significant enhancements to several key experiments:

1. Conventionally dynamically-polarized ammonia or butynol targets are diluted with significant quantities of other unpolarized or even worse, partially polarized background nuclei. Except for 5% aluminum added for its thermal properties, the HD targets are undiluted. The figure of merit of a polarized target is defined as the length of time required to attain a desired level of statistical accuracy. This varies with the square of the target dilution. SPHICE targets are at least a factor of three better than conventional proton targets and more that a factor of 30 better for neutrons.
2. The small dilutions have an added consequence. Because the backgrounds are small (or nonexistent) it is possible to make absolute cross section measurements. There are essentially no data from direct measurements of absolute cross sections made with conventional polarized targets.
3. Polarized neutron and proton measurements can be carried out simultaneously, which is significant for the control of systematic uncertainties in cases where proton-neutron comparisons are important.
4. The HD targets have true *frozen-spin*. They are not dynamically polarized during the experiment. This fact, together with spin-relaxation times on the order of months, permits the target manufacturing facility to be separated from the experimental hall by any arbitrary distance (e.g. Syracuse to BNL).

THE NEW LASER

The laser upgrade has been completed. The new frequency-quadrupled Neodymium-Yttrium-Lithium-Fluoride (Nd-YLF) ring-laser system has been installed side by side with the Argon-Ion laser. This new laser, in combination with a recent increase of the electron storage ring energy to 2.8 GeV, increases the maximum photon energy at LEGS to 470 MeV. The first 470 MeV beam was taken in the experimental hall April of 1998.

SASY

Early measurements of the GDH sum rule will be performed using an aerogel Cerenkov veto surrounding

the target. This will allow rejection of electron backgrounds online and permit a measurement of the total reaction cross section before we have the tracking and complete separation of channels. This aerogel veto is under construction and will soon be in place for the first double-polarization experiments.

THE TIME PROJECTION CHAMBER (TPC) FOR SASY

Work has begun on the construction of a central drift chamber to expand SASY's capabilities for charged particles (p , π^+ , π^-). Final design and engineering work has begun, and construction of several components is taking place at institutions in the LEGS Spin Collaboration (LSC). Construction of the gas handling and monitoring system is almost complete. A prototype of the TPC trigger scintillator has been constructed and successfully tested. The design has been completed for the superconducting solenoid that will house the TPC within the SASY calorimeter and potential manufacturers have been contacted. The magnet and cryostat will be purchased in FY 1999 by collaborators with funds from the National Science Foundation (NSF), and the steel yoke will be constructed at BNL. A nitrogen laser system to calibrate the TPC has been purchased and is being tested by collaborators.

DEUTERIUM PHOTODISINTEGRATION MULTIPOLE ANALYSIS

A full microscopic model for $D(\gamma p)n$ requires a unitary coupled-channel treatment of the NN and NA interactions, and several sophisticated calculations have been developed in recent years. Characteristic of these calculations is a discrepancy in the cross section that grows with energy above about 250 MeV creating a "dip" in the predicted 90° cross section that is absent from the data. A detailed comparison of the calculations of Wilhelm and Arenhövel with the LEGS data at 300 MeV shows that while the predicted cross section with photon polarization parallel to the reaction plane, $\sigma_{||}$, is in reasonable agreement with the data, the cross section in perpendicular polarization kinematics, $\sigma_{\perp}$, is predicted to be too high at forward and backward angles and too low at 90° .^[3]

An experimental multipole decomposition of this reaction would be an extremely useful tool in analyzing the successes and shortcomings of theoretical calculations. This has not previously been attempted, mainly because of the ambiguities that result from combining measurements from different experiments with different systematic uncertainties. This situation has been changed by the recent availability of a precision data set from LEGS

which reports both cross sections and beam asymmetries covering the energy region of the Δ resonance.

We have made the first multipole decomposition of the $D(\gamma p)n$ reaction^[4]. We have included electric and magnetic dipole and quadrupole photons in the initial state and all possible relative p-n angular momenta in the final state. This produces a multipole expansion containing 22 complex amplitudes. Since the data lie mainly below the 2π threshold where the inelasticities in most partial waves are small, we invoke Watson's theorem to specify the phases. According to this theorem, the multipole phases in this energy region should equal those found in np scattering. This leaves 22 magnitudes to determine from the data. To ensure that the fitted multipoles vary smoothly with energy, the energy dependence for each multipole is assumed to vary, at most, quadratically.

Since the fit is underdetermined with only two out of a total of 22 observables available, the result is necessarily model dependent. Nevertheless, a physically meaningful fit was produced with 8 multipoles determined by 11 parameters. A measure of the overall quality of the fit is seen by examining the difference between the data and the fit for the 204 sum and difference cross section data points. The standard deviation for this distribution is $0.20 \mu\text{b/sr}$. This is quite comparable to the average statistical experimental uncertainty of $0.13 \mu\text{b/sr}$. Using the complete covariance matrix to compute the uncertainties on the fit, the angle integrated total cross sections are determined to about $\pm 0.3\%$ (statistical).

Solutions of equivalent quality can be obtained with diverse parameter sets and we have examined the multipole decompositions of a wide variety. In all cases, the parallel cross section is dominated by electric multipoles and the forward/backward asymmetry that characterizes the angular dependence of the $\sigma_{||}$ data is produced by $E2 \bullet E1$ interference. In contrast, the perpendicular cross sections are dominated by magnetic multipoles, and a spurious 90° "dip" in the $\sigma_{\perp}$ predictions that characterize all recent coupled-channels calculations can be reproduced by changing the M2 strength distribution in our fit.

While the Wilhelm and Arenhövel calculations are in good agreement with the electric-multipole-dominated $\sigma_{||}$ data, they predict a shape for the magnetic-dominated $\sigma_{\perp}$ distribution that is clearly inconsistent with the measurements. We speculate that this rather unusual situation could be caused by an incomplete treatment of MEC which affects primarily the magnetic transitions where the hadronic currents are important. This has been found to be the case in recent coupled-channel calculations including pion retardation effects^[5]. It is

clearly desirable to further reduce the ambiguities in the multipole decomposition. For this, other polarization observables are required, and many are expected to be sensitive to nuclear potentials and currents. The beam-target double-polarization experiments using SPHICE and SASY should change this situation dramatically. ■

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Soft X-Ray Speckle and Surface Morphology

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When coherent light scatters from a random surface, the intensity of the scattered light can exhibit a pronounced random interference effect known as speckle. The speckle pattern contains information on the particular configuration of the surface structure. This is in contrast to the scattering of incoherent light which measures statistical averages of the surface structure only. The time dependence of the speckle is the basis for the technique known as dynamic light scattering, which can provide information on surface diffusion and other dynamical effects in equilibrium systems. The spatial resolution for the structural information is limited by the wavelength of the light: shorter wavelengths improve resolution.

In the visible photon energy range, coherent light scattering has been successfully used for some time to study the dynamical properties of liquids and surface adsorbed species^[1]. Some experiments have been carried out using hard X-rays in transmission^[2] or reflection^[3-5]. With measurements at the NSLS X1B beamline, we have recently demonstrated the feasibility of speckle experiments using soft x-rays^[6]. In contrast to hard x-rays, the soft x-ray range has the advantage that the reflection coefficients of the samples are much higher. As a result, a wider range of scattering geometries is accessible while surface sensitivity is still retained.

Here, we report on the measurement of soft x-ray

Figure T-1. AFM image of InP islands on InP. The height scale is in μm . Only an area corresponding to the footprint of the synchrotron radiation in the speckle experiment is shown. Top: Original Image. Bottom: One island has been erased (arrows) in order to simulate the effects of a change in the surface morphology on the speckle.

speckle patterns from InP islands grown on InP with an InAs wetting layer. Numerical simulations of the partially coherent scattering have been carried out according to Huygens-Fresnel scattering theory in the Fraunhofer approximation, in order to understand the observed speckle patterns. The experiments were carried out at the X1B undulator beamline. To increase the lateral coherence length of the incident X-ray beam, the synchrotron radiation was apertured using a 20 μm pinhole. To ensure a sharp illuminated area on the sample, the downstream pinhole to sample distance was kept small (3 cm). The angle of incidence of the synchrotron beam on the sample can be varied, here we present data for 7.4° angle of incidence using 266 eV radiation. A movable position sensitive soft x-ray detector was placed typically 54 cm away from the sample. The detector is a microchannel plate stack with a 2D spatially resolving resistive anode readout. The active area is composed of 1024 x 1024 pixels, which are 40 μm x 20 μm in size.

The sample investigated consists of InP islands grown on InP by solid source molecular beam epitaxy. The surface morphology was investigated by RHEED and laser light scattering during growth and ex situ by atomic force microscopy (AFM). An AFM image of the InP islands is presented in Figure T-1. The image has been clipped with an elliptical pupil function in order to show the footprint of the incident soft x-rays on the sample. The surface morphology as measured by AFM has been used to simulate the speckle pattern. Details of the calculations are reported elsewhere^[7]. Intuitively, the calculation can be envisioned in terms of summing the complex phase factors arising from the path difference between each point on the sample and the considered pixel on the detector. The finite lateral coherence length was determined by the simulation of measured Fraunhofer diffraction rings and taken into account in the speckle simulations. At the present stage, calculations were carried out assuming infinite longitudinal coherence, although we expect the longitudinal coherence length to be

$$L_{\text{long}} = \frac{\lambda^2}{\Delta\lambda} \approx 50\mu\text{m}$$

The measured speckle pattern of the InP sample using 266 eV photons along with the simulations are presented in Figure T-2. As indicated by the momentum transfer scales, the images were recorded/calculated symmetrically around the specular beam. For comparison, all three results are plotted on the same logarithmic colorscale, which is set by the dynamic range of the measured data. In order to study the effects of the beam coherence, the simulation in the middle panel includes the finite lateral coherence, while the simulation in the top panel assumes infinite coherence lengths.

Figure T-2. 2D Speckle images from the sample shown in Figure 1 (top). The glancing angle of incidence is 7.4°, the photon energy is 266 eV. The intensity is represented on a logarithmic colormap. From top to bottom: simulation assuming perfect coherence, simulation including a lateral coherence length of 20 μm , experimental data.

The simulations reproduce the main features in the experimental data. The overall elongated shape of the intensity and the size of the speckles agree with the experiment. Of course, quantitative agreement of the speckle structure cannot be expected, as the AFM and speckle experiments are likely to probe different sample areas. As expected, the simulations illustrate that a reduction in the coherence of the synchrotron radiation results in a loss of contrast. With regard to the dynamic range, the agreement between experimental data and the simulation including the lateral coherence is good, but not perfect. We ascribe this discrepancy mainly to the influence of the longitudinal coherence, which is of the order of the maximum path difference between light beams scattered from either end of the illuminated region on the sample.

The distribution of the simulated x-ray scattering is tilted with respect to the frame of the image. This is caused by the orientation of the InP islands on the sample. In Figure T-1 (top), the islands are elongated and are aligned with the [100] crystal direction on the substrate. The orientation of the islands creates the preferential scattering responsible for the tilt in the image. We observe a tilt in the same direction but with a smaller slope in the experimental data (especially when analyzing images at larger Q_x (not shown)), which indicates that the alignment of the sample crystal axes was slightly off with respect to the plane of incidence.

The effect of a change in the surface morphology on the speckle pattern was evaluated by erasing one InP island from the AFM image used to simulate the scattering. The missing island is indicated by an arrow on both images in Figure T-1. The scattering simulation was computed with the modified AFM image and the resulting speckle pattern was subtracted from the original simulation. The absolute value of the difference image is shown in Figure T-3 using the same logarithmic gray scale as in Figure T-2. There is a clear difference between

Figure T-3. Absolute value of the difference in the partially coherent speckle simulations for the two different surfaces shown in Figure T-1 top/bottom.

the two simulated speckle patterns, indicating that the soft x-ray speckle experiment is promising for the study of system dynamics. There are about 20 islands in the original AFM image which are responsible for the scattering. The removal of one island should therefore decrease the scattering by about 5% which is consistent with the difference image in Figure T-3: at the specular position, the intensity difference is about 1.3 orders of magnitude (i.e. a factor of 20) smaller than the intensity of the scattering. With a fast detector, which measures the intensity at one point only, studies of the dynamics at surfaces or buried structures will become possible. ■

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A New Method for Examining the Dynamics of Macromolecules: Time-resolved Synchrotron X-ray "Footprinting"

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A new method of determining structure is under development at the National Synchrotron Light Source (NSLS) called synchrotron x-ray footprinting. The goal is to probe the conformational dynamics and tertiary structure changes of proteins and nucleic acids with millisecond time resolution. This activity is being undertaken by the Albert Einstein College of Medicine and funded by NIH as a Biotechnology Resource Center. The technique of synchrotron x-ray footprinting is a melding of stopped-flow mixing technology and radiolytic chemistry using a high flux x-ray source. Radiolysis generates $\bullet\text{OH}$ according to equation 1:

As photons interact with water and generate a water ion and an electron. The ionized water molecules react with water to produce $\bullet\text{OH}$ according to the reaction outlined in eq. (1). The kinetics and dose of $\bullet\text{OH}$ and its use in footprinting studies has been extensively reviewed^[1,2].

The prediction underlying synchrotron x-ray footprinting is that irradiation of solutions by high flux "white light" x-ray beams at National Synchrotron Light Source (NSLS) beamline X9A yields sufficient concentrations of $\bullet\text{OH}$ so that footprinting studies of nucleic acids and proteins can be conducted with millisecond exposures to the x-ray beam. Figure T-4 shows the flux emanating from a bending magnet port (impinging on a sample typical for a synchrotron footprinting experiment) at the NSLS when the ring

Figure T-4. Photon flux intercepted by a 1-mm pathlength sample of ~10 microliters (3-4 mm diameter) at beamline X9A, National Synchrotron Light Source. Beam energy of 2.54 GeV, 250 mA beam current. The figure indicates the front-end flux available for the specified geometry, the flux absorbed by the beamline optics incident on a typical sample, the flux passing through and absorbed by the sample.

Figure T-5: Secondary structure of the *Tetrahymena* ribozyme and rate constants for folding of 22 separate regions of the ribozyme that were determined by x-ray footprinting. Regions that fold on similar timescales are highlighted in the same color. P5c, the first region to fold, is highlighted in green. Immediately after, 7 regions within the P4-P6 domain fold simultaneously (orange). Subsequently, P2 and P9 make contacts (purple) with the pre-formed P4-P6 scaffold. After several minutes, protections appear in the P3 and P7 domains indicating formation of the native, active structure (yellow).

energy is 2.54 GeV, as a function of energy. Taking into account losses due to the current beamline X9A optics, which consist of two Be windows (254 μ and 500 μ) and an Al window (200 μ), gives the flux incident on a typical 10 microliter sample as a function of energy. We can also calculate the transmitted flux subsequent to the 1-mm water path, the difference between the two curves is of course the absorbed dose. Note that total absorbance is small, thus the dose is homogeneously delivered through the 1-mm path.

Our first "proof of principle" of the method consisted of an analysis of the folding of an RNA catalyst, the *Tetrahymena* ribozyme^[3]. During the folding of both proteins and RNA, dramatic changes in solvent accessibility occur. The conformation changes of RNAs in fact, can limit the speed of chemical reactions carried out by RNA catalysts^[3]. As a result, the process by which

RNA molecules fold into their native conformation has received much attention. Early investigations into the folding of tRNA established approximate timescales for the formation of secondary (10^{-4} - 10^{-5} s) and tertiary interactions (10^{-2} - 10^{-1} s), with reorganization of incorrect secondary structure occurring more slowly (0.1 - 1 s)^[4]. Recent work has shown that folding of large RNAs is more complex, involving multiple pathways^[5,6]. Individual domains of an RNA may form at rates that differ by orders of magnitude, with some transitions requiring minutes to reach completion. Our synchrotron footprinting method, with the ability to monitor changes in the solvent accessibility throughout the RNA and monitor processes occurring from milliseconds to minutes, is ideally suited to investigate the complex folding of large RNAs.

The 385 nt. ribozyme derived from the *Tetrahymena* group I intron (Figure T-5) folds into a well-defined

tertiary structure in the presence of Mg^{2+} , which is required for activity^[4]. Biochemical and genetic methods have identified at least three domains of tertiary structure that, when separated, can re-associate to form the active ribozyme. A 160 nt domain containing paired regions P4-P6 folds independently^[7,8], and its structure has been determined by x-ray crystallography^[7]. The Mg^{2+} -dependent folding kinetics of the ribozyme were quantitated by determining the changes in solvent accessibility of individual sites as a function of time after the addition of magnesium ions. A powerful advantage of the footprinting method is that changes in protection in sites throughout the ribozyme can be monitored in a single experiment. Figure T-5 summarizes the kinetic determinations of folding for 22 separate regions of the ribozyme that show significant changes in protection upon addition of Mg^{2+} . The rapidly protected sites corresponded to nucleotides that are excluded from solvent by folding of the P4-P6 domain upon itself; the tertiary interactions within the domain are established in a concerted manner at a rate of about 1 s^{-1} at $42\text{ }^{\circ}\text{C}$ (colored in orange). A subset of nucleotides in P5c (Figure T-5) were protected about twice as rapidly as other regions in the P4-P6 domain (colored in green). This region is a Mg^{2+} -rich subdomain suggesting that formation of a "metal ion core"

in P5a-P5c could serve as a nucleation site for additional tertiary structure formation. Other regions highlighted in Figure T-5 show that tertiary contacts with the other domains of the ribozyme occur more slowly (colored in purple).

These synchrotron footprinting results yield a complete kinetic 'map' for the folding of the ribozyme allowing detailed questions about the folding mechanism to be answered for this and other large RNAs. The methods used for analysis of RNA structure are immediately translatable into examining the evolving protections that occur upon binding of proteins to both DNA and RNA, and experiments to probe such interactions in real time are underway. The footprinting of proteins, to examine the time-resolved changes in protection of a protein molecule, either in a folding reaction or in macromolecular assembly processes, is also being addressed through synchrotron applications. Although detailed discussion of the technical challenges of these applications are beyond the scope of this article, the modification of proteins by hydroxyl radical occurs on timescales equivalent to those of nucleic acid cleavage, use of advanced analytical techniques, like mass spectrometry can monitor these modifications and provide new probes of macromolecular structure^[9,10]. ■

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High-Resolution X-Ray Fluorescence Spectroscopy at X25: Where are the Electrons? Who are the Ligands?

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A newly designed multi-crystal X-ray spectrometer is described and applications in the field of X-ray fluorescence spectroscopy are discussed. The instrument is based on 8 spherically curved crystals aligned in the Rowland geometry (see schematic setup in Figure T-6). Each crystal has three degrees of freedom for adjustment, Bragg angle, azimuthal angle and distance to the source and detector. The design is very flexible and crystals of different size and bending radius can be employed. If all crystals have the same distance to the source scans can be facilitated by moving the motorized spectrometer table to change all Bragg angles simultaneously.

Currently eight 8.9 cm diameter Si (4,4,0) crystals bent to 86 cm radius are employed. At Bragg angles close to backscattering a solid angle of $1/180$ of the total 4π sr can be captured with sub eV energy resolution. To avoid intensity losses due to absorption on the long paths between source crystals and detector, a He-filled bag that encloses the entire beam path is inserted. An instrument combining such a large solid angle with high energy

resolution is ideally suited for analysis of weak quasi isotropic signals. Among the many possible applications the technique of $K\beta$ fluorescence spectroscopy is illustrated with two examples.

WHERE ARE THE ELECTRONS?

It has been shown that the main lines of the $K\beta$ fluorescence ($3p \rightarrow 1s$ transition) in transition metals are strongly influenced in shape as well as energy by the number of unpaired 3d electrons. Hence $K\beta$ spectra give an excellent signature of the oxidation and/or spin state. Due to their variety of oxidation states, transition metals play an important role in catalytic processes. Obviously the knowledge the oxidation state is of fundamental importance for the understanding of these processes.

One predominant example of a catalytic process in nature is the oxidation of water in photosystem II, a mechanism responsible for essentially all the oxygen in our atmosphere. The catalytic center for this process is a cluster containing 4 Mn atoms. The oxygen is released

after the fourth step in the so called Kok cycle, in each of which a visible light photon had been absorbed.

In collaboration with the Penner-Hahn, Christou and Armstrong Groups, high resolution Mn K β X-ray fluorescence spectra have been recorded on the dark-adapted S_1 state and the hydroquinone-reduced of photosystem II [2]. By comparison of the K β chemical shifts with those of appropriate model compounds, the S_1 state is found to contain equal amounts of Mn(III) and Mn(IV). In the hydroquinone-reduced sample, a significant fraction of the Mn is reduced to Mn(II), and the results are compatible with models involving conversion of Mn(III)₂Mn(IV)₂ to Mn(II)₂Mn(IV)₂ clusters.

In a collaboration with the Klein Group at Calvin Lab, we have then studied the evolution of the Mn oxidation states through different S-states in the Kok cycle. Our preliminary findings are consistent with the Berkeley model of Mn(II,III,IV) for S_0 , Mn(III₂,IV₂) for S_1 and Mn(III,VI₃) for both S_2 and S_3 states [3,4].

WHO ARE THE LIGANDS?

Besides the work on the very dilute protein samples, the large solid angle combined with the high incident flux at X25 enabled us to systematically study much weaker K β emission features appearing at higher emission energies (see Figure T-7). These transitions result from valence orbitals close to the Fermi level and, in some cases, even ligand orbitals. In particular we studied the so called "interatomic" or "crossover" x-ray transitions of ligand (N, O, F) 2s to metal (Mn) 1s [5], also known as K β ".

The energy of the K β " feature is very distinct even for ligands of neighboring Z, resulting in shifts of order 5 eV. It can therefore be used to identify the type of ligand. Furthermore, for a variety of oxygen ligated Mn compounds, the intensity of the K β " transition is found to decrease exponentially with increasing Mn-O. The normalized K β " intensity can be used to predict distances to ~0.1 Å. The result demonstrates the potential of this technique as a new alternative to tell not only who but also where the ligands are. ■

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Figure T-7. K-emission spectrum of MnF₂. The K α spectrum was simulated using the known K α /K β integrated intensity ratio of ~7.7. The main K β lines are split by the 3p-3d exchange interaction into a K β _{1,3} peak and a K β ' feature. These are shown magnified by a factor 15 (dashed line). The much weaker K β _{2,5} and K β " features which are not visible in the main figure are shown magnified by a factor 500 (dotted line).

Newly Commissioned 3-30eV Undulator Beamline U13UB

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The low-K (undulator regime) output of the insertion devices on both NSLS storage rings is essentially "third generation", by being nearly diffraction-limited in one direction (vertical). As such, they represent an important and unique component of the synchrotron radiation facilities available in the U.S. The NSLS undulator sources are U5U and U13U in the VUV photon energy range and X1 and X13 in the soft x-ray range.

The brightness (flux per unit phase space) of the U13U undulator in the photon energy range covered by the U13UB beamline, 3 to 30 eV, is shown in Figure T-10. This figure plots the spectral brightness for seven

values of the magnetic strength parameter K, for which the fundamental photon energy spans the 3 to 30 eV range. Note that the brightness of the undulator output is three orders of magnitude greater than a VUV bending magnet.

In terms of flux, the U13U undulator produces fundamental output greater than 2×10^{15} photons/sec/0.1%bandwidth/Amp in the 3-30 eV photon energy range. This range of fundamental photon energies $h\nu_1$ is readily achieved by changing the magnetic strength K from 4.7 to 1.4. The radiation is directed into a narrow spatial cone, e.g. the $1-\sigma$ opening angle is $210 \mu\text{rad}$ at $h\nu_1 = 15 \text{ eV}$.

Figure T-10. On-axis spectral brightness (flux per unit phase space) of the U13U undulator for seven values of the magnetic strength parameter K (proportional to the product of magnetic field strength and spatial period) spanning the photon energy range covered by the U13UB beamline, 3 to 30 eV. The highest brightness curve at each photon energy is associated with output at the fundamental wavelength. The underlying peaks are higher harmonics ($n \geq 2$) associated with higher K values which, for non-zero-emittance electron beams, appear at all integer multiples of the fundamental wavelength. Note that the brightness of the fundamental is three orders of magnitude greater than a VUV bending magnet.

This article focuses on the new U13UB beamline, which has been in operation since June 1998. The properties of the U13U source and the U13UB beamline are provided, followed by a general overview of the scientific opportunities which we envision for this beamline. Finally, a descriptive overview of two angle-resolved photoemission endstations destined for use at U13UB is given, including a sampling of the first experiments, which are ongoing.

COMPARISON WITH BENDING MAGNET SOURCE

In order to justify the use of a unique resource such as the U13U insertion device, it is necessary to show that a NSLS VUV bending magnet beamline could not produce the desired flux density at the sample. The flux output of 1 μ rad horizontal of an NSLS VUV bending magnet is approximately 1×10^{13} photons/sec/0.1%bandwidth/Amp, and the vertical opening angle of radiation in the 3-30eV photon energy range is 5-10 mrad. In order to compete with U13U in terms of raw flux, a bending magnet beamline must collect a large horizontal fan, e.g. NSLS U11 collects 55 mrad in the horizontal plane.

However, such a beamline is saddled with problems related to the large phase space area of the bending magnet source (horizontally, vertically, and in depth). Clever optical designs, such as the modified Wadsworth monochromator at U11, provide fairly good energy resolution, but at the expense of providing a rather large focal "spot" (1 mm). The transverse (horizontal and vertical) phase space volume of the U13U radiation is on

the order of 250 to 500 times smaller than that of U11, which enables U13U to provide significantly higher flux density at the sample.

From this discussion, it is clear that the experiments which will benefit the most from the high brightness undulator beam at U13UB are those for which flux density on the sample defines the figure of merit. This class of experiments includes all of angle-resolved photoemission spectroscopy (ARPES), for example, since the electron energy analyzer in these experiments can accept emission from only a finite area on the sample. Further, as the energy resolution of these analyzers is improved, the size of this acceptance spot decreases dramatically.

U13UB BEAMLINE DESCRIPTION

Beamline U13UB is one of three branch beamlines which share the output of the U13U NdFeB hybrid wiggler/undulator ($0.5 < K < 7.6$) insertion device on the VUV Ring. The other two branches are U13UA, a focused white light beamline, and U13UC, a coherent optics characterization beamline. Figure T-11 is an elevation layout drawing of the U13UB beamline, which was constructed by McPherson, Inc. of Chelmsford, MA.

The U13UB beamline is separated from the U13U centerline by a horizontally-deflecting (12° grazing angle of incidence) variable-radius ($50\text{m} < R < 200\text{m}$) integrally-water-cooled spherical mirror (M0B), which collects 1.66 mrad and focuses on the exit slit of the U13UB monochromator. The M0B mirror material is glidcop (ceramic copper alloy) explosion-bonded to a stainless steel substrate. The second optical element is a vertically-

Figure T-11. Elevation layout view of the U13UB high resolution VUV spectroscopy beamline, showing the paths of the monochromatic (lower) and white-light (upper) branches.

deflecting (7° angle of incidence — nearly normal incidence) integrally-water-cooled spherical ($R = 1.9\text{m}$) mirror (M1B), which collects 2.4 mrad and focuses (with $\sim 15:1$ demagnification) on the entrance slit (S0B) of the monochromator. The large total output power (287W at $K = 8$, 500mA ring current) and significant output power densities (up to 32.5W/mrad^2 at $K = 8$, 500mA ring current) require water cooling of M0B, M1B, and S0B. Owing to its nearly normal incidence geometry, the lion's share of the source power is absorbed by the M1B mirror. The M1B mirror is a one-inch thick piece of CVD SiC and is integrally water cooled: finite element calculations show that the temperature rise, stress, and resulting deformation are well within the mechanical limits of this material (in fact they are all small).

The U13UB beamline features a Normal Incidence Monochromator (NIM) in which a spherical ($R = 3\text{m}$) grating is used in fixed included angle geometry. The grating chamber houses two gratings on a rotating turret with detents to select either of the two gratings *in situ*. These two gratings (1200 and 2400 line/mm

groove densities), and the $\sim 3\text{m}$ entrance and exit armlengths in this monochromator, produce $>10,000$ spectral resolving power in overlapping photon energy ranges spanning 3 eV to 30 eV . The entire grating housing moves, under interferometric control, along the angle bisector between the entrance and exit beams, in order to track the photon energy-dependent focus of the diffracted exit beam. The grating angle scanning motion is provided by a DC servo system, also under interferometric control. The variable radius of the M0B mirror can be used to correct for the photon-energy-dependent astigmatism of the NIM as it scans through the $3\text{--}30\text{ eV}$ range.

OVERVIEW OF SCIENTIFIC OPPORTUNITIES USING BEAMLINE U13UB

Several types of scientific programs could be expected to benefit greatly by instrumenting the U13U wiggler for high resolution VUV work in the 3 to 30 eV range. Perhaps the most obvious application in solid state physics is ultra-high resolution angle resolved photoemission. In cases where the states of interest lie

Figure T-12. ARPES intensity plot of the Mo(110) surface recorded along the $\bar{\Gamma} - \bar{N}$ line of the surface Brillouin zone at 70°K . Inset: "fixed-mode" spectrum of the region around k_F (marked with box).

near the Fermi level, self-energy corrections are small, and reductions in the photon energy spread will significantly enhance the experiments. High T_c superconductors and heavy fermion systems are examples of such problems.

In addition, surface photo-voltage measurements, chemical dynamics applications, and photoelectron spectromicroscopy are all experiments for which spatial flux density is the relevant figure of merit, and could therefore be expected to be well matched to the U13UB beamline.

The section of beam pipe located vertically above the NIM beamline (see Figure T-11) houses relay optics which will provide a time-delayed (or advanced) white light beam, the focus of which will be collinear and coincident with that of the NIM beamline, for the purpose of pump-probe experiments. This capability is expected to be central to a large fraction of the biology experiments planned for U13UB.

ANGLE-RESOLVED PHOTOEMISSION ENDSTATIONS AND EXPERIMENTS

Angle-resolved photoemission, developed in the seventies, still represents the premier tool available for the study of the momentum dependence of the electronic structure in solids. Here the energy of the emitted photoelectron is measured as a function of the angle of emission. Combined with beamlines such as U13UB, a

whole range of new electron spectrometers is expected to lead to a resurgence of activity in this area. By multiplexing in energy and angle, these new instruments allow a considerable reduction in data acquisition times. At the same time, they offer extremely high energy and momentum resolution. With these improvements, ARPES may be used to obtain direct access to the real and imaginary components of the electron self energy, a measure of the interaction of an electron or hole with its environment.

Two instruments are scheduled to exploit the high photon fluxes available from U13UB. The first is a 200 mm radius Scienta SES200 analyzer that has recently been commissioned in the group of Peter Johnson (Physics - BNL) and the second is a Gamma Data 50 mm radius spectrometer currently being constructed by Eric Jensen (Brandeis U.) and Kevin Smith (Boston U.).

Initial experiments using the SES200 have already been completed. An example is presented in Figure T-12, which shows a measurement of the dispersion of an electronic band in the vicinity of the surface of molybdenum^[1]. Shown in more detail in the insert, the spectra clearly show a slight change in the rate of dispersion and a rapid narrowing of the band in the vicinity of the Fermi level. These changes are directly attributable to the interaction of the electrons with the lattice vibrations or phonons in this system.

Figure T-13. Three-dimensional photoemission map of $K_{0.3}MoO_3$, showing two parallel bands dispersing toward the Fermi level.

Another example of the use of high momentum resolution is shown in Figure T-13. Here are shown the results of a study of the blue bronze, $K_{0.3}MoO_3$, which is a quasi-one-dimensional system having two chains per unit cell^[2]. The valence electrons in this system form a charge density wave that results in a metal-semiconductor transition at 180°K. This study, carried out in collaboration with the Boston University Electronic Structure Group (K. Smith *et al.*), was able for the first time to clearly resolve two bands reflecting the interaction between the chains, as shown in Figure T-13. Furthermore, a detailed study of the temperature dependence of the wavevectors associated with these two bands provided a direct connection between the electronic structure and the temperature dependence observed for the wavevector of the charge density wave in this system.

As a final example of the new possibilities in ARPES, we show the results of a detailed temperature dependent study of optimally doped $Bi_2Sr_2CaCu_2O_8$ (BSCCO)^[3]. These studies have found evidence for the opening of a gap in the spectral response with the onset of long range coherence at the superconducting transition temperature T_c . Figure T-14 shows the emission from a region of momentum space when the sample is in the superconducting state. The “airplane wing” section corresponds to the superconducting paired electrons. Figure T-15 shows temperature dependent “angle integrated” spectra obtained from images such as those shown in Figure T-14.

Figure T-15 shows that the sharp feature immediately below the Fermi level characterizing the superconducting state appears in the spectra even above the transition temperature T_c , 91°K, and that it remains at constant binding energy at all temperatures. Further, the spectra show that the intensity of this feature changes continuously on passing through T_c . This latter observation is surprising because within the framework of a mean field or BCS model, if the sharp peak reflects the super fluid density, it should disappear at T_c and, further, the separation between it and the Fermi level should disappear. However, the authors of the study, Fedorov, *et al.*, noted that at the transition temperature, a dip rapidly develops in the spectra immediately beyond the sharp peak. By fitting the angle-resolved data collected within an angle of 0.5°, Fedorov, *et al.* were able to show that the development of this dip may be associated with the opening of a gap at the Fermi level, reflecting the onset of long range phase coherence as defined by the transition temperature T_c . The latter development represents the extension of the pseudogap, which is reported to exist above T_c up to 130K in the optimally doped materials, to all states in the system. ■

Figure T-14 (Spectral density plot of optimally-doped $Bi_2Sr_2CaCu_2O_8$ recorded in the Γ M direction of the Brillouin zone with the sample temperature held at 46°K. The incident photon energy is 21.2 eV. The most intense emission is indicated in red.

The U13UB beamline was assembled, installed, leak tested, and mechanically debugged by the technical members of the NSLS Experimental Systems Group, especially Gary Nintzel and Dennis Carlson. The beamline components were positioned by the NSLS Survey Team, headed by R. Scheuerer, to such a precision that getting undulator light to the sample was easy. All of the U13UB beamline components were designed and fabricated by McPherson, Inc. of Chelmsford, MA, including alignment of the NIM gratings and interferometer.

Figure T-15. Angle-integrated photoemission spectra of optimally-doped $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ recorded as a function of temperature and integrated along the Γ M line of the Brillouin zone. The different temperatures are indicated.

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A General Solution to the Diffraction Phase Problem in Systems with Two-Dimensional Periodicity

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Material systems periodic in two dimensions and aperiodic in the third (2D crystals) are very interesting scientifically and often very important technologically. Crystal surfaces, crystals with epitaxially grown layers of another material, and interfaces, are all 2D crystals. They are scientifically interesting because physical and chemical phenomena at surfaces and interfaces are often different from those in the bulk, and are technologically relevant because many devices depend for their performance on material interfaces. Systems with an interface between a crystal and an amorphous material (e.g., Si and its oxide) are not ideal 2D crystals. However in the region of the interface, they do have partial two-dimensional periodicity. Finally, self-organized systems on crystalline surfaces are also 2D crystals and are currently of great interest both scientifically and technologically. Knowing

the structure of these systems is crucial to understanding their properties and to the development of new processing techniques and devices.

Various x-ray methods have been used to investigate the structure of 2D crystals. These include reflectivity and diffuse scattering^[1,2], high resolution x-ray diffraction^[3], Multiple diffraction^[4] and standing waves^[5]. In all these methods, the structure cannot be directly determined. Instead it is necessary to first guess a structural model and then refine its parameters to obtain best fit with experiment. A step in overcoming this difficulty has been the development of a method to determine the layer structure using anomalous reflectivity^[6].

In this paper we present a new x-ray method to determine directly the atomic structure of 2D crystals. We have successfully tested the method both in

Figure T-16: Schematic diagram of the experiment. The sample consists of a GaAs crystal cleaved along the [110] plane. 4 monolayers of AlAs and 20 monolayers of GaAs were grown epitaxially on its surface. Two samples were prepared, with and without a 2000Å gold overlayer. The gold film was used in order to measure the phase derivative along the Bragg rods as described previously^[7,8].

simulations and in actual experiments. Using this method, we can determine the structure directly without the need to presuppose a structural model. As in all direct methods we measure not only the amplitude of the scattering factor but also its phase. The method we developed is general but it will be easier to explain using an example, shown in Figure T-16.

We consider a material system composed of a three-dimensional semi-infinite crystal with a known structure and a 2D crystal with an unknown structure. By definition, the 2D crystal is the difference between the whole system and the ideal 3D *semi-infinite* crystal. In our example, the 3D crystal is GaAs, extending all the way to the sample surface. The unknown 2D crystal is a few monolayers of epitaxial AlAs and its electron density is equal to the difference between the electron density of the ideal GaAs and that of the AlAs. Notice that if the GaAs is distorted relative to the ideal structure the difference will also show up in the 2D crystal representing the epitaxial AlAs and its interface with the GaAs.

The 3D crystal defines a 2D grid on planes parallel to its surface. At first we assume that the 2D crystal is periodic with respect to this grid. The complex scattering factor of the semi-infinite substrate crystal $S(k_z)$ is well-defined because its structure is known. However, the scattering factor of the 2D crystal $D(k)$, which will contribute to the scattering intensity along the Bragg rods, is not known. What is directly measured is the absolute value of the total scattering factor $|T|$ which is the absolute value of the complex sum of the two. We now consider two points along a Bragg rod at a small separation Δk_z from each other. Then,

$$S(k_z) + D(k_z) = |T(k_z)| e^{i\varphi(k_z)} \quad (1)$$

$$S(k_z + \Delta k_z) + D(k_z + \Delta k_z) = |T(k_z + \Delta k_z)| e^{i[\varphi(k_z) + \partial\varphi/\partial k_z \Delta k_z]} \quad (2)$$

Here, $\varphi(k_z)$ is the unknown phase of T and $\partial\varphi/\partial k_z$ is the derivative of this phase along the Bragg rod. Notice that these equations cannot be solved for the complex scattering factor of the unknown crystal, $D(k)$ because $\varphi(k_z)$ is not known. We have recently developed, and experimentally demonstrated, a method to measure the phase derivative $\partial\varphi/\partial k_z$ along the Bragg rods^[7,8]. In principle one could integrate the measured phase derivative in order to obtain the phase itself. The problem is that even a small amount of noise in the experimental data produces large errors in the phase, rendering the method useless. It was therefore necessary to find a way to use the phase derivative directly without the need to integrate.

In Eqs. 1 and 2 we have assumed that the real-space zero reference point of the x-ray dielectric function is at the sample surface. If this reference point is shifted in the z direction by z_0 , Eqs. 1 and 2 will have the form:

$$S(k_z) e^{ik_z z_0} + D(k_z) e^{ik_z z_0} = |T(k_z)| e^{i\varphi(k_z)} e^{ik_z z_0} \quad (3)$$

$$S(k_z + \Delta k_z) e^{i(k_z + \Delta k_z) z_0} + D(k_z + \Delta k_z) e^{i(k_z + \Delta k_z) z_0} = |T(k_z + \Delta k_z)| e^{i[\varphi(k_z) + \partial\varphi/\partial k_z \Delta k_z]} e^{i(k_z + \Delta k_z) z_0} \quad (4)$$

We now find a z_0 value such that

$$\Delta \bar{D} = |\bar{D}(k_z) - \bar{D}(k_z + \Delta k_z)| \quad (5)$$

has a minimum. Here $\bar{D}(k_z) = D(k_z) e^{ik_z z_0}$. Notice that in this case the complex numbers $\bar{D}(k_z)$ and $\bar{D}(k_z + \Delta k_z)$ have the same argument. If $\Delta \bar{D}$ is at its minimal value, its absolute value will be in general small compared to $|\Delta \bar{S}|$ and $|\Delta \bar{T}|$ and can therefore be neglected. Then the absolute value of the difference between Eqs. 3 and 4 has the form:

$$|S(k_z + \Delta k_z) e^{i\Delta k_z z_0} - S(k_z)| = |T(k_z + \Delta k_z) e^{i\partial\varphi/\partial k_z \Delta k_z} e^{i\Delta k_z z_0} - |T(k_z)|| \quad (6)$$

Notice that here all the values except for z_0 are known. So this equation can be used to determine z_0 . We can now use the ratio between Eqs. 3 and 4 to determine $\bar{D}$.

$$\frac{\bar{S}(k_z + \Delta k_z) + \bar{D}}{\bar{S}(k_z) + \bar{D}} = \frac{|T(k_z + \Delta k_z)| e^{i(\partial\varphi/\partial k_z + z_0)\Delta k_z}}{|T(k_z)|} \quad (7)$$

Where,

$$\bar{D} = D(k_z) e^{ik_z z_0} \cong D(k_z + \Delta k_z) e^{i(k_z + \Delta k_z) z_0} \quad (8)$$

This procedure provides $D(k_z)$ for all relevant Bragg rods in reciprocal space. Back-Fourier transforming it in three-dimensions into real space, provides the 3D real space dielectric function $\epsilon(\vec{\rho}, z)$. Here $\vec{\rho}$ is the position vector in the 2D unit cell and z is the distance in the direction normal to the plane of the 2D crystal.

It turns out that in many systems the value of z_0 can be estimated without measuring $\partial\varphi/\partial k_z$ along the Bragg rods. In systems where the thickness of the unknown 2D structure is small compared to its distance from the crystal surface, z_0 is approximately equal to this distance. In this case Eq. 6 can be used to determine $\partial\varphi/\partial k_z$ and Eq. 7 to determine the value of $\bar{D}$.

The method we developed is also applicable if the unknown 2D system is not fully periodic with respect to the 2D grid. It can be shown that in this case the exact same procedure provides an average complex dielectric function $\epsilon(\vec{\rho}, z)$

$$\varepsilon(\vec{\rho}, z) = \frac{1}{N} \sum_j \varepsilon(\vec{\rho} + \vec{R}_j, z) \quad (9)$$

Here, $\vec{\rho}$ is a position vector in the substrate-defined 2D unit cell; $\vec{R}_j$ is the 2D position of the j^{th} unit cell, z is the distance from the nominal interface and the sum is carried over all 2D unit cells.

We have demonstrated this method experimentally using the system shown in Figure T-16. The diffraction profiles were measured along one Bragg rod from $[-5 -1 1]$ to $[1 5 1]$. The data were then analyzed using the method discussed above, resulting in the complex scattering factor along the Bragg rod. This was then back-Fourier transformed in one dimension resulting in the Al density as a function of the distance from the crystal surface.

In Figure T-17 we show the Al concentration as a function of the distance from the surface. The top curve represents the Al concentration obtained from simulated diffraction data calculated for the nominal sample used in the experiment. We included in this data noise and background typical of those we have in our experiments. The results are shown in curve (c). Each peak in the figure represents one monolayer. The distance between monolayers is $2\text{\AA}$, namely we have atomic resolution. Notice that the analysis indeed faithfully reproduces the initial structure.

Curves 2a and 2b represent the structure obtained from the analysis of the experimental data. Note that Al is present not only in the 4 monolayers that were intended as the AlAs 2D crystal, but also there is some Al concentration in one or two additional monolayers which were intended to be pure GaAs. On the other hand the

structure of the cleaved crystal seen on the right of the AlAs layer has hardly been affected. It is also interesting to notice that the number of GaAs monolayers above the AlAs layer is different in the two samples in spite of the fact that the two samples were grown simultaneously but at two different places in the growth chamber.

It should be emphasized that the results shown in Figure T-17 are from analyzing just one Bragg rod. Determining the complex scattering factor along all the relevant Bragg rods and back Fourier transforming the results in 3D will provide the positions of the various atoms in the 2D-unit-cell as a function of the distance from the surface.

In conclusion, we have demonstrated a method to experimentally determine the complex scattering factor along the Bragg rods of a 2D crystal. We have shown that using this data we can determine the structure of the 2D crystal by back-Fourier transforming the results. We expect that this method will be useful in the investigation of crystal surface structures, interfaces between a crystal and epitaxially grown layers, crystal-melt and crystal-solution, and self-ordered systems grown on a crystalline surface. The method can also be used to investigate structural phase transitions at surfaces and interfaces. ■

Figure T-17. Al concentration as a function of distance from the sample surface. (a) and (b)- experimental results of two different samples. (c) The structure reconstructed from simulated data. All the results shown here were obtained without using the phase derivative along the Bragg rods.

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Beamline X20A: Silicon-germanium (SiGe) films, grown epitaxially on Si substrates, are a critical component in high-speed transistors. The lattice parameter of SiGe alloys is slightly larger than that of Si and, therefore, a very thin SiGe film grown on a Si substrate is compressively strained. When the SiGe film thickness exceeds a critical value, the mismatch strain is relieved by the introduction of misfit dislocations. If the amount of Ge in the alloy is increased gradually from pure Si up to the desired alloy composition (typically up to about 30% Ge), the dislocations nucleate by a multiplication mechanism which results in an array of dislocation pile-ups in the lower part of the graded layer and the top of the Si substrate. However, the upper part of the strain-relaxed SiGe film has a low threading dislocation density, thus making these films suitable as "substrates" for the strained Si or SiGe layers of different alloy composition which form the active regions of a field effect transistor. The strain field of the dislocations distorts or bends the crystal lattice planes, and the resulting mosaic structure depends on the arrangement of the misfit dislocations. An x-ray microbeam (5 x 1 micron diffracted beam footprint) was used to study the microstructure of various relaxed SiGe films and individual local tilted regions were observed for the first time. The 3D plot is derived from a series of 004 rocking curves (theta scans) taken at intervals of 2 microns along the surface of a uniform composition $\text{Si}_{0.7}\text{Ge}_{0.3}$ layer grown on top of a graded SiGe layer. Each peak represents a distinct region of the layer having a tilt angle (theta) different from that of its neighbors. The lateral size of these local tilted regions is about 12 microns.

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THREE MEETINGS & WORKSHOPS

MEETINGS

THE 1998 NSLS ANNUAL USERS' MEETING

John B. Parise

SUNY at Stony Brook

NSLS Users' Executive Committee Chairman

The NSLS users held their Annual Users' Meeting on Tuesday May 19, 1998 at Brookhaven National Laboratory (BNL). It allowed many users to meet the new BNL contractor, Brookhaven Science Associates (BSA), for the first time, obtain the latest news from the Department of Energy (DOE), and review and celebrate scientific accomplishments. On the Monday and Wednesday bracketing this meeting, users attended a total of six workshops.

The meeting began with an extended introduction by Peter Paul, BNL Deputy Director for Science & Technology. John Marburger, Director of BNL then elaborated on these remarks, pointing out that users of the facilities at BNL are its primary customers and summarizing some of the trauma which resulted in the termination of the AUI contract to operate BNL and the successful bid by BSA. He indicated a willingness to represent the interests of users in Washington DC, and particularly the need to improve infrastructure and user support.

The subject of the meeting then turned to science. Larry Carr of the NSLS gave a presentation entitled "Infrared at the NSLS - A Long Wavelength Probe of Small Things on Short Time Scales". Larry described the facilities at U12IR along with spectra showing the performance advantage over conventional sources. The signal throughput for small samples exceeded that obtained with the spectrometer's internal source (a high pressure mercury arc lamp) by more than 2 orders of magnitude in the far infrared. Spectra down to 1 cm^{-1} were shown, including an observation of enhanced signal at 1.4 cm^{-1} (42GHz).

Cev Noyan of the T.J. Watson Research Center of IBM described the "Determination of Mechanical Properties of Thin Film Interfaces with X-Ray Microdiffraction" at the X20A beamline. The (buried) interfaces between polycrystalline Al, Cu and W thin-film features and their substrate (single crystal Si) were characterized using the Si (004) reflection. Topographic images of the Si around and under the metallization

John Marburger, New Director of Brookhaven National Laboratory, updating users on recent changes at BNL and its new manager, Brookhaven Science Associates.

features were constructed, showing that a fully elastic "mosaic" structure develops in the Si under the thin film features.

The first speaker after the morning break was Michael Hart, Chairman of the NSLS. He pointed out the positive light in which the NSLS was painted by the recent BESAC review and that the FY99 presidential budget includes an increase for support of users of some 5.2%. He cautioned, however, that these increases might prove illusory. Michael Hart also pointed out the dramatic increase in the number of life science users over the past 5 years and the expectation that they will soon constitute the largest user group at the NSLS.

Some of the concerns with funding by the U. S. Department of Energy (DOE) were addressed by Robert Marianelli, Director of the Division of Chemical Sciences for the Office of Basic Energy Sciences (BES). He assured the users of the importance of basic science to the mission of the DOE. The BES program contributes 2 billion dollars per year, or 17% of all federal funding for the physical sciences. In response to questions regarding the budget negotiation process, he agreed that it is every citizen's right to make their elected representatives aware of decisions which adversely affect their interests.

Barbara Illman (University of Wisconsin, USDA/FS Forest Products Lab) next described part of her research program at beamlines X26A and X27. With the microprobe at X26A, she and collaborators are employing new, non-destructive synchrotron techniques for detecting iron and manganese oxidation states in wood to study biochemical mechanisms of fungal decay. She and Betsy Dowd (NSLS) work at X27 to develop novel synchrotron techniques for element mapping in 3-dimensional images of preservative-treated wood.

In the time remaining in the morning session the users had an opportunity to thank representatives from NSLS staff and BNL Plant Engineering who worked to repair power cables responsible for a shutdown in December 1997. The repair to the NSLS were so well managed that most users did not know there had been a major power failure until the User Meeting.

The Scientific Highlight speech was given directly after lunch. Nobelist James Watson, President of the Cold Spring Harbor Laboratory, presented a retrospective of his life's work "From the Double Helix to the Human Genome Project". This was followed by a presentation by Wayne Hendrickson of Columbia University/HHMI. In collaboration with researchers from Dana-Farber

Left to right, NSLS Annual Users' Meeting Organizing Committee: John Hill (BNL Physics), Harald Ade (NCSU), Nancye Wright (NSLS), Elaine DiMasi (BNL Physics), Linda Feierabend (NSLS) and John Parise (SUNY at Stony Brook).

Cancer Institute, SmithKline Beecham Pharmaceuticals, and Tulane University Medical Center, they were able to obtain small crystals of the HIV-1 gp120 envelope glycoprotein in complex with the CD4 receptor and a neutralizing human antibody. The crystals were needles of only 30-40 microns in cross-section, and required the intense synchrotron radiation on NSLS beamline X4A for data collection. They were able to solve the structure at 2.5 Å resolution. The structure reveals the details of the CD4-gp120 interface, a conserved binding site for the chemokine receptor, evidence for a conformational change upon CD4 binding, the nature of a CD4-induced antibody epitope, and specific mechanisms for immune evasion. The results provide a framework for understanding the complex biology of HIV entry into cells and should guide efforts at intervention.

Ian Robinson of the Physics Department, University of Illinois, Urbana spoke about grazing X-rays and electrochemical interfaces. The examples presented included the electrochemical oxidation of copper, specifically at the Cu(111) surface. Structural studies of the native oxide formed in air show an epitaxial film of Cu₂O (cuprite) which gradually thickens over several hours following electropolishing. Both hexagonal orientations of the interface form simultaneously with equal proportions. The situation is very different in acidic solution, where the oxidation and reduction can be driven by an external potential. There, only one epitaxial orientation of the film is found to grow thick; the other orientation forms, but always remains a single monolayer in thickness.

Peter D. Johnson of the BNL Physics Department gave a talk on recent advances in High Resolution Photoemission applied to the study of low dimensional oxides, in particular the High T_c superconducting materials. New developments in instrumentation now allow the energy and angular distributions of the photoelectrons to be measured in parallel, and with much higher energy resolution than in earlier studies.

The Keynote Address was delivered by Dr. Ernest J. Moniz, Under Secretary of Energy at the US Department of Energy. Given the recent history of BNL, his opinions were of interest to an audience beyond the users of the

NSLS and the numbers of people in the auditorium swelled during his presentation. Dr. Moniz began by commenting that BNL is regarded as amongst the best of the research laboratories in the country and that the new BNL management team lead by Dr. John Marburger is "hitting the ground running". He also emphasized that the recent Birgeneau report, which emphasized the growing importance of the four DOE synchrotrons in the nation's research programs, praised the NSLS for the number of users served and its education and outreach to new users.

Other important parts of the meeting included the reception the evening before the meeting, poster sessions, a barbeque luncheon outdoors under a tent and an equipment exhibit attended by 17 vendors. All were held in Berkner Hall and all were popular with the users. The election of three new members to the User Executive Committee took place during the Annual Meeting: John Hill (BNL Physics Department), Carol Hirschmugl (U. of Wisconsin), and Lisa Kelly (U. of Maryland) were elected as general members, and the UEC chose Barbara Illman to be its Vice-Chair (Chair-Elect). The Annual Meeting was capped off that evening by a banquet at the Port Jefferson Country Club which overlooks the Long Island Sound. ■

Left to right: John Parise (SUNY at Stony Brook), current NSLS User Executive Committee Chairman; Carl Zimba (NIST); and Joel Brock (Cornell U.) former UEC Chairman, between User Meeting sessions.

WORKSHOPS

Using Powder Data to Solve Crystal Structures

David Cox
Peter Stephens
(SUNY @ Stony Brook)

This workshop, held on May 18, 1998 at the Annual NSLS Users' Meeting, was motivated by the recent rapid advances in the application of powder diffraction techniques to the *ab initio* solution of unknown structures. In contrast to the well-known Rietveld method for structure refinement, this is a relatively new application which has been driven mainly by the development of high-resolution synchrotron x-ray techniques, especially for framework structures such as zeolites, fullerene derivatives and other molecular compounds, and small organic molecules of pharmaceutical significance. The roughly 80 attendees were treated to a state-of-the-art exposition by an international cast of speakers from the USA and Europe, where much of the cutting-edge work in this field is currently taking place.

After Henk Schenk (U. of Amsterdam) started the proceedings with an introduction to the use of direct methods for structure solution from powder data, zeolites took center stage with talks by Lynne McCusker (ETH, Zurich) about her structural "toolbox" and two new tools for solving zeolite structures, texture and structure envelopes, and Ralf Grosse-Kunstleve (Yale U.) on a third new tool, which allows useful crystal chemical information to be incorporated via Fourier recycling and a search for unique topologies (the "FOCUS" method). John Newsam (Molecular Simulations Corp.) described some successful approaches to structure determination based on model construction and simulated annealing, and Dick Harlow (DuPont Corp.) closed out the morning session with an update on the \$1000 "DuPont Challenge", to be awarded to the first person to solve the structure of HAlF_4 (an intermediate product in the industrial synthesis of AlF_3 catalysts) from powder data collected at the NSLS. No winner has yet been announced!

Molecular and organic compounds were the focus of the afternoon talks, led off by Ken Shankland (Rutherford Appleton Lab.) with a description of real-space techniques that are suitable for organic molecules, including examples of structures solved by simulated annealing and genetic algorithms. Jim Kaduk (Amoco Corp.) discussed the structure solution of several moderately large organic molecules using more conventional techniques, while Robert Dinnebier (U. of Bayreuth) compared standard tools (such as direct methods) and some unconventional algorithms, including extended use of chemical constraints and rigid bodies, grid searches, and the use of pseudo-atoms for compact units such as phenyl rings to a variety of structure solutions.

Peter Stephens (SUNY, Stony Brook) discussed several aspects of lineshape modelling, including a newly-developed algorithm for handling anisotropic broadening due to lattice strains, and in the final talk, Bob Von Dreele (Los Alamos National Lab.) described some first attempts at protein crystallography with powder samples, in which with appropriate constraints he was able to refine the structure of whale myoglobin (1401 atoms in a 65,000 Å³ unit cell!) from high-resolution data taken at the NSLS.

On this impressive note, Peter Stephens closed the meeting by urging the audience to make more use of the several high-resolution synchrotron powder instruments available, and to apply these powerful new tools for *ab initio* structure solution to more complex materials of scientific and commercial interest.

The success of the workshop was in no small part due to the support of the following sponsors whose generosity is gratefully acknowledged: the International Centre for Diffraction Data, Air Products, Amoco, Chevron, Clariant, Mobil, UOP, SUNY X3B1 Powder Diffraction Facility, X7A PRT. ■

WORKSHOP

***In Situ* Surface Manipulation**

Ian Robinson
(University of Illinois @ Urbana)

A workshop on *in situ* manipulation of surfaces using X-Ray diffraction methods was held in conjunction with the Annual NSLS Users' Meeting on Wednesday, May 20, 1998 from 9:00am to 5:30pm. The workshop's emphasis was the structure and properties of surfaces and interfaces in their working environments. An exciting and stimulating series of talks was presented, covering a wide range of physics, chemistry and materials topics that have been addressed by experiments at NSLS. Valuable support for the meeting came from the Users' Executive Committee. The attendance varied from 25 to 50 as participants selected parts of the program of interest to them.

Bill Elliot from the University of Missouri spoke about growth morphology of Ag during homoepitaxy, where he reported roughness exponents at different growth temperatures. Hubert Zajonz from the BNL Physics Department and Odile Robach of the University of Illinois compared their heteroepitaxial deposition systems, Cu on Ru(0001) and Ni on Ag(001). New and unique unit-cell structures are stabilized by epitaxial forces in the thin-film state.

Joel Brock of Cornell University presented his new results on the nucleation and ordering kinetics of Cu films electrochemically deposited on Pt(111). His group used a periodic drive of the applied potential to attain good statistics with excellent temporal resolution. Mary Ryan and Jia Wang of the BNL Department of Applied Science then presented their respective structural studies of the passive oxide film formed on iron and the Tl-Br coadsorbate on Au(111). In both cases, the chemical

complexity of these highly relevant compound systems was impressive, and structural details down to the oxygen site occupation and the Au displacements were discovered.

After an outdoor barbecue lunch, Ken Evans-Lutterodt of Lucent Technologies talked about a new kind of order detected in very thin SiO₂ layers on Si, an interface of great importance to the semiconductor industry. A discussion broke out concerning the different aspects of structure probed by reflectivity and truncation rod measurements. This theme was developed further by Pulak Dutta of Northwestern University who spoke about self-assembled and Langmuir monolayers on Si substrates. Elaine DiMasi of BNL Physics followed with animated results of surface segregation in liquid Hg alloys, which showed very different temperature dependence from pure Hg. Then Mike Bedzyk, also of Northwestern University, introduced the standing-wave technique with its *in situ* applications to the interface between calcite and water.

Art Baddorf of ORNL introduced his original ideas about dynamic Smoluchowski smoothing of surfaces and its coupling to surface vibrations. High-frequency optic modes lead to transient structures containing paired atoms (just like in a reconstruction) whose electronic response drives a bigger relaxation than in ideal non-vibrating surfaces. The final speaker of the day was Joe Woicik of NIST, who captured some overflow audience from the already closed EXAFS workshop for his presentation on EXAFS and diffraction determinations of InGaAs interatomic spacings. ■

WORKSHOPS

Crystallography Under High Pressure: Techniques for Everyone

Jiuhua Chen
(SUNY @ Stony Brook)

Synchrotron radiation has greatly benefited high pressure research. A high pressure apparatus has either a very small amount of sample or a large amount of pressure medium surrounding the sample; therefore, *in situ* high pressure studies with x-rays have a great demand for synchrotron radiation. This workshop was held on May 20, 1998, in conjunction with the NSLS Users' Meeting and co-sponsored by the Center for High Pressure Research (CHiPR). In this one-day workshop, the most recent technological developments and scientific research with synchrotron radiation under high pressure were reviewed. Ten invited speakers presented the latest high pressure techniques and research at the NSLS, APS, ESRF, SPring-8 and PF.

After a brief introduction by Jiuhua Chen (workshop organizer, SUNY at Stony Brook), Donald Weidner (Director, CHiPR, SUNY at Stony Brook) and Russell Hemeley (CHiPR, Carnegie Institute at Washington) reviewed the research activities of the large-volume press (LVP) and the diamond anvil cell (DAC) at the NSLS, respectively. Many pioneer developments of methodology and technology for LVP and DAC, such as rheological measurement, uniform laser heating and translating imaging plate system, were presented. A comprehensive HP system including both LVP and DAC is emerging at the NSLS. The studies have a very important impact in understanding the interior of the earth as well as some other aspects of physics and material science.

In the late morning session, two special topic talks on ultrasonic measurement of sound velocities with LVP and x-ray spectroscopy with DAC were given by Robert C. Liebermann (CHiPR, SUNY at Stony Brook) and Jean-Pascal Rueff (NSLS). A simultaneous measurement

of ultrasonic sound velocity and x-ray diffraction has been achieved for the first time at the X17B1 beamline. This development opens new opportunities for establishing absolute pressure scales. Spectroscopic techniques, including inelastic scattering, x-ray absorption and emission, with a Be-gasket DAC provide valuable information for understanding the electronic structures of materials under high pressure. A general discussion on Rietveld refinement with energy dispersive powder diffraction data using synchrotron radiation light source, as well as a neutron "heavy" source, was given by Robert B. Von Dreele (Los Alamos National Laboratory), and some pioneer trials of LaBeil fit and Rietveld fit for high-pressure diffraction data collected at the NSLS were also discussed.

The afternoon sessions focused on the reports of high pressure research from the third-generation synchrotron radiation facilities. Francesco Sette (ESRF) presented the development of inelastic x-ray scattering measurements in the ~20 KeV energy region with energy resolutions tunable in 1.5 - 7 meV range. This technique overcomes the kinematic limitations of the traditional neutron inelastic scattering system and favors high-pressure experiments with small samples. An overview of the high pressure research at the ESRF was given by Daniel Hausermann. The high pressure cell has been a widely accessed tool at the ESRF. In the past year, about one third of the over 20 optimized beamlines have been used to carry out the high pressure experiments. The 12-second cycle "Fastscan" imaging plate detector enhanced the capabilities of LVP and laser-heating DAC. Combining an IP detector and a background-subtraction technique, which was initiated at CHiPR, reaction and synthesis kinetics was studied using the LVP. Osamu

Shimomura (SPring-8) introduced the recent high pressure research in Japan (both Photon Factory and SPring-8). The result of spinel - perovskite+perclase phase boundary study using the double-stage large-volume apparatus, SPEED1500, was published as the first research paper of the SPring-8 facility. The press offers the highest pressure so far reached in an LVP in conjunction with synchrotron radiation.

The high-pressure research at GeoSoilEnviroCARS was reviewed by Yanbin Wang (APS). Both the DAC and LVP systems at the APS have been complemented based on the success of CHiPR at the NSLS. HPCAT is a proposed integrated high-pressure facility at the APS. David Mao (CHiPR, Carnegie Institute at Washington) described the beamline design which will integrate the well established HP x-ray diffraction programs and the new frontiers in HP x-ray spectroscopy. ■

Biological and Environmental Applications

Barbara Illman
(University of Wisconsin)

Biological and environmental sciences are increasingly using synchrotron techniques in diverse areas of research. A workshop on biology and environmental applications was held on May 18, 1998 in conjunction with the 1998 Annual NSLS Users' Meeting. This workshop included research conducted on a variety of X-ray and VUV beamlines at NSLS. Speakers discussed current and future developments in X-ray computed microtomography, EXAFS, microXANES, STXM, soft x-ray microscopy, fluorescence spectroscopy, and x-ray crystallography for studies in molecular and developmental biology, human pathology, bioremediation of toxic wastes, impact of toxins on ecosystem dynamics, plant pathology, and forest ecology. James Penner-Hahn (U. of Michigan) presented research on spatial and temporal imaging of Zn speciation during development of fish eggs using microXANES. Paul Bertsch (U. of Georgia) presented research on the distribution and chemical speciation of metals and metalloids in environmental samples by micro X-ray fluorescence and micro X-ray absorption spectroscopies. Mark Chance (Albert Einstein Coll. of Med.) described the use of synchrotron X-radiation for studies on the structural

biology of RNA folding. Cathryn Lawson (BNL Biology) told of protein crystallography to unlock the secrets of the tick-borne pathogen that causes Lyme disease. Don Huber (Purdue U.) told how microXANES was used in monitoring oxidative reactions to determine basic mechanisms of plant pathogenesis. Ulrich Neuhaeusler (SUNY at Stony Brook) summarized the soft X-ray spectromicroscopy research at X1A, including studies of hydrated colloidal systems. Formation of a PRT for the recently developed X-ray microtomography beamline at NSLS was described by Barbara Illman (U. of Wisconsin, Forest Products Laboratory). Two speakers gave talks for this workshop and for the workshop on Applications of Infrared Synchrotron Radiation. George Flynn questioned the potential of organic-carbon-based molecules on extraterrestrial samples using FTIR and STXM measurements. Lisa Miller (Albert Einstein Coll. of Med.) presented applications of synchrotron infrared microspectroscopy to the study of biological cells and tissues. The talks in this workshop represented synchrotron-based research that is making an impact on the biological and environmental sciences. ■

WORKSHOPS

XANES and NEXAFS Spectroscopy: Theory and Applications

Boyan Boyanov
(North Carolina State University)

As x-ray absorption spectroscopy (XAS) matures as an analytical technique, more emphasis is placed on understanding and quantifying the fine structure in the near-edge region of the absorption spectrum. This year's workshop, which is the continuation of a series of XAS Workshops held at the NSLS Users' Meeting, reviewed recent progress in theoretical, acquisition, and analysis techniques, and facilities development at NSLS. More than 80 people attended the workshop.

The current state of the full multiple-scattering (FMS) technique for calculating XANES spectra was reviewed by Bruce Ravel from the National Institute of Standards and Technology (NIST). Bruce gave attendees an overview of the development goals and capabilities of FEFF8. Examples covered ranged from fairly routine simulations of the near-edge structure of K_2CrO_4 , to difficult and time-consuming calculations for diamond. The factors affecting the precision and difficulty of the calculation, as well as the advantages and disadvantages of the FMS method in comparison to molecular orbital approaches, were discussed.

Stephen Wasserman from the Advanced Photon Source (APS/ANL) described the application of principle component analysis (PCA) for chemical speciation using XAS. Steve's presentation included many useful tips on practical issues, particularly on the difficult problem of deciding how many components describe the data in sufficient detail. The main advantages of the PCA method are that it provides an estimate of the number of distinct species observed in a series of measurements even when detailed analysis is not possible, and the ability to test whether a model compound is a constituent of the system being examined, without the need to know the other species present.

The present status of the Dow/NIST materials characterization facility at beamline U7A was reviewed by Daniel Fischer from NIST. The facility enables experimenters to perform new soft-x-ray materials science to study the chemical nature of diverse materials. Practical industrial problems currently being investigated include model catalyst systems, polymer surfaces and their interfaces, hard disk lubricant chemistry, self assembled monolayers, and high T_c superconducting tapes.

Stephen Urquhart from North Carolina State University (NCSTU) discussed the use of XAS for the characterization of polymer systems. Stephen's talk explored the chemical information content of polymer NEXAFS spectra and the use of molecular models for the interpretation of the relationships between polymer structure and x-ray absorption spectra. He examined the significant perturbations of high-resolution spectra caused by electronic delocalization, and showed how the chemical sensitivity of NEXAFS can be utilized to perform chemical microanalysis of real-life polymers.

The final talk was given by Angelika Osanna from SUNY Stony Brook, who described the use of image stacks at beamline X1A to obtain images of sub-100 nm resolution of polymer and biological systems. Angelika gave a real-time demonstration of the acquisition and analysis software developed by Chris Jacobsen (also from Stony Brook), and described ongoing efforts to extend an existing database of amino acid spectra to oxygen and nitrogen edges.

The workshop concluded with a discussion by the participants of some outstanding issues faced by researchers in the field. The discussion covered a wide range of topics such as: the ability of self-consistent calculations to minimize the need of ad hoc adjustments

to important model parameters; the widespread need for reference libraries, particularly of polymers and amino acids; the limited applicability of the widely used "gaussian+arctan" deconvolution of near-edge structure, particularly in inorganic systems where effects are often very long-range in nature; factors that determine the

temperature dependence of near-edge spectra (or the lack thereof); and the dependence of the detailed shape of soft x-ray near-edge spectra on the experimental setup, particularly when electron or fluorescence detection is used, and its implications for the ability to quantitatively analyze such spectra. ■

Infrared Synchrotron Radiation and Applications

Lawrence Carr
(Brookhaven National Laboratory, NSLS)

This workshop was held on May 18, 1998 at the NSLS and attended by approximately 60 people from more than 25 different institutions. Gwyn Williams (NSLS) began by reviewing the special characteristics of infrared synchrotron radiation (IRSR), the performance advantages of the NSLS VUV Ring, and the beamlines specifically built to utilize this radiation. Presently two IR beamlines are operational, and a third (for microspectroscopy) is very near completion. The remaining three IR beamlines have all delivered "first light" and beam distribution systems are being constructed. Wayne McKinney (Lawrence Berkeley National Laboratory) described the design and performance of their recently completed IR beamline at the ALS, along with their successful effort to reduce noise. A microspectrometer endstation is operational and already in use for the study of micro-organisms related to bioremediation. The high brightness of synchrotron radiation enables various measurement methods, such as ellipsometry, to be applied to small specimens. Alex Wittlin and collaborators from the Max Planck Institute (Stuttgart, Germany) have developed a custom ellipsometer for use in the far infrared, and have studied the "pseudo-gap" behavior in the c-axis response of small high- T_c oxide crystals. Grazing incidence reflection-absorption spectroscopy is another method that benefits from high brightness. Carol Hirschmugl (U. of Wisconsin - Milwaukee) reviewed the grazing incidence technique available at NSLS beamline U4IR and described two areas

of active research; the study of absorbates on noble metals and thin oxide films on various transition metal surfaces. In the former case, additional insight has been gained into the interaction between various types of absorbates and the conduction electrons in the metal. Perhaps the best illustration of high brightness synchrotron infrared enabling an existing, wide-spread measurement method is microspectroscopy. John Reffner, former director of research at Spectra-Tech, Inc. reviewed microspectrometer instrumentation and pointed out areas where improvements were still available in conjunction with the synchrotron IR source. He remarked that a reduction in detector area could provide better signal-to-noise. One of the earliest examples of microsampling with infrared synchrotron radiation was the work by Rus Hemley and co-workers from the Geophysical Laboratory (Carnegie Institution of Washington). Their program makes use of an IR microscope-style optical system for probing materials inside ultra-high pressure diamond anvil cells. This group continues to investigate hydrogen at high pressure and low temperature for evidence of metallic behavior, as well as other "simple" materials such as ice, for which a new quantum ordered phase has been discovered. IR microsampling and chemical identification of extraterrestrial samples was described by George Flynn (SUNY at Plattsburgh). Interplanetary particles, collected from the Earth's upper atmosphere or extracted from meteorites found on the surface, are typically heterogeneous. Flynn's group uses two different NSLS

x-ray beamlines for elemental and some chemical mapping, as well as the infrared microspectrometer at U4IR for complete chemical identification. The latter allows the identification of both mineral phases and the presence of both simple and more complex (e.g. polycyclic aromatic hydrocarbons) organic compounds. Lisa Miller of the Albert Einstein College of Medicine presented two examples of how synchrotron infrared microspectroscopy can be used to study biological cells and tissues. In her work on diseased bone (e.g. osteoarthritis), microtomed sections of bone tissue are examined for both protein and mineral content. By using an extrinsic photoconductive detector such as Ge:Cu, the microspectroscopy can be readily extended down to 400 cm^{-1} . Thus, bone mineralization can be tracked through both carbonate and phosphate vibrational features, and differences between normal and diseased bone are spatially resolved. The effect of typical cosmetic treatments on the properties of human hair were also shown. This work, by J.-L. Bantignies (ELF Atochem), shows how IR

microspectroscopy can be used to image hair structures (e.g., the cortex and medulla) and detect variations in the water content as a function of hair treatment. The last two presentations addressed applications of the pulsed nature of synchrotron radiation to the field of time-resolved spectroscopy. Most synchrotron sources produce light in pulses shorter than 1 ns in duration. David Tanner (U. of Florida) discussed the U12IR beamline at the NSLS and the synchronized Ti:sapphire laser system that can be used to perform pump-probe spectroscopy. A time resolution of better than 1 ns has been achieved. Initial measurements on semiconductors and superconductors were described, as well as several candidate experimental systems. Richard Palmer (Duke U.) described an interesting time-resolved measurement system proposed for the Duke storage ring based FEL. Time-resolved spectroscopy could be performed in a manner similar to that being performed at the NSLS, namely pump-probe spectroscopy using a synchronized laser as the excitation source. ■

Beamline U4IR: Synchrotron Infrared Microspectroscopy (SIRMS) of Acetone-Induced Pitting of Al Alloy AA2024-T3. The figures show 100 by 100 micron chemical maps from the surface of a copper-containing aluminum aerospace alloy ultrasonically rinsed in acetone followed by distilled water prior to exposure to a salt mist. The image above shows a map of aliphatic hydrocarbon bond deformation surrounding one of many corrosion pits formed on the alloy, and the figure on the right shows carboxyl group bond stretching within the same pit. This work has aided in development of a model of how an accepted surface preparation process leads to photoinduced formation of acetic acid followed by localized corrosion at copper rich precipitates and hence unacceptable corrosion behavior. This work is supported by an AFOSR multiuniversity research initiative on the mechanism of aluminum alloy corrosion and the role of chromate inhibitors.

G. Halada, J.R. Kearns, C.R. Clayton (SUNY Stony Brook), G.L. Carr and G.P. Williams (NSLS)
Collected with Omnic software (Nicolet), plotted with Microcal Origin.

FOUR **OPERATIONS**

OPERATION

VUV RING

Stephen Kramer
VUV Ring Manager

Figure 1 shows the breakdown of the VUV Ring operating statistics for the Fiscal Year 1997. The monthly breakdown of most significant operational performance statistics are presented in Figures 2 through 6. The operational statistics continue to show improvement above the record year of FY 1996. The fraction of the time during the year resulting in unscheduled downtime was only 1.5%. The probability that beam was not available when it was scheduled for operations was only 2.38%. However the total beam time actually delivered to the users was 6 hours greater than was scheduled, or 100.1% of scheduled. This resulted from the early return from maintenance and a reduction in accelerator study hours actually used. The accelerator performance continued to improve with higher injection rates and longer beam lifetimes. Injections are now routinely done in less than 3 minutes with the average approaching 2 minutes. The beam lifetime had been lower due to higher vacuum resulting from the two openings of the ring during the year and due to vacuum leaks. However, from August through the end of the year the lifetime has exceeded the pre-shutdown values by about 8%. This was achieved despite the new gas load introduced by the new U12IR mirror being inserted into the ring vacuum chamber and a leak in the front-ends of two beam ports. To help reduce the impact of these new gas loads, a new beam scrubbing shift (vacuum chamber conditioning shift) was introduced during the owl shift (0:00 to 08:00) of the two day study period that occurs once a month. This shift helps desorb the gas that was introduced by these new sources more rapidly than would occur during normal operations and helps reduce the base pressure of the ring.

The major improvement to the VUV Ring during this fiscal year occurred during the winter shutdown when another large aperture beam port was installed for the U2IR beamline. The ceramic gap just after this beam chamber had started to leak during the fall 1996 and was also replaced during the winter shutdown of the ring. Despite the increased work load from the ceramic gap replacement, the effective planning of the Mechanical Group allowed the ring to be brought on four days early

and scrubbing of the vacuum with beam to take place during the three day holiday weekend. This allowed operations to begin on schedule but with longer beam lifetime resulting from the reduced vacuum pressure. During the spring, a front-end valve started leaking in the half of the ring opened during the winter shutdown. This valve was replaced during the May shutdown requiring the same half of the vacuum chamber to be vented again. Recovery to pre-shutdown values of the beam lifetime required about the same integrated current as the winter shutdown, but since this vacuum work had not been anticipated, much of the beam scrubbing had to be done during operations.

Other improvements in the ring resulted from changes in the damping of higher order mode of the main RF cavity and other changes in this RF control system. Testing began on the implementation of a fast RF feedback system on the main RF system. This system should suppress the noise introduced by that RF system on the beam and it appeared to work quite well when operated by itself. However, when the bunch lengthening RF system was also working the beam showed increased fluctuations and will require more work to allow these two systems to work together. Other improvements were in the diagnostics of the electron and photon beams. New extended dynamic range Beam Position Monitor (BPM) receivers were installed on all of the ring's pickups. This will allow measurements of the beam position down to lower values of current. This of little concern for normal operating currents but will help better understand the electron beam model for the ring by allowing measurements at low beam current. A new fast turn-to-turn BPM measurement system was prototyped and the full system should be operational in FY 1998. This BPM will allow modeling of the non-linear properties of the electron beam. Other diagnostic improvements that are planned will help understand the fast beam fluctuations and how to reduce their impact on the users.

The long awaited improvement in the VUV radiation shielding did not take place during FY 1997, as it was originally planned. This was due to the difficulty in finding suppliers of the heavy concrete shielding blocks.

However, this supply difficulty has been overcome and the shielding is planned to be installed around half of the ring during the winter 1997-1998 shutdown. Once the improvement in the radiation levels in that half of the ring is measured the remainder of the shielding will be scheduled for a future shutdown. When the radiation shielding has been installed new studies of the Top-Off Method of Injection (TOMI) will be performed, in order to demonstrate that TOMI offers the users the ultimate improvement in beam stability over long time periods both from the source and the optics points of view. TOMI will also eliminate the age old conflict between longer beam lifetime and higher brightness of the photon beam. To insure that the frequent injection pulses are not seen by the users, new shorter pulse kickers will be installed in FY 1998 to allow the injected beam to be added to the

stored beam with minimum disturbance to the stored beam.

During the next fiscal year studies will be carried out to increase the operating beam energy of the VUV Ring. This will continue the improvement in beam lifetime that has dominated the past improvements. In addition, studies are being carried out to develop methods of providing for real-time variation of the undulator gaps in the ring. Although this is common place on the high energy rings, the lower energy of the VUV Ring makes the impact of these gap changes more significant for the other users. The initial studies to control the orbit and betatron tunes proved insufficient and additional correction of horizontal-vertical coupling effects also will have to be compensated. ■

Figure 1: The breakdown of the VUV Ring usage based on total time (not scheduled time) for FY 1998.

VUV Ring Time Usage

FY 1998 Ring Performance Statistics

Ave. fill Current: 830 mA
 Ave. Charge Rate: 251 mA/min
 Ave. Lifetime at 500 mA: 283 min
 Total user integrated current:
 3033 A-Hrs (126 A-days)
 Total hours of operation: 5978 hours
 Average operating current: 507 mA

VUV Ring Performance FY 1998

Figure 2: The VUV Ring injection charge rate average over all fills in each month.

Figure 3: The total integrated current for the VUV Ring accumulated each month.

Figure 4: The injection current averaged over all fills in a month for the VUV Ring.

Figure 5: The VUV Ring vacuum pressure at 500 mA beam current averaged over each month.

Figure 6: The VUV Ring exponential beam lifetime at 500 mA beam current (seven bunch operation only) averaged over each month.

VUV STORAGE RING PARAMETERS AS OF NOVEMBER 1998

Normal Operating Energy	0.808 GeV
Peak Operating Current (multibunch ops.)	1.0 amp ($1.06 \times 10^{12}e^-$)
Circumference	51.0 meters
Number of Beam Ports on Dipoles	17
Number of Insertion Devices	2
Maximum Length of Insertion Devices	~ 2.25 meters
$\lambda_c(E_c)$	19.9 Å (622 eV)
B(ρ)	1.41 Tesla (1.91 meters)
Electron Orbital Period	170.2 nanoseconds
Damping Times	$\tau_x = \tau_y = 13$ msec; $\tau_z = 7$ msec
Lifetime @ 200 mA with 52 MHz	360 min
(with 211 MHz Bunch Lengthening)	(590 min)
Lattice Structure (Chasman-Green)	Separated Function, Quad, Doublets
Number of Superperiods	4
Magnet Complement	<div style="display: flex; align-items: center;"> <div style="font-size: 3em; margin-right: 10px;">{</div> <div> 8 Bending (1.5 meters each) 24 Quadrupole (0.3 meters each) 12 Sextupole (0.2 meters each) </div> </div>
Nominal Tunes (ν_x, ν_y)	3.14, 1.26
Momentum Compaction	0.0235
RF Frequency	52.886 MHz
Radiated Power	20.4 kW/amp of Beam
RF Peak Voltage with 52 MHz (with 211 MHz)	80 kV (20kV)
Design RF Power with 52 MHz (with 211 MHz)	50 kW (10 kW)
Synchrotron Tune (ν_s)	0.0018
Natural Energy Spread (σ_e/E)	5.0×10^{-4} , $I_b < 20$ mA
Bunch Length (2σ)	9.7 cm ($I_b < 20$ mA)
(21 _{ms} with 211 MHz Bunch Lengthening)	(36 cm)
Number of RF Buckets	9
Typical Bunch Mode	7
Horizontal Damped Emittance (ϵ_x)	1.62×10^{-7} , meter-radian
Vertical Damped Emittance (ϵ_y)	$\geq 3.5 \times 10^{-10}$ meter-radian (4.0×10^{-9} in normal ops.)*
Power per Horizontal Milliradian (1A)	3.2 Watts

Arc Source Parameters

Betatron Function (β_x, β_y)	1.18 to 2.25 m, 10.26 to 14.21 m
Dispersion Function (η_x, η_y)	0.500 to 0.062 m, 0.743 to 0.093 m
$\alpha_{xy} = -\beta'_{xy}/2$	-0.046 to 1.087, 3.18 to -0.96
$\gamma_{xy} = (1 + \alpha_{xy}^2)/\beta_{xy}$	0.738 to 0.970 m ⁻¹ , 1.083 to 0.135 m ⁻¹
Source Size (σ_x, σ_y)	536 to 568 μ m, >60 to >70 μ m (170-200 μ m in normal ops.)*
Source Divergence (σ_x, σ_y)	686 to 373 μ rad, 19.5 to 6.9 μ rad (55-20 μ rad in normal ops.)*

Insertion Device Parameters

Betatron Function (β_x, β_y)	11.1 m, 5.84 m
Source Size (σ_x, σ_y)	1240 μ m, >45 μ m (220 μ m in normal ops.)*
Source Divergence (σ_x, σ_y)	112 μ rad, >7.7 μ rad (22 μ rad in normal ops.)*

* ϵ_y is adjustable

OPERATIONS

X-RAY RING

Roger Klaffky
X-Ray Ring Manager

During the November 18-23, 1997, 2.8 GeV operations period and throughout the December shutdown, there was continued progress towards completion of 2.8 GeV beamline shielding. Before the March/April 2.8 GeV operations period, studies shifts were set aside so that all lines could 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 deionizer 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° F or if the power supply water exceeds 115° 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 were three Be windows that still needed to be replaced: X10B, X11A, and X22C. New safety shutters rated for operation up to 500 mA were installed in the X21 and X25 frontends. The final requirement remaining for high current operation was that a simulation

X-Ray Ring Time Usage

The breakdown of the X-Ray Ring usage based on total time (not scheduled time) for FY 1998.

The total integrated current for the X-Ray Ring accumulated each month for FY 1998.

and testing be completed to confirm a lifetime projection for the 10 degree crotch assembly. In preparation for the Spring 1998 shutdown, a 1.5 foot deep hole was cut through the X-ray Ring shield wall for the installation of a transmission line for a new RF cavity. The new hole was required because the RF1 and RF2 cavities have to be split to provide room for the possible future installation of another IVUN.

During the Spring 1998 shutdown, a new all-copper cavity was installed replacing the existing RF2 cavity. This system is unique, having two 125 kW amplifiers driving a hybrid combiner capable of supplying in excess of 160 kW to the cavity and beam. The original cavity and drive loop limited this power to 100 kW. A new all-copper drive loop with improved cooling and a refined window design was also installed and tested to 150 kW continuous input power. Prior to installation the cavity underwent extensive testing in the NSLS RF test room. The cavity was baked and achieved a pressure of 1×10^{-9} Torr while being powered to 60 kW. The new window was tested at power levels of 120-150 kW. Running at 150 kW will supply another 60 kW to the beam and allow the system to run more reliably at 350 mA with three transmitters if one system trips. The cavity has also run to 65 kW, and when in operation will increase the total voltage gradient to the beam, improving reliability when operating at 2.8 GeV. The new cavity has improved cooling and superior internal surfaces which will result in better vacuum after conditioning. All higher harmonic modes were damped with antennae to levels lower than the other RF cavities. The RF resonant frequency was successfully corrected by replacing the large Helicoflex vacuum seal with a larger cross-section seal.

Another major task planned for the Spring 1998 shutdown was the installation of the new Oxford Instruments X17 superconducting wiggler. In preparation for the 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 was installed for the power supplies and water cooling will be hooked up to these supplies. The new wiggler installation had to be delayed due to problems encountered during acceptance tests at BNL. Two specific issues were identified as the new wiggler was being prepared for cold tests. The first problem was a leak between the helium and nitrogen vessels in the wiggler. The other defect was an electrical short. Both of these discoveries were cause for rejection. After consultation with the manufacturer, a series of electrical checks was performed in an attempt to pinpoint the electrical short

and to determine if the two defects were related. At that point all agreed that repairs on site at BNL were not feasible and arrangements were made to ship the wiggler back to the UK for further evaluation and repair.

The X17 straight section chamber downstream of the wiggler was replaced during the Spring shutdown because a water-to-vacuum leak had 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, and climbed into the 5×10^{-7} Torr region in December 1997. 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. Because there was no water cooling, the wiggler had to be turned off until after the chamber replacement occurred.

The beryllium window replacement program, which was begun in the Winter 96 shutdown, was completed during the Spring shutdown. During this time, the upstream-most beryllium windows in beamlines X-10B, X11A and X22C were replaced, bringing the total number of beamlines upgraded to 38. Consequently, all NSLS X-ray beamlines became compatible with ring operation up to and including 438 mAmps at 2.584 GeV or 300 mAmps at 2.8 GeV.

There were a number of interlock tasks completed during the Spring 98 shutdown. The X14A beamline security system was converted from a Phase I system to a Phase II modular system and a Phase II system was installed on the new X14B beamline. Fast valve electronics were installed for the X10, X13, X14, X15, X16, and X23 front ends. To render these systems operational, their logic systems will be modified (so the X-ray Ring will dump if they trip) or molybdenum fast valve blades will be installed. The installation of cables for the Critical Device Overtemperature System (CDOS) took place bringing this system closer to completion. X-ray Ring tunnel emergency stops were inspected and rewired where required.

A number of electrical power distribution tasks were also completed during this shutdown. A power panel (SP8) inside the ring was moved to a more accessible location. When the last panel is moved during the upcoming Winter shutdown, all power panels inside the ring will have been relocated. Emergency power was hooked up to X-ray, UV and Booster micros, to back up the existing UPS systems, and to the Biology Cold Rooms.

The deionizer pumps for the NSLS machine water systems were moved to a more accessible location. New

hosing and proteus units were installed on different water-cooled X-ray Ring components, as a part of the maintenance program.

After the Spring shutdown, commissioning of the X-Ray Ring went smoothly. The new RF system parameters were adjusted to restore routine injection to 350 mA. The X17 Active Interlock system was recertified for the new X17 chamber PUEs downstream of the wiggler. The chamber conditioned rapidly with the wiggler at 4.7 Tesla.

To track the horizontal motion of the X-ray ring chambers, sensor stands will be installed and data acquired during operations from high sensitivity LVDT probes

mounted on the stands at various PUE locations. Ultimately, data acquired from the horizontal sensors measuring the ring chamber motion at each of the 48 PUEs during X-ray fills, will be input to the digital feedback system to correct for this motion. Carbon fiber tubes for the sensor stands arrived in March 1998. Prototype stands were built and tested for thermal stability.

Studies of the low emittance lattice indicated that additional sextupole strength was required to operate with the low emittance lattice at 2.8 GeV. Operations with the low emittance lattice commenced on September 1998 at 2.584 GeV. ■

X-RAY STORAGE RING PARAMETERS AS OF SEPTEMBER 1, 1998

Normal Operating Energies	2.584 GeV/2.800 GeV
Maximum Operating Current	0.35/0.25 (10^{12} e-)
Lifetime	~20 hours
Circumference	170.1 meters
Number of Beam Ports on Dipoles	30
Number of Insertion Devices	5
Maximum Length of Insertion Devices	< 4.50 meters
$\lambda_c(E_e)$ at 1.25/1.36 T	2.23 Å (5.6 keV)/1.75 Å (7.1 keV)
$\lambda_c(E_e)$ at 5.0 T (W)	0.56 Å (22.2 keV)/0.48 Å (26.1 keV)
B(ρ)	1.25/1.36 Tesla (6.875 meters)
Electron Orbital Period	567.2 nanoseconds
Damping Times	$\tau_x = \tau_y = 6$ msec; $\tau_z = 3$ msec/ $\tau_x = \tau_y = 4$ msec; $\tau_z = 2$ msec
Touschek (0.25A)	≥ 22 hrs ($v_{RF} = 804$ kV)/ 57 hrs. (1120 kV)
Lattice Structure (Chasman-Green)	Separated Function, Quad Triplets
Number of Superperiods	8
Magnet Complement	<div style="display: inline-block; vertical-align: middle;"> <div style="font-size: 3em; vertical-align: middle; margin-right: 0.5em;">{</div> <div> 16 Bending (2.7 meters each) 40 Quadrupole (0.45 meters each) 16 Quadrupole (0.80 meters each) </div> </div>
32 Sextupole	(0.20 meters each)
Nominal Tunes (v_x, v_y)	9.15, 6.20
Momentum Compaction	0.0056/0.0062
RF Frequency	52.88 MHz
Radiated Power for Bending Magnets	144/198 kW (0.25A)
RF Peak Voltage	804/1120 kV
Design RF Power	400 kW
v_s (Synchrotron Tune)	0.003
Natural Energy Spread (σ_e/E)	$8.6 \times 10^{-4}/9.2 \times 10^{-4}$
Natural Bunch Length (2σ)	10.5 cm
Number of RF Buckets	30
Typical Bunch Mode	25
Horizontal Damped Emittance (ϵ_x)	$7.2 \times 10^{-8}/7.8 \times 10^{-8}$ meter-radian
Vertical Damped Emittance (ϵ_y)	$7.2 \times 10^{-11}/7.8 \times 10^{-11}$ meter-radian
Power per Horizontal Milliradian (0.25A)	23/32

Arc Source Parameters

Betatron Function (β_x, β_y)	1.0 to 3.8 m, 7.9 to 26.5 m
Dispersion Function (η_x, η_y)	0.47 to -0.11, -0.39 to 0.22
$\alpha_{xy} = -\beta'_{xy}/2$	-0.49 to 1.62, -3.4 to 4.5
$\gamma_{xy} = (1 + \alpha_{xy}^2)/\beta_{xy}$	0.952 to 0.962 m ⁻¹ , 0.81 to 0.52 m ⁻¹
Source Size (σ_x, σ_y)	371 to 612 μ m, 27 to 53 μ m
Source Divergence (σ_x, σ_y)	476 to 324 μ rad, 9 to 7 μ rad

Insertion Device Parameters

Betatron Function (β_x, β_y)	1.60 m, 0.35 m
Source Divergence (σ_x, σ_y)	260 μ rad, 35 μ rad

OPERATIONS

BEAMLINE TECHNICAL IMPROVEMENTS

Roger Klaffky

Beamline Technical Liaison

VUV BEAMLINES

There were a number of technical VUV beamline improvements during FY 1998. In preparation for the start of user operations on the U2B beamline, an infrared microscope was purchased which, when utilizing the NSLS as a source, will perform with a 1000-fold improvement over the commercial instrument.

Recommissioning of the U3A beamline continued with special emphasis on the beam requirements for Advanced X-ray Astrophysics Facility (AXAF) X-ray mirror reflectivity calibrations in the 1000-2000 eV energy range. These requirements include adequate stability for arcsecond alignment, a sharp beam profile, less than 0.1 % beam impurity and adequate flux for energies up to 2000 eV. Monochromator upgrades continued with implementation of lookup table based tracking of monochromator crystal elements. Periodic error corrections for both crystal rotary tables were calibrated and energy scanning algorithms based on lookup table tracking were defined, debugged and implemented in a LabView monochromator control program. Adequate tracking was demonstrated using Na- β alumina crystals for the 800-2100 eV range. Scraper slits were installed downstream of the horizontally-focusing mirror to achieve a sufficiently sharp beam profile. The stability, alignment characteristics, spectral purity, and flux characteristics for the beam in the 1-2 keV range were determined to be adequate for the AXAF calibration program.

Design of the U3B diagnostic beamline was completed and bids were received for fabrication and polishing of the critical water-cooled mirror. Construction was underway on the mirror vacuum chamber and beamline support structure. The U3B beamline will provide a previously unavailable energy measurement capability for the electron beam in the VUV storage ring.

On U3C a four-reflection high order trap (HOT) developed by Hettrick Scientific was tested in order to reduce photons outside the desired bandwidth to less than 1% for instrument calibration. This system has six sets of traps covering different spectral regions up to 540 eV, and provides beam purities greater than 99% below 250 eV. A HOT optimized for U3C was designed with the intention of installing it in FY99.

As part of the DOE Facilities Initiative, spherical gratings and a moveable slit will be added to the U4A beamline, replacing toroidal gratings and fixed exit slits. The ARP chamber will be fitted with a sample transfer and load lock system. Plans for these improvements were finalized with installation expected in FY99.

In order to complete construction of the U7A soft X-ray materials characterization facility, the former facility was completely dismantled and reconstructed. The new facility incorporates a refocusing mirror (commissioned in FY98) which produces a sub-millimeter spot at the sample position. The refocusing mirror also provides a rapid means of switching the soft X-ray beam from the U7A monochromator between the surface science end station and the materials end station. Also, a sample manipulator and preparation/load lock chamber were installed which allow rapid sample entry as well as catalyst calcining and pretreating under atmospheric pressures. The new sample manipulator allows multiple (30) samples to run in an automated fashion.

The U9A NSLS storage ring vacuum R&D beamline was completed in FY98. Photon-stimulated desorption measurements were carried out on various vacuum surfaces, including KEK B-factory chambers.

To optimize signals on the U9B fluorescence spectrometer, an xyz sample positioning system was installed with more than 1 cm travel in each direction. Also, a new photomultiplier was installed to detect the

intensity of light passing through the samples that are being simultaneously monitored for fluorescence. The process of upgrading the data acquisition system was underway with the purchase of new control software for the multiparameter analyzer.

The U10A infrared beamline neared completion with the installation of the front end mirror box and a diamond window to separate the UHV front end vacuum from the rough vacuum (or purge gas) used in the instruments. The Bruker IFS66v/S infrared Fourier transform vacuum interferometer was installed on the U10A beamline. Two reflectance units have been built for the interferometer. The first allows the use of the detector area inside the spectrometer. The second unit employs a single reflection whereby light from the sample is collected by a large mirror and then focused into an external detector. This arrangement is used almost exclusively for large, liquid-helium cooled detectors which must be located outside of the spectrometer.

The U11 beamline was used for photoionization mass spectrometry (PIMS), high resolution angle-resolved photoemission spectroscopy, and absorption spectroscopy of gases. The biology chamber quartz lens used to collect fluorescence and focus it onto the entrance slit of an emission monochromator was replaced by a custom-built off-axis toroidal mirror, thereby extending the wavelength range and improving efficiency by eliminating chromatic aberration. Also, a 1.5 Tesla room temperature electromagnet with optical access through the iron pole pieces was in the process of being fit to operate at U11 for magnetic circular dichroism experiments.

During FY 98 all major components for the U12A IR beamline were procured with installation of the optical components and completion of the beamline vacuum envelope expected by the end of 1998. Final alignment and commissioning is slated for early in 1999.

The U13UB beamline is devoted to UV/VUV spectroscopy in the 5-30eV range. The U13 undulator/wiggler source can be directed to either: (1) a focused white light branch with variable (0-10ns) delay, or (2) a high resolution ($h\nu/\Delta h\nu > 10^4$) monochromatic branch. The monochromatic branch, which contains the bulk of the mechanical and optical components of the entire beamline, became fully operational in June 1998.

During FY98 the U15 beamline was used primarily for studies of soft X-ray excited luminescence in doped nanocrystals. To conduct these studies changes were made to the U15 main chamber, and pumping systems. The TGM monochromator motor control unit was recommissioned. The U16B beamline was restored to an operating condition and made available to General Users during FY98.

X-RAY BEAMLINES

The first tests of a new X1A scanning transmission X-ray microscope were carried out in FY98. This microscope has kinematic mounts for exchange of its optics, and a motorized detector platform to allow rapid exchange between different detectors, and positioning of configured detectors by taking scanned images of detector alignment. In its first test, these capabilities were used to demonstrate (in collaboration with Denis Joyeux of Institut d'Optique and Francois Polack of LURE, France) Nomarski phase contrast in a scanning microscope. The successful test of the basic components of the microscope paves the way for implementation of the system in a sealed chamber for spectromicroscopy at the nitrogen and oxygen edges.

On the X1B beamline, new refocusing optics were installed and aligned. These changes should facilitate experiments requiring high brightness such as those using the soft X-ray spectrometers.

On the X3 beamline there were a number of upgrades. The X3 beamline equipment protection interlock was modified to include a front valve which was previously unprotected. The X3A1 cryogenic system was repaired to eliminate helium leaks and modified to allow continuous operation during recharge. A new 1000x1000 element CCD detector was installed in May 1998 which can be used on both the A1 and A2 stations. Progress was made in writing new diffractometer-control software in coordination with APS staff.

An Raxis-IV imaging plate detector was put into operation on the X4A beamline during FY98. The automation of the scanner resulted in a large improvement for users. A further improvement will be made in 1999 when a CCD detector is installed to reduce the turnaround time between exposures. On the X4C beamline 1.5 horizontal mradians of beam was focussed into 0.42 mm(FWHM) on the experimental table. An ADSC single cell CCD detector system was purchased and integrated with Windows driven PC software and preliminary debugging of the motion control system took place.

Further progress occurred in FY98 towards running strongly polarized hydrogen-deuteride ice (SPHICE) targets at the X5 LEGS beamline. There were 3 major target production runs with the BNL dilution refrigerator and 17 Tesla magnet system presently at Syracuse University. Full size (3cm dia. x 5 cm long) HD targets with over 50% H polarization were produced and successfully extracted from the dilution refrigerator. After the first round of experiments has been completed, the SPHICE target factory will be moved to the NSLS and recommissioned in a new lab adjacent to the LEGS

beamline. To facilitate the production of SPHICE targets a helium liquefier was obtained from the University of Virginia. Work also took place on the construction of a central drift chamber to expand the spin asymmetry(SASY) calorimeter capabilities for charged particles (p, π^+, π^-). Final design and engineering work began and construction of several components was underway at several of the LEGS collaborating institutions. A prototype of the time projection chamber (TPC) trigger scintillator was constructed and successfully tested. A major beamline upgrade was implemented providing a new front-end transport system capable of switching between an existing Ar- Ion laser and a new frequency-quadrupled Neodymium-Yttrium-Fluoride(Nd-YLF) ring-laser system. The combination of this new ring laser and the recent increase in the storage ring energy to 2.8 GeV has increased LEGS gamma-ray energies to 470 MeV. The first 470 MeV beam was observed in April 1998.

Stimulated by an effort to develop a facility to perform time-resolved in-situ measurements of catalytically-active materials, several changes were implemented at X7B during FY98. An externally exhausting fume hood was installed to vent both the hutch and a commercial gas cylinder cabinet, in order to satisfy safety standards for gas flow and reactivity experiments. To reduce heat loading of the monochromator crystal, graphite wafers were inserted in the beam-defining slit package located upstream of the premirror position. A new portable detector/sample trolley system was designed and constructed. The heart of the system is a MAR 345 automated detector system based on image plate technology. The system makes it possible to adjust the sample to detector separation over more than a meter with 50 micron accuracy. The detector system can also be moved above and below the plane of the incident beam by 30 degrees with 0.01 degree accuracy. The entire assembly sits on a kinematic mounting system that makes alignment of the detector system simple and reproducible. Finally, A Labview based NSLS DAC beamline control system was implemented during the second half of FY98.

Operations expanded on beamline as it became a dedicated station for X-ray footprinting. A dedicated stopped-flow mixing device was constructed and put into operation. This device enabled experiments on a 10-20 millisecond or longer timescales. The efficiency of operation of the X9B X-ray spectroscopy beamline was enhanced with software and hardware advances in beamline control. There were improvements to the AUTOFIT1.2 package which allows the use of advanced ab initio XAS codes by users understanding structural analysis.

At X10A and X10B a new two-dimensional CCD detector(Bruker Smart 1500) was purchased to enhance real time measurements of small-angle and wide-angle scattering. A new data acquisition and analyzing system was also scheduled for installation. A Siemens FSD scintillation counter was also purchased for use at X10A and X10B. This detector handles count rates up to 10 counts per second, permitting some experiments to be performed without attenuation. A new oven was constructed for polymer stretching in order to investigate the deformation process of polymers at elevated temperatures. Conversion of the VAX beamline control system to a LINUX system was also underway.

There were a number of upgrades and replacements on the X11A beamline in the past year. The operating system on all the PRT computers was upgraded and a macro command utility was added to the data collection software to allow automatic data collection as a function of sample position, temperature, etc. The 13-element Canberra detector was serviced to optimize its performance. Commissioning and testing of a sagittal focussing system continued. A new computer controlled slit assembly was installed. A new collimating mirror was received and found to meet specifications when measured at the BNL Instrumentation Division. Design of a mirror support mechanism began.

The X13A SGM instrument was commissioned with an existing grating. Two new gratings were procured to make use of the EPW for magnetic spectroscopy development. A new crystal monochromator was developed for X13B for use with the IVUN and EPW.

A new beryllium window with two ports was installed at X15A to provide a 5 inch wide white beam with a 1 mm thick aluminum filter for diffraction-enhanced imaging (DEI), and a 1 inch wide white beam for X-ray standing wave (XSW) experiments. The hutch and beamline were reshielded for 2.8 GeV operation. A rail system was installed under the XSW chamber to facilitate switching from the XSW mode to the DEI mode.

At X15B design, construction, and full commissioning of a new room-pressure sample chamber and hutch box took place. The upgraded chamber, which complements the existing high vacuum(10^{-6} - 10^{-9} Torr) and ultra-high-vacuum (10^{-10} - 10^{-12} Torr) chambers, allows transmission or fluorescence X-ray absorption measurements in air or controlled atmosphere at room or cryogenic ($\lesssim 10$ K) temperatures.

At X17B1, the beamline control system was upgraded by replacing the old VAX computer with a PC Linux platform running a standard software, SPEC. A 2048 by 2048 real-time CCD detector with 65 micron resolution was used scattering experiments using high energy X-rays of up to 90 keV.

A new optical lab for pressure measurement was set up near the X17C beamline. It will develop into a new Raman spectroscopy experimental lab in the future. A new electric discharge drilling machine was set up in the lab to enable users to drill rhenium gaskets for the diamond anvil cell. A new Dell XPS R 400 workstation was acquired for data analysis by users. In the future this computer will replace the micro VAX beamline control computer.

At X18B the experimental hutch was enlarged and cable feedthroughs installed to permit the use of a larger array of experimental equipment, including solid state detectors. An additional fluorescent screen was installed in the beampipe to facilitate alignment. The data collection software was upgraded. A small device was fabricated to collect a small part of the incident monochromatic beam and reflect from a standard metal sample to provide a reference spectra for thick or supported samples used for fluorescence XAS.

The most significant beamline upgrade at X19A was the replacement of the damaged zerodur mirror with a new silicon mirror. A factor of 10 improvement in throughput resulted with a significant decrease in the focused beam size. Additional diagnostics were installed for alignment purposes. At X19C an analyzer axis for the detector arm was designed and implemented. This enables detailed analysis of harmonic contamination on white beam topographs.

On the X20 beamlines filtering transformers were installed for the protection of the more control electronics and beamline equipment safety systems. Also, new stepping motor controllers were installed for hutch and diffractometer motors. Unshielded ribbon cables were replaced with shielded ones and capacitance filtering was installed on the E500 system for the new controllers. At X20B New Focus Picomotors and a controller were purchased for the tilt and bend of the monochromator crystal. Design of a vertical focussing mirror began. It will intercept approximately 2mm of the beam and give a 50 micron vertical FWHM spot at the sample position. The mirror should provide a factor of 13 increase in brightness and eliminate harmonic contamination. An IBM RS/6000 Model 590 computer was installed and upgraded to AIX Version 4.2. This will serve as a fast data analysis machine, primarily for the in-situ phase transformation studies done in real time at X20C.

At X21 commissioning and operation of a 7-element analyzer took place.

In addition to normal maintenance at the X22 beamlines, there were a number of upgrades. At X22A a new Pt-coated Si focussing mirror was installed. Also, Soller slits were upgraded for electrochemistry

experiments. At X22B a new Pt-coated Si focussing mirror was also installed, the liquid surface spectrometer was upgraded and a new UHV chamber for liquid metal surface studies was completed. At X22C a new Pt-coated Si focussing mirror was returned to the manufacturer for repolishing because its roughness exceeded specifications. An azimuthal rotation stage was fabricated for use on the Franke-Heydrich spectrometer in studies of resonant orbital ordering.

At X23A2 an energy discriminating SiLi fluorescence detector was installed. Also, the data acquisition system was converted to a LabView based system to record DAFS data.

At X23B problems with the collimating mirror were settled and the beamline operated with good flux and excellent energy resolution. This allowed minor problems with the monochromator to be uncovered and fixed. The beamline computer was upgraded to a Power Macintosh to increase the speed and to take advantage of the full capabilities of the NSLS-DAC software.

At X24A a new UHV chamber for PRT members and general users was brought into operation. The chamber will have a hemispherical analyzer and precision manipulator for surface science experiments. Modifications were made to the downstream optical slit assembly to permit the use of a feedback control of the X-ray beam position over a limited range at the experiment. The position control is desirable for experiments such as EXAFS measurements or electron spectroscopy.

New toroidal collimating and focussing mirrors were received which should improve resolution at X24C by at least a factor of two and increase flux at energies above 1000 eV. The new mirrors will replace old paraboloidal mirrors that had an effective roughness of 3 nm rms and a figure slope error of about 20 microradians rms. The surface roughness of the new mirrors is 0.5 nm rms or less, and the slope errors are within 4 microradians of circular.

A new front end safety shutter was installed to permit X25 to operate at currents in excess of 350 mA (at 2.584 GeV).

At the X27A X-ray Computed Microtomography beamline, the computational speed of the reconstruction algorithm was improved by more than 10 times in FY 98. Implementation of a parallelized Pentium farm provided another factor of 8 or better improvement in reconstruction speed. More alignment and data acquisition functions were automated in 1998 for user-friendly operations. Multilayers in the X27A monochromator were replaced, as well as the Be window on the output port of the monochromator. ■

Beamline X10A: This study details the morphological development and molecular orientation of linear low-density polyethylene (LLDPE) *blown films*. In the blown film process, crystallization takes place under stress and temperature gradient resulting in an anisotropic distribution of the lamellae crystallite. We use a combination of small and wide-angle x-ray scattering to investigate the detail morphological feature and orientation of a metallocene ethylene-hexene copolymer (density $\rho=0.918\text{g/cm}^3$) blown films. The film exhibits an orthorhombic crystalline structure with two oriented lamellae, as shown in the figures. The first type is the commonly observed row-nucleated lamellae whose normal lies in the machine direction. The in-plane order in these lamellae are completely random. The second type of lamellae have their normal perpendicular to the machine direction and accounts for ca. 25% of the total crystallite. These lamellae has preferred crystalline orientation with the crystalline α - and b -axis aligned parallel and perpendicular to the machine direction. These results imply that two different crystallization mechanisms take place during the film blowing process.

H. Shao, M. Rabeony, and K S. Liang (Exxon)

FIVE **PROJECTS**

x

y

z

PROMISE

THE DEEP ULTRA-VIOLET FREE ELECTRON LASER (DUV-FEL)

Erik D. Johnson
DUV-FEL Project Manager

It is certainly no secret that *the* activity at the source development laboratory has become the Deep Ultra-Violet Free Electron Laser (DUV-FEL). Adopted as one of four BNL initiatives, the DUV-FEL has been the focus of considerable activity in the last year. The objective of the program is to provide a sound technological foundation for sources and science that will lead to the next generation of synchrotron radiation based research. BNL shares the view with many other laboratories that these machines will be Free Electron Lasers, and that getting to short wavelengths will require single pass configurations.

The BNL effort has, from its inception, held the view that to be useful experimental tools these machines must wed the best aspects of solid state lasers with accelerator technology. The source must have the characteristics of stability and optical quality people have come to expect in laser technology, while operating at wavelengths that are only readily accessible using accelerator based amplifiers. The High Gain Harmonic Generation (HG) approach proposed by Yu *et al.* is the central concept for the DUV-FEL.

A photon beam from a solid state laser is made to couple with an electron beam in a short "energy

Photo A: (from left to right) Richard Heese, Erik Johnson and Bill Graves reveal the secret location of the DUV-FEL!

modulation" undulator. This introduces an energy modulation in the electron bunch that is converted to a spatial modulation in a magnetic dispersion section. The electron beam thus has a micro-bunching imposed on it which has characteristics related to the seed laser. A second longer "amplification" undulator is used to produce coherent radiation at either the fundamental (same wavelength as the laser and microbunching) or a higher harmonic (shorter wavelength than the seed laser). In the HGHG scheme, the stability properties of the laser (central wavelength, bandwidth, pulse length) are translated into the FEL output, while the accelerator effectively acts as an (optical) dispersion-free harmonic generator and amplifier. In principle the scheme provides exactly what we desire in a fourth generation source. In practice, this all has to be proved experimentally, which is one of the main goals of the DUV-FEL project.

To make this project possible, several years ago the NSLS collected together an assortment of recovered equipment that could be used to assemble the experiment. The Source Development Laboratory (building 729) houses the linac from the x-ray lithography project, and a long undulator (NISUS) originally built for directed energy weapons research. For our DUV-FEL project, the linac must become a high brightness machine with a photocathode gun as an electron source. To operate in

HGHG mode the DUV-FEL needs a flexible laser system not only to drive the gun, but to provide the seed beam for the FEL. The demands on the laser are sufficiently stringent that it was built specifically for this application.

The transformation of the linac from a ring injector to an FEL driver has been guided during the last year by Richard Heese and Bill Graves, and accomplished by a great deal of hard work from people all over the NSLS. This activity includes some fairly basic infrastructure as well as the novel technical developments one might anticipate in such an endeavor. For example, precise temperature control of the linac is required. In fact it runs at 45°C, while accessory cooling for power supplies must be provided. The design of this system was performed by the NSLS utilities group and installation was accomplished with Plant Engineering personnel (see Photo B).

To get its "Cinderella" treatment in moving from ring injector to FEL driver, the linac had to be totally reconfigured. This included the installation of a photocathode gun by Xijie Wang based on his work at the Accelerator Test Facility. Another major improvement is the addition of a pulse compression chicane system developed by Bill Graves to increase the peak current of the electron beam, which will significantly improve the FEL performance. This involves an enormous amount

Photo B: Some of the people responsible for the installation and operation of the process water system for the DUV-FEL. Clockwise from left, Mike Ponticiello and John Fogus (Plant Engineering), Mike Schwartz, Ron Beaman, and Lenny Santangelo (NSLS Utilities).

Photo C: Part of the crew responsible for the mechanical design and installation of the DUV-FEL linac system. From front on the left of the beam dump, Mike Radulescu, Mal Tardd, and John Skaritka. Working from back to front on the right side of the beam dump, Pete DeToll, Chris Stelmach, Bob Scheuerer and Skip Thomas. Working left to right in front, Jeff Aspenleiter, Dick Wiseman (hand on transit), Mike Caruso and Bill Bambina. In back (left to right) Sal Pjerov, Peter Gross and Roger Hubbard.

of mechanical work in design, fabrication and installation. Only a handful of the people who have worked on the machine thus far were available for the picture given here (Photo C).

A key element of our project is the integration of laser science into the FEL. Lou DiMauro from the BNL Chemistry Department has been leading this effort for the DUV-FEL. The laser we selected is a Titanium Sapphire system with a regenerative amplifier. It is a chirped pulse amplification system that produces adequate power for running the photocathode gun and for seeding the FEL. The chirped pulse can in principle be used to produce a chirped pulse amplified (CPA) FEL. Simulations show that pulses the order of 10 femtoseconds can be generated with pulse energies of nearly a millijoule

in the deep ultra-violet. Apart from the technological development of the laser system, extensions of laser based science now practiced in the laboratory need to work their way into the machine and the science it is intended to serve. This has been part of our project philosophy from the start, and we expect it to be important in making FELs genuinely useful for research. Part of the team working with Lou on the laser systems is shown in Photo D.

Of course once you have brought the components together, you have to connect them, condition them, and make them work. Reconfiguring the linac has been as much an electronics and control task as a mechanical task. During the past year, the linac computer control system was entirely revamped to use the same type of software as

PhotoD: Part of the laser development group in the laser lab (clockwise from left) Brian Sheehy (Chemistry), Todd Clatterbuck (SUNY SB), Paul Montanez (NSLS), and on the wallpaper here for the first time (virtual) Lou DiMauro (Chemistry).

Photo E: Some of the people who make the linac go; Clockwise from left Jim Garrison, Jeff Rothman, Richard Heese, Bill Graves, Charles Nielson, Kate Feng-Berman and Boyzie Singh.

the NSLS machines. The electrical systems have been brought up to current codes, and extensive modifications to the low level RF drive system and interlocks were performed to enhance their reliability. Finding and fixing each bug as it crops up is a demanding task that requires perseverance and experience. Fortunately the NSLS has broad experience in this area as exemplified by the people in Photo E. At the time of writing, the system has been powered up and high power RF conditioning of the accelerating sections has been started.

During the next year, the plans include bringing the linac into full operation, and preparing our undulator for conducting the first FEL experiments with the DUV-FEL. Initially we will start running as a self amplified spontaneous emission (SASE) FEL in the visible. In this mode, a high quality electron beam passes through the undulator and starts up from noise. From an experimenter's viewpoint, what you get is essentially loud noise at the end, but it is a stringent test of the electron beam quality and is less complex to operate than a seeded beam FEL. As technological achievements allow, we will move as rapidly as possible to seeded beam operation and to shorter wavelengths.

The development of Fourth Generation Sources is becoming an important priority for the DOE. Many of the proposed experiments nation wide, including parts of the DUV-FEL, are being conducted in collaboration with scientists from universities and other national laboratories. The DUV-FEL project has progressed to a state of development where it will be the focus of major activity in this area over the next several years, and we hope, in establishing the basis for future source technology and science. ■

Map of the SDL, marked with the locations of photos taken. (A through E).

PROJECT

THE ACCELERATOR TEST FACILITY

Ilan Ben-Zvi
ATF Head

The interest in Free-Electron Lasers operating in very short wavelengths, down to hard x-rays, is growing.

A key aspect of the mission of Brookhaven National Laboratory (BNL) is to construct, operate and use large facilities for the breadth of scientific disciplines supported by the Department of Energy. An important component of the success of BNL comes from innovative accelerator R&D, since new accelerator technologies hold the key to new achievements in applications and user facilities. We are now facing the emergence of a new technology: very high-brightness electron beams. This technology has far-reaching implications for x-ray sources and other applications.

Part of the NSLS' mission is to develop radiation sources for the future. The NSLS has been the first light source to pursue the development of Free-Electron Lasers (FELs) as the next generation synchrotron light sources. BNL has been a leader in the national effort on FEL development, hosting the workshops "Prospects of a 1 Å FEL" in 1990 and "Towards Short Wavelength FELs" in 1993, as well as developing its Conceptual Design Report and pursuing subsequent efforts to develop a national collaboration in the field. In the past decade, the NSLS made significant contributions to the science of FELs such as the theories on Universal Scaling of exponential regime FELs in 3-D, the High-Gain Harmonic Generation and beam conditioning. Experimentally we have led in the development of record high-brightness electron beams and methods of measurements of these and the shortest wavelength measurement of Self Amplified Spontaneous Emission (SASE).

The NSLS developed a vision of the next generation of light sources and is assembling the beam physics and technology to make this happen. Emphasizing single-pass FEL amplifiers, which have advantages over oscillators in the short wavelength regime where cavity mirrors are unavailable, the NSLS developed the High-Gain Harmonic-Generation (HGHG) FEL approach as the best strategy to build a high quality short wavelength FEL. High-brightness electron beam R&D was undertaken in a major way, since this is the key to achieving short-

wavelength FELs as well as other significant applications, such as linear colliders, laser accelerators and more. The BNL Accelerator Test Facility (ATF) is the proving ground for these advances.

What is the ATF? First and foremost, it is a user facility for accelerator and beam physicists, operated by the NSLS and the BNL Center for Accelerator Physics. There is no other proposal-driven, peer-reviewed facility like the ATF that is dedicated for long range R&D in accelerator and beam physics.

The ATF has a unique combination of a high-brightness electron beam, synchronized-high-power lasers, a well-equipped 3 beamline experiment hall, and advanced diagnostics and control systems. The ATF's program in RF guns is recognized internationally. With its synchronized lasers and electron beams of unprecedented brightness, the ATF is an ideal site for R&D on advanced accelerator concepts, FELs, femtosecond X-ray sources and similar topics. These tools have been crucial to recent ATF record achievements: the measurement of Self Amplified Spontaneous Emission (SASE) at 1 μm and 0.63 μm , and laser acceleration by the Inverse Cerenkov and Inverse FEL mechanisms.

The generation and acceleration of very high brightness electron beams is a key technology for short wavelength FELs as well as other applications, including linear colliders, Compton backscattering for the production of femtosecond x-rays, laser accelerators and more. A high brightness means that the electron bunch has a high density in 6-D phase space. To achieve high brightness beams, it is necessary to do the following: 1) Master the production of such beams in special electron guns. 2) Develop diagnostics that provide information of the 6-D distribution of electron bunches on sub-picosecond time scales. 3) Control the 6-D distribution of the bunch in various ways. 4) Accelerate the electrons to high energies without diluting the brightness.

NSLS scientists working at the ATF measured the slice emittance of a 10 ps electron bunch with a 1 ps resolution, achieved a record high 6-D electron phase-space density and directly measured electron bunching

on an optical scale. Another diagnostic under development at the ATF is tomographic analysis of the distribution of electrons in transverse phase space. The next step is to pursue non-linear emittance compensation. Laser photocathode RF guns have provided a major improvement in the brightness, which was further enhanced by the introduction of (linear) emittance compensation. The dream of another major improvement by the introduction of non-linear corrections has been brought within reach by the development of the slice-emittance diagnostic and phase space tomography.

Key to our plans is the development of sub-harmonically seeded FELs in which harmonic generation converts a laser seed to much shorter wavelength radiation. A proof-of-principle High-Gain Harmonic-Generation (HGHG) FEL experiment is now being executed at the ATF in the infrared using a CO₂ laser seed. Both the SASE and HGHG work will be extended into the VUV at the Source Development Laboratory (SDL), using the NISUS wiggler.

Another feature, made necessary by the low energy, is the use of a very short period undulator. It is clear that to make use of lower emittance electron beams short period undulators must be developed. The SAE measurement at the ATF was made with an undulator built by MIT with a period of 8.8 mm. Another undulator, with the same period but using a superconducting magnet, was developed by the NSLS. These are the shortest period undulators in actual use anywhere.

Using the MIT microundulator and the ATF beam, scientist from the ATF, MIT and LBL measured for the first time spectral spikes in the spontaneous emission of the microundulator. These fundamental shot noise-driven

fluctuations in the incoherent radiation carry information about longitudinal and transverse phase space of the beam.

Measurements in other fields that have been recently completed at the ATF include work by Russian scientists on an ultra-high precision single-shot beam position monitors. The monitors demonstrated two axis beam position measurement to a resolution of 150 nm for a 0.5 nC beam pulse. Scientists led by BNL's AGS department and Instrumentation Division have demonstrated an optical ultra-fast particle detector. Scientists from Columbia and Yale started measurements of a novel dielectric wake field accelerator.

The ATF and the NSLS are in the center of the national effort to develop short wavelength FELs, and effort that at this time encompasses also SSRL, APS, TJNAF and universities. Whenever possible, we have collaborated with other laboratories, as exemplified by the highly successful "Next Generation Photocathode RF Gun" developed with SLAC and UCLA, the SASE and HGHG experiments at the ATF being developed with ANL using a wiggler from Cornell, as well as collaboration with industry. In the near future, an exciting new experiment is being proposed for the ATF. This is the VISA experiment (short for Visible SASE). It is planned as a collaboration of BNL (NSLS), UCLA, SLAC (SSRL), LLNL and LANL. In this experiment a four meter long wiggler with a period of 1.8 cm will be installed early 1999 at the ATF to produce SASE to saturation at visible wavelengths. This experiment is considered by SSRL as an important milestone on the way to a hard x-ray FEL. Following the experiment at the ATF, the wiggler will be increased in length to six meters and installed at the SDL to extend the experiment to the VUV at about 100 nm. ■

PROJECT

NSLS FACILITY IMPROVEMENTS

Frank Terrano
NSLS
Assistant to Chairman

WORK PLANNING AND CONTROL SYSTEM

FY 1998 was a year of change at BNL as well as at the NSLS. Not so much a physical change as a cultural one. Along with the new Brookhaven Science Associates (BSA) management of BNL, there came an increased emphasis on elevating the awareness of safety and environmental protection throughout the Laboratory. Along with the heightened awareness came new and more formalized systems and procedures, designed to ensure only the highest standards are maintained in these areas. The NSLS, with its community of two and one half thousand users doing research at the facility annually, has always maintained the utmost concern for individual safety and the environment, and continues to be in the forefront when addressing these issues. In the early part of FY 1998 the NSLS agreed to take on a pilot program for the (BNL) Laboratory which entailed the development of a Work Planning and Control System with its primary focus on improving control over the external work coming into the facility. The system was designed to provide a consistent method for identifying and analyzing job hazards, planning work, and coordinating job activities through the issuance of Work Permits for all external work coming into the NSLS. The pilot program met with great success, largely through the efforts of NSLS Building Manager, Mike Kelly and NSLS ES&H Officer, Tom Dickinson. By April 1998 the pilot was concluded and by July the rest of the Laboratory was on the new Work Planning and Control System. The process worked so well that its scope has been expanded to now include a review of all internal work performed by NSLS technical staff, with work permits to be issued as required via an internal review process. Mike Kelly has assumed the responsibilities of Work Control Manager for the overall program at the NSLS.

SPACE MODIFICATIONS

In response to the increasing need for laboratory/setup areas at the NSLS, room 1-110 (contiguous to the NSLS Green-Chasman Library), formerly used as a storage space has been reconfigured. During the fall of 1998, modifications were completed which converted the space into three separate laboratory/setup areas. Located adjacent to the experimental floor, the space is now much better utilized and provides some needed relief for our users. Additional storage has been made available in the basement of building #535 (connected to the experimental floor by tunnel) to accommodate the equipment and materials previously stored in room 1-110. Renovations to the new NSLS vacuum technician lab, occupying additional space recently acquired by the NSLS, together with the new interlock technician lab were completed in the fall of 1998. Both areas are in new locations in the lower level of building #535, connected by tunnel to the NSLS experimental floor, and are designed to provide improved workspaces as well as enhanced working conditions for the technicians. The actual move of both groups is planned to take place shortly after the December 1998 shutdown.

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 the new 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. There will also be some reconfiguration of the lighting plan to take better advantage the fixture locations with regard to the working areas around the experimental floor. Installation is planned to take place after the December 1998 shutdown. ■

Beamline U13UB: Angle-resolved photoemission (ARPES), developed in the seventies, still represents the premier tool available for the study of the momentum dependence of the electronic structure in solids. Here the energy of the emitted photoelectron is measured as a function of the angle of emission. Combined with beamlines such as U13UB, a whole range of new electron spectrometers is expected to lead to a resurgence of activity in this area. By multiplexing in energy and angle, these new instruments allow a considerable reduction in data acquisition times. At the same time, they offer extremely high energy and momentum resolution. With these improvements, ARPES may be used to obtain direct access to the real and imaginary components of the electron self energy, a measure of the interaction of an electron or hole with its environment. The results of a detailed temperature dependent study of a high T_c superconductor illustrate the new possibilities in ARPES. The figure at right shows image of the electron density in momentum space of optimally doped $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_8$ when the sample is in the superconducting state. The "airplane wing" section corresponds to the superconducting paired electrons. The figure above shows temperature dependent "angle integrated" spectra; the sharp feature reflecting the paired electrons is clearly fixed in binding energy, even above T_c .

S. L. Hulbert (NSLS), A. V. Fedorov, T. Valla, and P. D. Johnson (BNL Physics)

SIX ORGANIZATION

ORGANIZATION

NSLS ADVISORY COMMITTEES

1998-1999

Users' Executive Committee

The Users' Executive Committee (UEC) provides for organized discussions among the user community, NSLS administration, and laboratory directorate. It aims to communicate current and future needs, concerns, trends within the user community to NSLS staff and management, and to disseminate to the users information about NSLS and BNL plans. Members were:

Chairperson: John Parise, SUNY @ Stony Brook

Vice-Chairperson: Barbara Illman, USDA/FS Forest Products Laboratory

Past-Chairperson: Joel D. Brock, Cornell University

Secretary: Paul Stevens, Exxon Research & Engineering Co.

General Members: John Hill (BNL-Physics), Carol Hirschmugl (U. of Wisconsin),
Lisa Kelly (U. of Maryland Baltimore County)

NSLS User Administrator: Eva Rothman, Ex-Officio

Beamline Support/R&D Head: Erik Johnson, Ex-Officio

Special Interest Group Representatives

Special Interest groups in areas of common concern communicate with NSLS management through the UEC. Group representatives were:

Biological Scattering, Diffraction	Malcolm Capel, BNL-Biology
Imaging	George Cody, Carnegie Inst. of Washington
Industrial Users	Jean Jordan-Sweet, IBM Research
Infrared Users	Gwyn Williams, BNL-NSLS
Nuclear Physics	Steven C. Whisnant, U. of So. Carolina
Time Resolved Spectroscopy	G. Lawrence Carr, BNL-NSLS
Topography	Michael Dudley, SUNY @ Stony Brook
Students and Post Docs	Patrick Dai, Tufts U.
UV Photoemission, Surface Science	Boris Sinkovic, U. of Connecticut
XAFS	Mark Chance, Albert Einstein Coll. of Med.
X-ray Scattering, Crystallography	Luz J. Martinez-Miranda, U. of Maryland

General User Oversight Committee

The GUOC resolves disputes between General Users, PRTs, and NSLS staff. Members were:

Simon Bare, UPO
Dale Sayers, NCSU

Mark Chance, Albert Einstein Coll. of Med.
Joel Brock, Cornell U.

Science Advisory Committee

The Science Advisory Committee (SAC) evaluates the science programs at the NSLS and makes recommendations to the Chairman. The SAC met in March of 1998 and its members were:

Boris Batterman, CHESS, Cornell U.
 Stephen Burley, Rockefeller University
 Stephen Harrison, HHMI, Harvard U.
 Keith Hodgson, SLAC
 Jochen Schneider, HASYLAB, DESY
 Paul Sigler, HHMI, Yale U.

Martin Blume, Brookhaven National Laboratory
 Sol Gruner, CHESS, Cornell U.
 Franz Himpsel, U. of Wisconsin, Madison
 John Parise, SUNY @ Stony Brook
 Albert J. Sievers, Cornell U.
 Sunil Sinha, APS, Argonne National Laboratory

General User Proposal Study Panels

The Proposal Study Panels met in November 1997, March 1998, and July 1998 to review and rate General User research proposals for the FY 1998 cycles. PSP members are drawn from the scientific community and usually serve a two year term. Members during FY 1998 were:

UV Panel

Daniel Fischer NIST
 David Hanson SUNY
 Laszlo Mihaly SUNY
 Boris Sinkovic NYU
 David Tanner U. of Florida

X-Ray Imaging/Other Panel

Harald Ade NCSU
 Malcolm Howells LBL
 Chris Jacobsen SUNY
 Steve Sutton U. of Chicago

X-Ray Biology

Aneel Aggarwal Columbia U.
 Jens Birktoft Hoffmann-LaRoche
 Roger Burnett Wistar Institute
 Howard Einspahr Bristol-Myers Squibb
 Christopher Hill U. of Utah
 James Hurley NIH
 John Kuriyan Rockefeller
 Thomas Terwilliger LANL
 Gregory Van Duyne U. of Pennsylvania

X-Ray Spectroscopy Panel

Chuck Bouldin NIST
 Bruce Bunker U. of Notre Dame
 Lars Furenlid NSLS
 Steven Heald APS
 James Penner-Hahn U. of Michigan
 Linda Powers Utah State U.
 Dale Sayers U. of North Carolina

X-Ray Scattering/Diffraction Panel

Joel Brock Cornell U.
 Roy Clarke U. of Michigan
 Jerome Cohen NWU
 Benjamin Chu SUNY
 Takeshi Egami U. of Penn
 Doon Gibbs BNL-Physics
 Simon Moss U. of Houston
 Sunil Sinha ANL
 Cullie Sparks ORNL
 Thomas Russell IBM

Allocation Panel

The Allocation Panel met in November 1997, March 1998, and July 1998 to allocate time on beamlines to each General User proposal for the FY 1998 cycles. Allocation Panel members are drawn from the scientific community and usually serve a two year term. Members during FY 1998 were:

UV Members

Harold Ade NCSU
 David Heskett U. of R.I.
 Jan Hrbek BNL-Chemistry
 Paul Stevens Exxon
 Elio Vescovo BNL-NSLS

X21-IDECS

John Tranquada BNL-Biology
 Eric Jensen Brandeis U.
 Eric Shirley NIST
 Akio Kotani U. of Tokyo
 C.C. Kao BNL-NSLS

X-Ray Members

Malcolm Capel BNL-Biology
 Mark Chance Albert Einstein College
 Alex Darovsky SUNY-Stony Brook
 Lars Furenlid BNL-NSLS
 John Hill BNL-Physics
 James McBreen BNL-DAS
 Craig Ogata HHMI
 Jean Jordan-Sweet IBM
 Thomas Thurston BNL-Physics
 Gavin Watson U. of Maryland

NSLS STAFF

- * Temporary
- ** Reports to Denis McWhan,
BNL Assoc. Dir. for Basic Energy Sciences
- [] NSLS Personnel with Multiple Functions
- () PRT personnel or member of non-NSLS division

ORGANIZATION

NSLS STAFF

(signed) Michael Hart, Chairman

October 1, 1998

VUV BEAMLINE GUIDE

Beam line	Research Program	Energy (eV)	Affiliation	Local Contact	Spokesperson
U1A	SEXAFS, ARUPS, XPS	100-1000	Exxon	M Sansone 516-344-3265 516-344-5759	P Stevens 908-730-2584
U1B	(Conceptual)		Exxon	M Sansone 516-344-3265 516-344-5759	P Stevens 908-730-2584
U2A	IR	6 meV-3 eV	Carnegie Inst. Wash	R Lu 516-344-5502 202-686-2410	R Hemley 516-344-5917 202-686-2410x2465
U2B	IR Microscopy, Spectroscopy	6 meV-0.6 eV	Albert Einstein	L Miller 516-344-5609 516-344-2091	M Chance 516-344-5787 718-430-4136/2894
U3A	Detector and system calibration; meas. of material optical const.; spectrometric properties	70-2100	Smiths Astro Obs Bechtel Nevada Los Alamos Natl Lab Pacific NW Natl Lab	A Burek 516-344-5521 516-344-5503	R Blake 516-344-5708 505-466-0566
U3B	(Conceptual)				
U3C	Photoelectron spect.; exp. system and detector calibrations	40-1600	Los Alamos Natl Lab Sandia Natl Lab SFA, Inc	M Sagurton 516-344-5708	R Bartlett 516-344-5708 505-667-5923
U4A	Photoemission spectroscopy to study electronic structure in solids, surfaces	15-200	Boston U No Carolina St U BNL-NSLS Rutgers U	516-344-5504	K Smith 516-344-5504 617-353-6117
U4B	Soft x-ray and/VUV photoemission spectroscopy and photo absorption	20-1200	Naval Res Lab BNL-NSLS SRRC	J Park 516-344-7290 516-344-4744	Y Idzerda 202-767-4481

Beam line	Research Program	Energy (eV)	Affiliation	Local Contact	Spokesperson
U4IR	Grazing incid. Reflectivity in UHV; transmission & normal incidence reflectivity	2.5 meV- 0.5 eV	BNL-NSLS	G Williams 516-344-3634 516-344-7529	G Williams 516-344-3634 516-344-7529
U5	Diagnostic instrument monitors beam parameters during operations and machine studies		BNL-NSLS	R Nawrocky 516-344-5505 516-344-4449	R Nawrocky 516-344-5505 516-344-4449
U5UA	Spin polarized angle- resolved ultraviolet photoemission	10-200	BNL-NSLS BNL-Physics ANL U of Texas	E Vescovo 516-344-5505 516-344-7399	E Vescovo 516-344-5505 516-344-7399
U6	(Available)				
U7A	XPS and XAS on surface and in bulk, under vacuum and atm.P	150-1200	BNL-Physics BNL-Chemistry Dow Chem NIST U of Michigan Rutgers U Texas A&M U	Q Dong 516-344-5507 516-344-5358	D Fischer 516-344-5177
U7B	Photoemission; SEXAFS	15-300	BNL-NSLS	Q Dong 516-344-5507 516-344-5358	S Hulbert 516-344-7570
U8A	ARUPS NEXAFS	10-130	UCA@Riverside	J Yarmoff 516-344-5508 909-787-5336/3537	J Yarmoff 909-787-5336/3537
U8B	ARUPS NEXAFS	10-1000	IBM	K Zhang 516-344-5508	R McFeely 914-945-2068
U8C	Zone plate monochr. evaluation; reflectivity measurements of thin films and multilayers	200-600	IBM	R McFeely 516-344-5508 914-945-2068	R McFeely 914-945-2068
U8D	(Conceptual) Scanning soft x-ray microscopy		IBM	R McFeely 516-344-5508 914-945-2068	R McFeely 914-945-2068

Beam line	Research Program	Energy (eV)	Affiliation	Local Contact	Spokesperson
U9A	(Planned) PSD measurements of UHV materials at room temperature	White Beam	BNL-NSLS	C Lanni 516-344-7766 516-344-4100	C Foerster 516-344-4754
U9B	Absorption.;CD;MCD; flores/phosphorescence spectroscopy (incl. times-res. fl.)	3-12	BNL-Biology	J Sutherland 516-344-5509 516-344-3406	J Sutherland 516-344-5509 516-344-3406
U10A	(Commissioning GU {1998} High Brightnes IR measurements on "bad metals"	6 meV-3 eV	BNL-NSLS BNL-Physics SUNY @ Stony Brook	G Carr 516-344-6220 516-344-2237	G Homes 516-344-5510 516-344-7579
U10B	IR microspectroscopy	50 meV-0.6 eV	BNL-NSLS Northrop Grumman	G Carr 516-344-5510 516-344-2237	G Williams 516-344-7529
U11	Gas phase photo-ionization and spectroscopy	5-30	BNL-DAS BNL-Biology BNL-NSLS	J Sutherland 516-344-5509 516-344-3406	B Klemm 516-344-5511 516-342-4001
U12A	Soft x-ray photoemission (SXPS); XAS; NEXAFS	100-800	Oak Ridge Natl Lab BNL-NSLS	S Hulbert 516-344-5913 516-344-7570	D Mullins 516-344-5512 423-574-2796
U12IR	IR and far-IR measurement time-resolved (pump/probe IR & far-IR spectroscopy	0.3 meV-1 eV	U of Florida BNL-NSLS	G Carr 516-344-6220 516-344-2237	D Tanner 352-392-4718
U13UA	High res VUV/soft x-ray abs and electron spectr. (incl. spin-polarized photoemission)	White Beam	BNL-NSLS	S Hulbert 516-344-5913 516-344-7570	S Hulbert 516-344-5913 516-344-7570
U13UB	UV/VUV spectroscopy; hi resol. Angle resolved photoemission of solid surf.;pump-probe GU-98	5-30	BNL-NSLS BNL-Physics Boston U Brandeis U	S Hulbert 516-344-5513 516-344-7570	E Jensen 617-736-2865
U13UC	Interferometry	White Beam	Lucent Tech	S Spector 516-344-5513	O Wood 908-582-4457

Beam line	Research Program	Energy (eV)	Affiliation	Local Contact	Spokesperson
U14A	VUV/soft x-ray spectroscopy Auger Photo-Electron Coincidence Spectroscopy (APECS)	15-300	BNL-NSLS Rutgers U	S Hulbert 516-344-5514 516-344-5930	S Hulbert 516-344-5514 516-344-7570 R Bartynski 908-445-4839
U14B	Diagnostic Instrument: VUV Ring beam position monitoring		BNL-NSLS	J Hastings 516-344-5514 516-344-3930	J Hastings 516-344-5514 516-344-3930
U15	Soft x-ray spectroscopy (solids and gases)	300-800	BNL-NSLS SUNY@Buff SUNY@SB	A Wen 516-344-5515 516-632-7918	D Hanson 516-632-7917
U16A	(Available)				
U16B	Core level Photo-emission; soft X-ray photoabsorption spectroscopy; Auger photoelectron coincidence spectroscopy (APECS)	5-1000	BNL-NSLS	S Hulbert 516-344-5516 516-344-7570	S Hulbert 516-344-5516 516-344-7570

X-RAY BEAMLINE GUIDE

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X1A	Soft x-ray imaging	0.25-1.0	SUNY@SB No Carolina St U BNL-NSLS LBNL ANL	S Wirick 516-344-5601 516-632-8095	J Kirz 516-632-8106 C Jacobsen 516-632-8093 516-344-4723
X1B	Soft x-ray spectroscopy	0.2-0.9	BNL-Physics KFA - Juelich Fritz-Haber-Inst Boston U	P Johnson 516-344-5701 516-344-3705	P Johnson 516-344-5701 516-344-3705
X2A	(Available)				
X2B	X-ray microtomography	6.5-30, WB	Exxon	S Bennett 516-344-5610 516-344-4719	J Dunsmuir 908-730-2548
X3A1	Short wavelength crystallography, diffraction and scattering	18.2-29.7	SUNY@Buff SUNY@SB Amoco Corp Alfred U	L Ribaud 516-344-5603 716-645-6800x2218 G Wu 516-344-5603	P Coppens 716-645-6800,x2217
X3A2	Diffraction; x-ray spectroscopy; scattering; crystallography; small angle scattering	4.34	SUNY@SB SUNY@Buff Amoco Corp Alfred U	L Ribaud 516-344-5603 716-645-6800x2218 G Wu 516-344-5603	P Coppens 716-645-6800,x2217
X3B1	X-ray spectroscopy; powder diffraction	4-40	SUNY@SB SUNY@Buff Amoco Corp Alfred U	L Ribaud 516-344-5603 716-645-6800x2218	P Coppens 716-645-6800,x2217

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X3B2	Surface science	4-20	SUNY@Buff SUNY@SB Amoco Corp Alfred U	L Ribaud 516-344-5603 716-645-6800x2218	P Coppens 716-645-6800,x2217
X4A	Multiwavelength anomalous diffraction analysis of crystalline biological macromolecules	4-30	Howard Hughes	C Ogata 516-344-5604 516-344-7435	W Hendrickson 212-305-3456
X4B	(Conceptual)				
X4C	Diffraction meas. from biological macromolecules	4-15	Howard Hughes	C Ogata 516-344-5604 516-344-7435	W Hendrickson 212-305-3456
X5A	Laser Electron Gamma Source (LEGS); medium energy nuclear physics	140-470	BNL-Physics Norfolk St U Ohio U U of So Carolina SUNY@SB Syracuse U U of Virginia VA Polytech & St U U of Paris KFA-Juelich Frascati Natl Lab U of Rome II Osaka U	C Thorn 516-344-5605 516-344-7798	A Sandorfi 516-344-7951
X5B	Diagnostic				
X6A	(Available)				
X6B	(Available)				

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
				516-344-5930	516-344-2738
X7A	High resolution structural studies through powder diffraction	5-45	Air Products & Chem BNL-Physics Mobil Carnegie Inst of Was UOP U of Pennsylvania SUNY@SB Chevron UCA@Santa Barbara NIST	D Cox 516-344-5607 516-344-3818	D Cox 516-344-5607 516-344-3818
X7B	Crystallography; wide angle scattering	5-21	Swedish Nat Res Coun BNL-Chemistry	J Hanson 516-344-5707 516-344-4378	T Koetzle 516-344-4384
X8A	Calibration of mirrors & dector systems; material optical constants; spectrometric properties	0.26-5.9	Los Alamos Natl Lab Smiths Astro Obs Bechtel Nevada Sandia Natl Lab Pacific NW Natl Lab	M Sagurton 516-344-5708	R Blake 505-466-0566 516-344-5521
X8C	Diffraction from biological macromolecules and detector calibrations AXAF	5-20	Los Alamos Natl Lab Natl Res Coun/Canada UCA@LA Hoffman-La Roche BNL-Biology	M Sagurton 516-344-5708	J Berendzen 505-665-2552
X9A	Time-resolved x-ray footprinting;	White Beam	Albert Einstein Rockefeller U Sloan-Kettering Inst	M Sullivan 516-344-5609 516-344-3800	M Chance 718-430-4136 718-430-2894
X9B	Small angle scattering; EXAFS; multiple anomalous diffraction (MAD); protein x-ray diffraction	2.1-18.8	Albert Einstein NIH	M Sullivan 516-344-5787 516-344-3800	M Chance 718-430-4136 718-430-2894
X10A	Diffraction; small angle scattering; crystallography; scattering; SAXA; WAXS	6-15.2	Exxon	S Bennett 516-344-5610 516-344-4719	R Kolb 908-730-2970
X10B	Scattering; crystallography PXD, Reflectivity	8 and 12	Exxon	S Bennett 516-344-5610 516-344-4719	R Kolb 908-730-2970

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X10C	EXAFS	4.0-24.0	Exxon	M Sansone 516-344-3265 516-344-5759	P Stevens 908-730-2584
X11A	EXAFS	4.5-35	Naval Res Lab Mobil U of Connecticut No Carolina St U Paul Scherrer Inst BNL-DAS The DuPont Co U of Washington Rice U Ill Inst Tech (IIT) Naval Surf War Cntr Georgia Inst Tech U of Notre Dame Advanced Fuel NJ Inst of Tech	K Pandya 516-344-5611 516-344-7734	D Sayers 919-515-4453
X11B	EXAFS	1.7-8	Naval Res Lab NJ U of Tech Mobil No Carolina St U The DuPont Co BNL-DAS U of Connecticut Paul Scherrer Inst Naval Surf War Cntr Ill Inst Tech (IIT) U of Washington Rice U	K Pandya 516-344-5611 516-344-7734	D Sayers 919-515-4453
X12A	Instrument development and x-ray physics		BNL-NSLS	J Ablett 516-344-2738 516-344-5627	P Siddons 516-344-2738
X12B	Time-resolved and static x-ray diffr. of macromol. crystallography	7.6-13.06	BNL-Biology	M Capel 516-344-5712 516-344-5234	M Capel 516-344-5712 516-344-5234
X12C	Macromolecular crystallography; multi- wavelength anomalous dispersion (MAD)	8-13	BNL-Biology	R Sweet 516-344-5642 516-344-3401	R Sweet 516-344-5642 516-344-3401

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X13A, X13B	R&D optics development; soft x-ray utilization		BNL-NSLS	E Johnson 516-344-5913 516-344-4603	E Johnson 516-344-5913 516-344-4603
X14A	Scattering crystallogr. spectroscopy phase ID and resid. strain meas. high temp x-ray diffraction	2.5-40	Oak Ridge Natl Lab Univ of Ill Urb Cham U of Tennessee U of Houston Sandia Natl Lab	J Bai 516-344-5614 516-344-2583	G Ice 423-574-2744
X14B	(Planned) Manuf. res. for hard x-ray lithogrpny exposures for precision microfabrication	White	BNL-NSLS	E Johnson 516-344-5913 516-344-4603	E Johnson 516-344-5913 516-344-4603
X15A	X-ray standing waves	3-20, WB	ANL BNL-NSLS Northwestern U No Carolina St U Ill Inst Tech (IIT)	Z Zhong 516-344-5617	M Bedzyk 847-252-7763 516-344-7395
X15B	X-ray absorption spectroscopy of complex, reactive, very dilute systems	1.8-10	Lucent Tech	P Northrup 516-344-3565	P Citrin 908-582-5275
X16A	Surface diffraction	4-12	Lucent Tech U of Illinois	W Lehnert 516-344-5715 516-344-3635	K Evans-Lutterodt 908-582-2154 516-344-2125
X16B	rapid access for sample characterization; powder characterization; thin film characeterization	7.85	Lucent Tech	W Lehnert 516-344-5715 516-344-3635	K Evans-Lutterodt 908-582-2154 516-344-2125
X16C	Novel spectroscopy; diffraction	3-16	Lucent Tech U of Illinois	W Lehnert 516-344-5715 516-344-3635	K Evans-Lutterodt 908-582-2154 516-344-2125
X17B1	X-ray scattering at high pressure and temperature	20-100, WB	BNL-NSLS SUNY@SB	Z Zhong 516-344-5717 516-344-5617	J Hastings 516-344-3930 516-344-5514
X17B2	Medical research: radiotherapy	27-35	BNL-NSLS BNL-Medical Stanford U Ill Inst Tech (IIT) Stanford U	N Gmür 516-344-5617 516-344-2490	W Thomlinson 516-344-3937

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X17C	Hi P research; energy dispersive diffraction from microscopic sample volumes	10-100, WB	Naval Res Lab LLNL Carnegie Inst of Was U of Chicago	J Hu 516-344-5917 202-686-2410	H Mao 202-686-4454 E Skelton 202-767-3014
X18A	Diffuse and surface scattering	4-20	U of Missouri U. of Maryland BNL-DAS U of Illinois Purdue U Penn St U	S Ehrlich 516-344-5618 516-344-7862	G Liedl 317-494-4100
X18B	X-ray spectroscopy	7-40	UOP Chevron AlliedSignal BNL-NSLS Dow Chem PPG Industries General Electric	S Khalid 516-344-5718 516-344-7496	P Siddons 516-344-2738 S Khalid 516-344-7496
X19A	X-ray absorption spectroscopy: EXAFS and XANES	2-8	BNL-DAS Rutgers U BNL-NSLS U of Kentucky UCA@Davis	S Khalid 516-344-5718 516-344-7496	S Khalid 516-344-5718 516-344-7496
X19C	Topography; liquid surface scattering	4-25, WB	Johns Hopkins U Army Research Lab SUNY@SB NASA U of Wisconsin Northrop Grumman Dartmouth Coll U of Chicago U of Illinois	Z Huang 516-344-5719 312-702-7229	M Dudley 516-632-8500
X20A	X-ray Scattering and diffraction	6.3-12	MIT IBM	J Jordan-Sweet 516-344-5720 914-945-3322	R Birgeneau 617-253-4937
X20B	Scattering at fixed energy	17.4 (fixed)	IBM MIT	J Jordan-Sweet 516-344-5720 914-945-3322	J Jordan-Sweet 516-344-5720 914-945-3322

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X20C	X-ray scattering and diffraction with high flux by multilayer monochromator	4-11	IBM MIT	J Jordan-Sweet 516-344-5720 914-945-3322	J Jordan-Sweet 516-344-5720 914-945-3322
X21	Inelastic x-ray scattering	5-10	BNL-NSLS	C Kao 516-344-5721 516-344-4494	C Kao 516-344-5721 516-344-4494
X22A	X-ray scattering of thin films, multilayers, catalytic materials, electrochemical interfaces	10 NOM.	BNL-Physics BNL-DAS U of Maryland	J Hill 516-344-3736 516-344-5622	D Gibbs 516-344-4608 516-344-5622
X22B	X-ray scattering and surfaces; magnetic stud. under magnetic fields	5-10	BNL-Physics ANL	E DiMasi 516-344-5622 516-344-2211	D Gibbs 516-344-4608
X22C	X-ray Scattering studies of magnetism; structure and phase behavior of metal surfaces	3-12	BNL-Physics ANL U of Maryland	J Hill 516-344-5622 516-344-3736	D Gibbs 516-344-4608
X23A2	EXAFS	4.7-30	NIST	J Woicik 516-344-5823 516-344-5236	J Woicik 516-344-5823
X23A3	Topography; small-angle scattering	5-30, WB	NIST	D Fischer 516-344-5507 516-344-5177	G Long 301-975-5975 516-344-5623
X23B	EXAFS; scattering; crystallography	3-10.5	Naval Res Lab	J Kirkland 516-344-5723 516-344-2258	T Elam 202-767-3015

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X24A	Atomic, molecular, and optical physics with x-rays	1-5	NIST BNL-NSLS Northwestern U U of Rhode Island U of Tennessee Brooklyn Coll - CUNY U of Maryland	B Karlin 516-344-7863 516-344-5624	T Jach 301-975-2362
X24C	Photoemission and reflectance spectroscopy	6-1800 eV	Naval Res Lab	J Rife 516-344-5724 202-767-4654	M Kabler 202-767-2223
X25	High intensity, high brightness photon beam; high Q-resolution elastic scattering	3-30	BNL-NSLS	L Berman 516-344-5625 516-344-5333	L Berman 516-344-5625 516-344-5333
X26A	X-ray Microscopy; structural biology; atomic and molecular physics	4-20, WB	U of Chicago/CARS BNL-DAS BNL-Biology U of Georgia/SREL Cold Spring Harb Lab SUNY@SB	G Shea-McCarthy 516-344-5626 516-344-2229	S Sutton 630-252-0426
X26C	Dev. and applic. of x-ray microscopy techniques; Laue diffraction protein crystallography	4-30, WB	BNL-Biology BNL-DAS U of Chicago/CARS U of Georgia/SREL Cold Spring Harb Lab SUNY@SB	G Shea-McCarthy 516-344-5726 516-344-2229	R Xu 516-367-8872
X27A	NSLS R&D only; instrument development and x-ray physics		BNL-NSLS	B Dowd 516-344-5738 516-344-7092	P Siddons 516-344-2738
X27B	NSLS R&D only; Develop exposure techniques; matls. char.; and tool development for microfab.		BNL-NSLS	E Johnson 516-344-5627 516-344-4603	E Johnson 516-344-5627 516-344-4603

Beam line	Research Program	Energy (keV)	Affiliation	Local Contact	Spokesperson
X27C	Simultaneous small & wide angle x-ray scattering (SWAX) in real time		AlliedSignal BNL-Physics General Electric USAF Montell Polyolefins NIST SUNY@SB NIH	F Yeh 516-344-5627 516-632-7892	B Hsiao 516-632-7793
X28A	Instrument development; and detector development for the X-ray Ring		BNL-NSLS	E Johnson 516-344-5913 516-344-4603	E Johnson 516-344-4603
X28B	Photon-based diagnostics and detector development for the X-ray Ring		BNL-NSLS	E Johnson 516-344-4603 516-344-5913	E Johnson 516-344-4603
X28C	Photon-based diagnostics and detector development for the X-ray Ring		BNL-NSLS	P Stefan 516-344-2117 516-344-5728	P Stefan 516-344-2117
X29A	(Available)				
X29B	(Available)				
X30	Diagnostic Instrument		BNL-NSLS		

SEVEN

APPENDICES

Beamline X3A2: The interaction of polyelectrolytes with oppositely charged surfactants can induce complex formation with highly ordered structures. These ordered structures have a host of possible applications ranging from improved mechanical behavior to unusual optical, electrical, and biological properties. Small-angle x-ray scattering was used to investigate the nanostructures of complexes formed by poly(sodium methacrylate-co-N-isopropylacrylamide) (P(MMA/NIPAM)) copolymer gels interacting with tetradecyl-trimethylammonium (TTAB) at room temperature. In sequence with decreasing charge density of P(MMA/NIPAM) chains, structures of $Pm\bar{3}n$ cubic, face-centered cubic close packing of spheres (FCC), and hexagonal close packing of spheres (HCP) were respectively determined for the first time in polyelectrolyte-surfactant complexes.

S.-Q. Zhou, F. Yeh, B. Chu (SUNY at Stony Brook), and C. Burger (Max Planck Institut, Germany)

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