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PROGRESS REPORT OF MATRIX GROUP

for

Materials Research and Beam Line Operation Utilizing NSLS

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Abstract

MATRIX, a participating research team of Midwest x-ray scattering specialists, continues to operate beam line X-18A at NSLS. Operations of this line now provides state-of-the-art capabilities to a wide range of people in the Materials Science and Engineering research community.

Improvements of the beam line continue to be a focus of MATRIX. Throughout this past year the emphasis has been shifting towards improvement in "user friendly" aspects. Simplified control operations and a shift to single-user personal computer has been a major part of the effort.

Over the past year all 232 operational days were fully utilized. Beam line tests coupled with MATRIX members combined to use 284 days. General user demand for use of the beam line continues to be strong and four groups were provided 48 operating days.

Research production has been growing as NSLS and the beam line become a more stable type of operation. For 1992 the MATRIX group published six articles. To date, for 1993 the same group has published, submitted, or has in preparation nine articles.

Recent research milestones include: the first quantitative structural information on the as-quenched and early stages of decomposition of supersaturated Al-Li alloys; the first quantitative diffuse scattering measurements on a complex system (Co substitute for Cu YBCO superconductor); demonstration of capabilities of a new UHV surface diffraction chamber with in-situ characterization and temperature control (30-1300K); feasibility of phasing structure factors in a quasicrystal using multiple Bragg scattering.

1.0. Introduction

The MATRIX Participating Research Team (PRT) was formed by people from some key midwestern institutions and includes people with overlapping interests in X-ray scattering. MATRIX is organized with an Executive Committee that establishes the policies for the operation of MATRIX. The Director of MATRIX has the responsibility for the administration of the operation in accordance with the policies of both the Executive Committee and NSLS.

MATRIX

Executive Committee

G. L. Liedl, Purdue University, Director
R. Colella, Purdue University
J. B. Cohen, Northwestern University
H. Chen, University of Illinois-Champaign-Urbana
H. Taub, University of Missouri-Columbia

Other Members,

S. Durbin, Purdue University
S. Ehrlich, Purdue University
P. Georgopoulos, Northwestern University

The MATRIX research effort is in PHASE TRANSFORMATION/CHARACTERIZATION where there are two major divisions of our activity related to the dimensionality of the problem, i.e., three dimension or two dimensional type problems.

Recognition of superior accomplishments of a MATRIX PRT member was noted in the past year. Jerome B. Cohen's contributions to the applications of diffraction in the understanding of materials phenomena was recognized by two major international awards this year. He received the 1992- Acta Metallurgica Gold Medal and the 1992-Robert F. Mehl Award and Institute of Metals Lecturer. Much of the work which was the basis for these awards was related to his research utilizing the MATRIX beam line. More recently, he was honored for his many accomplishments to the field through election to the National academy of Engineering.

2.0. Beam Line Projects

A number of improvements in the beamline operations have taken place during the past year. Among these improvements is the modification to improve beam line vacuum and to allow monochromator adjustment during operations. Other major efforts have been directed toward software improvements to allow on-line visual monitoring of measurements and analysis of data. User support has been improved with an updated Beam Line Manual and Beam Line User's Guide. Appendix II has the updated Beam Line Manual and the Beam Line User's Guide is included as Appendix III.

3.0. Beam Line Utilization

Over the past year 232 operational days were available. The following table shows how these days were utilized. As in the past our practice involves the controlled development of the beam line and we reserve some time for such tests. Part of the 46 operational days assigned to the beam line are for tests and training. A small part is scheduled for changes in beam line configuration. Most of the beam line days are the single and five bunch days being scheduled by NSLS. Trend has been to increase such days and since flux is a limiting factor for most users these days are not very useful for members and can not be used for General Users. Therefore, MATRIX uses these days for testing and training. All eight MATRIX members used some time last year and the distribution is as shown below. Our general user requests normally exceed the 25% allotment and we try to satisfy them. This year four different groups were accommodated and given the days as indicated in the following.

**MATRIX Beam Line Schedule
January Through December, 1992**

<u>Activity</u>	<u>Affiliation</u>	<u>Scheduled Days</u>
Beam Line Tests/Training		46
MATRIX Members		138
H. Chen	U. of Illinois	9
J. Cohen	Northwestern	32
R. Colella	Purdue	9
S. Durbin	Purdue	9
S. Ehrlich	MATRIX	27
P. Georgopoulos	Northwestern	6
G. Liedl	Liedl	22
H. Taub	U. of Missouri	24
General Users		48
F. Lu	BNL	7
S. Williams	Northwestern	25
J. R. Dennison	Utah State Univ	6
M. Bloch	ANL	10
	Total	232

PROGRESS REPORT

4.0. Research Milestones of Matrix Research Projects

**EARLY STAGES OF δ' PRECIPITATION IN Al-3.2 WT % Li ALLOY,
V. Mahadev and G. L. Liedl (Purdue University)**

Binary Al-Li alloys derive their strength predominantly from precipitation hardening, the strengthening precipitate being Al_3Li , the metastable δ' phase. Studies have identified that the δ' phase is highly ordered with the L1_2 order, is coherent with the solid solution (f.c.c. α phase), and has a very small misfit.

The decomposition in the latter stages, the coarsening regime, is generally accepted to follow the LSW theory. Quantitative studies using microscopy and scattering methods confirm the LSW behavior in the latter stages. Very little experimental evidence is available for the early stages of aging and thus there exists some uncertainty in this regime. Both resistivity and DSC measurements have suggested the formation of precursors such as G.P. zones prior to the formation of δ' . However, the interpretation of the data is still open to question. Moreover, direct evidence for the existence of these zones has not yet been reported.

As with other ordered intermetallic compounds, the order parameter must be considered in addition to the composition as a variable in describing the ordered phase. Because of the uncertainty in the order parameter the sequence in the precipitation is controversial. Khachaturyan et al. addressed this issue using a mean-field model to predict the stability of each phase. Their model yields conditions for nucleation and growth and for spinodal decomposition. Some qualitative aspects of nucleation and growth at early stages were indicated by Ceresara et al. Recently Schmitz and Haasen, utilizing a HREM method to image small precipitates, reported on a study of the early stages of a 7 at.% Li alloy aged at 463K. Their analysis indicated a classical nucleation and growth mechanism with a possible congruent ordering reaction during quenching.

To aid in the interpretation of the results, a simple simulation model was developed. The model assumes a uniformly distributed set of ordered stoichiometric δ' particles in a completely

randomly ordered matrix. Further a Gaussian particle size distribution is used in the model. This distribution is based upon a previous study of the particle size distribution for later stages of aging in this alloy. Values for the average particle size and volume fraction of the δ' precipitates were determined directly from the measured intensities of the superlattice scans.

Simulations were performed by computing the intensity distribution in the minimum volume. This intensity was then normalized with respect to the Laue monotonic scattering of a random alloy of overall composition $C_{Li} = 0.125$, the average composition of the alloy. Using the model intensity, short range order parameters were calculated. The best fit between measured and simulated short range order parameters was obtained for an Al to Li atom ratio of 3 to 1 for the δ' phase, and a Gaussian distribution using measured mean values and ranges from a quarter of the mean to three times the mean in the simulation. If one considers that the particle size and the volume fraction of the δ' phase change with aging time, the data is consistent with a classical nucleation and growth mechanism. Furthermore, the data can be extrapolated to correlate with the previously reported data on polycrystalline samples aged under similar conditions for longer times.

A STUDY OF THE LOCAL ATOMIC STRUCTURE IN $Hg_{0.80}Cd_{0.20}Te$ USING X-RAY DIFFUSE SCATTERING,

J. P. Quintana and J. B. Cohen (Northwestern University)

Semiconductors based on the $Hg_{1-x}Cd_xTe$ system are in wide use as infrared sensitive materials. The band gap in $Hg_{1-x}Cd_xTe$ is tunable with composition from being a semimetal to a semi-insulator over the entire composition range¹. The published phase diagram for this system consists of a continuous liquidus across the entire range of x ². While no long-range order has been demonstrated, various studies of the local atomic environment in $Hg_{0.8}Cd_{0.2}Te$ have been attempted. Even though quantitative distributions are difficult to obtain with prior NMR and Raman studies they suggest that, rather than local clustering, local ordering is occurring with a preference for 3:1 Hg-Cd near-neighbor quadruplets. Various researchers have attempted EXAFS on this system to determine the near-neighbor displacements. However, the uncertainty inherent in EXAFS measurements is of the same magnitude as the expected strain difference between Cd-Te and Hg-Te bonds.

The local atomic arrangements in a commercial n-type wafer of $Hg_{0.8}Cd_{0.2}Te$ were investigated by measuring the diffuse x-ray scattering in two volumes in reciprocal space. A change in contrast between the two measurements was achieved by making one volume measurement at 12037 eV and a second volume measurement at 12270 eV, i.e., near the HgL_{III} edge. The difference between these two measurements yielded intensity only due to Hg-Hg, Hg-Te, and Hg-Cd pair interactions. In all three patterns, peak-like features were apparent at the forbidden Bragg peak positions on thermal diffuse scattering ridges that joined major Bragg reflections; these are primarily attributed to second order displacement effects on the mixed cation sublattice. The first two Warren-Cowley short-range order parameters were determined to be $\alpha_{\frac{1}{2}\frac{1}{2}} = -0.050(26)$ and $\alpha_{110} = 0.118(35)$. Simulations of the structure revealed small ordered regions with a preference for 3:1 Hg-Cd near-neighbor configurations. The near-neighbor Hg-Te bonds contract from that calculated from the average crystal's lattice parameter, and this Hg-Te distance is less than the distance in HgTe.

SYNCHROTRON X-RAY DIFFRACTION AND SCANNING TUNNELING MICROSCOPY STUDIES OF LIQUID CRYSTAL FILMS ADSORBED ON SINGLE-CRYSTAL GRAPHITE

H. Taub, P. Dai, S.-K. Wang, and J. E Buckley, University of Missouri and S. N. Ehrlich, Purdue University

Recently, there has been considerable activity in applying scanning tunneling microscopy (STM) to structural determinations of solid organic monolayers. Systems of particular interest include monolayers of molecules which form liquid crystal phase in bulk such as the n-alkylcyanobiphenyls (nCB) but which are found to form crystalline monolayers when adsorbed on solid substrates. We have conducted a series of STM and synchrotron x-ray diffraction experiments to investigate the structure of 10CB films adsorbed on single-crystal graphite. To

our knowledge, this is the first time both techniques have been applied to the same sample of an organic film. Our principal result is the discovery of a new commensurate monolayer phase of 10CB on the graphite (0001) surface which, although differing from the previously reported monolayer structure of 10CB on graphite, is similar to that observed for 8CB on MoS₂. Thus our results suggest the existence of two generic crystalline structures of NCB monolayers on surfaces of hexagonal symmetry. We also found the monolayer d-spacing inferred by STM to be 10% larger than that obtained by x-ray diffraction. After considering a number of alternative explanations, we attributed this discrepancy to a systematic error introduced in calibration of the STM length scale against the graphite substrate.

PHASE DETERMINATION OF X-RAY REFLECTIONS IN A QUASICRYSTAL

R. Colella et.al., (Purdue University)

It has been shown recently that multiple Bragg scattering can be used for phase determination of x-ray structure factors. The general idea is to monitor the intensity of a weak reflection as the crystal is rotated around the scattering vector. When a strong reflection is excited simultaneously, the diffracted intensity exhibits a peak as a function of ψ , the azimuthal angle of rotation, with asymmetric side bands. It has been pointed out that phase information can be obtained from the asymmetric sidebands using n-beam dynamical theory, even when dealing with mosaic crystals of general shape.

In this work we report the observation of multiple diffraction effects (called: Renninger effect) in a quasicrystal. Since a quasicrystal does not possess long range periodicity in the usual sense, it is not clear that all diffraction features present in ordinary crystals should be visible in quasicrystals. However, we know that strong and sharp Bragg diffraction spots are produced by quasicrystals. We also know how to predict the positions of nodes in reciprocal space.

Since a quasicrystal is not a periodic structure, the notion of centrosymmetry (or lack of it) can only be established in the whole crystal. On the other hand, for the same reason, (lack of long range periodicity), no two pieces of the same quasicrystalline material are identical. If we cut a big quasicrystal into, say, ten pieces, their diffraction patterns are not expected to be absolutely identical (neglecting for a moment size effects), although we doubt that any discernible difference will be visible. Therefore, the notion of centrosymmetry (or lack of) can only be established on average. It may be useful to compare with the situation of a regular, periodic, crystal subject to thermal motion. We can say that the ten pieces resulting from cutting a large quasicrystal are the analog of an "ensemble" of systems, in the sense of statistical mechanics. The "average structure" is the analog of an ensemble average. In other words, the ten small quasicrystals constitute a set which is related to the average structure mentioned above in about the same way as several instantaneous snapshots of a regular, periodic, crystal subject to thermal motion are related to its ideal periodic, static structure.

Our conclusion, from the results is that there is no point in the crystal that can be taken as a center of symmetry. The question of the presence or absence of centrosymmetry is of crucial importance in the process of determining atomic locations. So far, most of the work along this direction has been done using a method based on contrast variation effects in neutron diffraction.

In conclusion, we have demonstrated the feasibility of phasing structure factors in a quasicrystal using multiple Bragg scattering. The phase values for the triplet invariants obtained by this method are model independent, and can be used to assess different structural models.

5.0. Research Output

The impact of the MATRIX group is evident from the publications related to the use of the beam line. As NSLS and the beam line have become more dependable the research output has increased as seen from the table below.

	Research Output									
Year	1984	85	86	87	88	89	90	91	92	93
No. Publications	5	8	5	4	11	6	7	9	6	9+
Thesis Completed	-	-	3	-	3	2	2	3	2	-
General User Projects	-	-	3	1	1	3	7	7	6	3+

6.0. Publication Citations for 1991 to date

(Most Recent Reprints for 92 and 93 are given in Appendix I)

1991

1. Joanne R. Levine, J. B. Cohen and Y. W. Chung, "Thin Film Island Growth Kinetics: A Grazing Incidence Small Angle X-ray Scattering Study of Gold on Glass," *Surface Science*, 248, pp. 215-224, (1991).
2. S. A. Hoffmann, C. Venkatraman, S. N. Ehrlich, S. M. Durbin, and G. L. Liedl, "Structural and Transport Measurements in $\text{La}_{1.8}\text{Sr}_{0.2}\text{NiO}_{4+\delta}$," *Physical Review B*, 43, 10, pp 7852-58 (1991).
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5. K. Mahalingam, V. Mahadev, G. L. Liedl and T. H. Sanders, Jr. "Precipitation Behavior of δ' in a Binary Al-Li Alloy," *Script Met*, 25, pp. 2181-86 (1991).
6. R. Colella, "X-Ray Diffraction Studies of Surface Structures," Part I and Part II. Invited review article. To be published in *Landolt-Bornstein, Volume III/24: "Physics of Solid Surfaces."* Edited by G. Chiarotti (Springer-Verlag).
7. R. Colella, "Truncation Rod Scattering: Analysis by Dynamical Theory of X-Ray Diffraction," *Phys. Rev. B*, 43, 17, pp 13827-13832, 1991.
8. R. Buschert and R. Colella, "Photostriction Effect in Silicon Observed by Time-Resolved X-Ray Diffraction," *Solid State Communications*, 80, 6, pp 419-422, 1991.
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2. V. Mahadev, K. Mahalingam, G. L. Liedl, and T. H. Sanders, Jr., " δ' Precipitation in a Binary Al-3.2 wt % Li Alloy, Kinetics of Ordering Transformations in Metals," H. Chen and V. Vasudevan eds., TMS, Warrendale, PA, pp. 337-48, 1992.
3. S. N. Ehrlich, L. D. Chapman and N. M. Lazarz, "Time-Resolved X-ray Diffraction of Hot Phonons in Quartz," *Solid State Communications*, 87, p. 451 (1992).
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5. P. Zschack, J. B. Cohen and Y. W. Chung, "Structure of the $\text{TiO}_2(100)$ 1 x 3 Surface Determined by Glancing Angle X-ray Diffraction and Low Electron Diffraction," *Surface Science*, 262, pp 395-408 (1992).
6. D. R. Haeffner and J. B. Cohen, "The Structure of G.P. Zones in an Al-Cu-Zn Alloy," *Acta Metall.*, 40, #4, pp. 831-838 (1992).

1993

1. B. D. Butler and J. B. Cohen, "Atomic Displacements Caused by Carbon Interstitials in Austenite," *Acta Metall. Mater.*, Vol. 41, #1 pp. 41-48 (1993).
2. J. P. Quintana and J. B. Cohen, "A Study of the Local Atomic Structures in $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$ Using X-ray Diffuse Scattering," *J. Mater. Res.*, 8, No. 4, pp 855-863, (1993).
3. J. B. Cohen, "Local Atomic Structure in $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$ and $\text{Cd}_{0.725}\text{Mn}_{0.275}\text{Te}$ " in *Diffusion in Ordered Alloys*, Eds. B. Fultz, TMS, in press.
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6. T. E. Burns, J. R. Dennison and S. N. Ehrlich, "Synchrotron X-Ray Diffraction Studies of Multilayer Nitrogen Physisorbed on Graphite," in preparation.
7. J. P. Anderson and Haydn Chen, "Atomic Short-Range Order Determination in Au-25 at % Fe and Ni-12.5 at % Si, in preparation.
8. V. Mahadev, K. Mahalingam, G. L. Liedl and T. H. Sanders, Jr., "Very Early Stages of δ' Precipitation in a Binary Al-3.2 Wt % Li Alloy," submitted to *Acta Metall.*
9. R. Colella, H. Lee, and L. D. Chapman, "Phase Determination of X-Ray Reflections in a Quasicrystal." To be published in *Acta Cryst.*

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Appendix II

MATRIX X-18A

BEAM LINE MANUAL

The purpose of this manual is to give an overview of the MATRIX X-18A beam line. Major components of the beam line, the diffractometer, detectors, the computer, motor indexers and drivers, etc., are described. Some advice about using the beam line is also provided. Appendices include measurements which have been made on the characteristics of the beam line. More detailed information about systems and components such as detectors, electronics, etc. can be found by reading the appropriate manuals. Computer software information is available through on-line help files and manuals.

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THE BEAM LINE

Introduction

The X-18A MATRIX beam line is shown in Figure 1. For the purpose of this manual the X-18A beam line starts at the isolation valve just outside of the shield wall and ends at the Be window located just in front of the hutch. The major components of the beam line are the fixed aperture, the first Be window, the horizontal and vertical slits, the monochromator, the mirror and the second Be window. There are also two fluorescent paddles, one after the monochromator and one after the mirror, which are used for alignment and diagnostic purposes. The hutch slits assembly will also be included in this section since it is physically attached to the end of the beam line. The total distance from the source to the sample position is 23500 mm.

The beam line is pumped by five ion pumps, one on the front end, one near each of the slits and mirror chambers, one under the monochromator chamber and one at the end of the beam line. There are three gate valves to allow different sections of the beam line (each with its own ion pump) to be bled up to atmosphere without affecting the rest of the beam line.

There are four parts of the beam line which are water-cooled because they are exposed to the white beam. These are the front end fixed aperture, the first Be window, the horizontal and vertical slits and the first crystal of the monochromator. The first three are cooled using the cooling water provided by NSLS while the first monochromator crystal is

Figure 1

cooled by our own water chiller to maintain it at a constant temperature. The water flow to each of these components is monitored by a Proteus flow switch.

Beam line safety requirements include the control and display of critical vacuum pressure and cooling water flow. The status of each of the five ion pumps (whether they are on or off), the water flow to each of the water-cooled components (whether the flow rate is above or below a set point), and the status of the isolation valve (whether it is fully open or not) is displayed on the panel just to the right and above the hatch door. ALL OF THE GREEN LIGHTS SHOULD BE ON. If there is a fault anywhere the red light associated with that component will go on (the green light will go off) and the safety shutter in the front end will automatically close. The safety shutter must be opened by the user and can only be done so if the panel shows all green lights again. If any red light goes on PLEASE NOTIFY THE MANAGEMENT so that the problem can be fixed.

The fixed aperture is used to cut the horizontal fan of the beam from 8 milliradians (dictated by the front end aperture) down to 6 milliradians so the beam can pass through the first Be window. The fixed aperture is 7682 mm from the source. The first Be window is 5 mm high, 50 mm wide and 10 mil thick. It is located 7998 mm from the source. When the isolation valve is closed it separates the X-18A beam line vacuum from the front end vacuum, which is connected to ring vacuum and is shared by both X-18A and X-18B. The beam line vacuum is contained at the end of the beam line by the second Be window which is 1 inch in diameter and 10 mil thick, and is located just outside the hatch.

The Beam Line Slits

The beam line slits are the first major components of the beam line which can be adjusted by the user. They are located 9127 mm from source point. We have both horizontal and vertical slits in the chamber and a viewport so we can see where the x-ray beam hits the slits (they are coated with fluorescent powder). The horizontal slit is an upside down V which lets 5.9 mrad through at the bottom and goes to zero at the top. The vertical slit is a set of 6 slits of heights 4 mm, 3 mm, 2 mm, 1 mm, 0.6 mm and 0.3 mm, going from bottom to top. They can both be moved using stepping motors controlled by the computer.

The vertical cross-section of the white beam coming through the first Be window was mapped by placing a detector at the end of the beam line and scanning the 1 mm high vertical slit of the beam line slits assembly through the beam at two energies, 8 keV and 15 keV. A plot of these scans is shown in Figure 2. Note that the hot part of the beam is in the center of the whole beam getting through the first Be window (the peaks are symmetric).

The beam line slits have several uses. When the focusing mirror is being used the angular dispersion of the beam is increased leading to poorer resolution. This loss in resolution can be lessened by narrowing the beam in the horizontal direction so that the beam is reflecting off less of the sides of the cylindrical mirror and more off the center of the cylinder. This, of course, will also cut down on the intensity of the beam reaching the sample, so a compromise must be reached.

When the beam line slits are 'wide open' the beam is larger than the cross-section of

Vertical Cross-section of Beam

Figure 2

the mirror so some radiation can get down the beam pipe and reach the sample. This 'stray' radiation may contain some higher harmonics which the mirror is meant to diminish. The beam line slits should therefore be adjusted appropriately if these effects are important.

The Monochromator

The X-18A monochromator is located 10117 mm from the source. It has two Si (111) crystals and has an energy range of about 4 - 20 KeV. It is not a fixed exit geometry and thus the beam is low at lower energies and high at higher energies. The relative height of the beam exiting the monochromator as a function of energy is shown in Figure 3.

The angle of the two crystals with respect to the beam is adjusted by a Huber circle which is external to the monochromator chamber, and is connected to the crystal mounts by a tubular support which seals with the chamber using two Viton o-rings. The first crystal of the monochromator is cooled by water circulated through a chiller (set at 64° F) located under the beam line. The inside of the monochromator can be seen through a viewport on the X-18B side. The angle of the monochromator is controlled by one stepping motor through a 10:1 gear reducer so that one revolution of the motor corresponds to 0.1° of motion.

The fine adjustment of the second crystal angle and tilt is accomplished through two piezoelectric crystals by applying a voltage of between 0 and negative 1000 volts to each of them. The dual high voltage supply used is the one with the red LED readouts. The top half is for angle and the bottom half is for tilt. Since these supplies are only capable of supplying a voltage of up to 1000 volts it is tough to apply too much voltage to the

VERTICAL MOTION OF BEAM VS ENERGY

Figure 3

piezoelectric crystals. However, when using these it is important to touch only the voltage adjust knobs. DO NOT CHANGE THE POLARITY OF THESE HIGH VOLTAGE SUPPLIES TO POSITIVE AND DO NOT CONNECT THE PIEZOELECTRIC CRYSTALS TO ANY OTHER HIGH VOLTAGE SUPPLY.

When changing energy by more than a keV it is usually necessary to finely adjust the angle of the second monochromator crystal, using the top half of the dual high voltage supply, in order to peak the intensity. If you are changing the angle of the monochromator crystals by many keV it is usually a good idea to change the angle in steps, finely adjusting the angle of the second crystal with the piezoelectric crystal to peak the intensity as you change energy so you never lose the beam.

Adjusting the tilt of the second monochromator crystal, using the bottom half of the dual high voltage supply, has the effect of moving the beam horizontally in the hutch (there doesn't seem to be much loss in intensity when doing this). The two crystals are parallel only at a certain tilt voltage. When this condition is achieved the beam will stay fairly stationary in the horizontal direction as the energy is changed over its entire range. If the crystals are not parallel then as you change the energy the beam will move horizontally. Therefore if you are planning on changing the energy during the course of your experiment it is important that the tilt of the second crystal be parallel to the first, otherwise the beam will surely move sideways. The easiest way to do this is to view the beam, using the TV monitor, on a fluorescent screen placed at the sample position. Mark the position of the beam on the monitor at the lowest energy you are planning to use. Change the energy to the highest you are planning to use and make sure the beam hasn't moved from the mark you made on the TV monitor. This alignment is checked regularly by the management,

but should be checked if it is important.

The calibration of the monochromator has been done by the management and details are contained in Appendix A. If you have any questions about the calibration of the monochromator please ask, or measure it yourself. However, DO NOT CHANGE THE CALIBRATION OF THE MONOCHROMATOR ON THE COMPUTER. THIS IS TO BE DONE BY THE MANAGEMENT ONLY. An optical encoder is attached to the stepping motor of the monochromator and its readout in degrees of Bragg angle is given by the up-down counter at the top of the electronics rack.

The Mirror

The X-18A mirror is located 11750 mm from the source. It is a cylindrical aluminum mirror which is electroless nickel-plated and then coated with platinum. It is 700 mm long, 100 mm wide and 44 mm high. The radius of curvature of the cylinder is 78 mm. The width of the cylinder at the top of the mirror is 78 mm. The mirror is designed to focus the source point of the synchrotron at the sample position. The cylindrical shape of the mirror focuses the beam horizontally while focusing in the vertical is achieved by bending the mirror along its length. The mirror sits 11750 mm downstream of the source point and the same distance upstream of the sample position, making the total length of the beam line from source to sample 23500 mm. The mirror is designed to focus at the sample with an incident angle of about 6.6 milliradians. This makes the cutoff energy about 12 KeV.

In order to use the beam line focusing mirror the Be window at the end of the beam line must be raised so that the beam reflected up from the mirror can still pass through

it. The standard configuration of the beam line is with the focusing mirror in place. Those who do not wish to use the mirror must request this change in normal beam line configuration before the schedule is made. In this way, those not using the mirror can be scheduled consecutively, and time can be scheduled for raising the beam line, refocusing the mirror and realigning the diffractometer.

Mirror Support

The mirror is supported at two points, at the upstream end and at the downstream end. Each of these supports has a vertical and horizontal motion. In addition, the mirror can be bent along its length.

Vertical Motion

The vertical motion of each mirror support can be used to adjust the height and the angle of the mirror with respect to the beam. The vertical motion is achieved by mounting each end of the mirror on a vertical slide which is then supported by a mechanical actuator (1 turn = 0.0254 mm travel) mounted on the bottom of the mirror support frame. A stepping motor is fastened to the worm of each mechanical actuator so that the vertical motion of the mirror can be controlled by the computer. The proper angle and height of the mirror is adjusted by the management. If a problem is perceived in the focus of the mirror PLEASE NOTIFY THE MANAGEMENT.

Horizontal Motion

The horizontal motion is used to align the mirror so that its length is parallel to the beam. The horizontal motion is achieved by mounting each end of the mirror on a

horizontal slide (which is then mounted on the vertical slide above). The motion of each of these slides can be controlled by a micrometer (1 turn = 0.5 mm travel), which can then be locked. This motion is used only when aligning the mirror and SHOULD ONLY BE TOUCHED BY THE MANAGEMENT.

Mirror Bending

The mirror is bent along its length in order to focus the beam vertically. Each end of the mirror is bent independently. A lever arm is attached to the yoke in which each end of the mirror sits. By moving the opposite end of the lever arm down, a torque is applied to the end of the mirror causing it to bend. A linear feedthrough on each end of the mirror support is used to move the lever arm attached to the opposite end of the mirror, thereby bending or not bending it. When not bending the mirror it is supported freely on each end with no torque being provided by the lever arms, but it may still bend under its own weight. THE BENDING OF THE MIRROR SHOULD ALSO BE DONE BY THE MANAGEMENT ONLY.

The Hutch Slits Assembly

At the end of the beam line is a 1 inch diameter, 10 mil thick Be window. Just after this window is the hutch slits assembly. It is kept evacuated by the pump inside the hutch. The assembly can be fit with extensions of different lengths to bring this vacuum within different distances of the sample position. A set of four slits, two vertical and two horizontal, inside this assembly can be adjusted by external stepping motors controlled by

the computer. The center and separation of both vertical and horizontal pair of slits can be moved and scanned by the computer. A scintillation detector is attached to the bottom of this assembly for use as a monitor of the incident beam intensity. X-rays for this monitor detector are provided by passing the incident beam through a 1 mil thick piece of Kapton inside the assembly, placed 45° to the beam. The window at the end of this assembly is 1 inch diameter, 3 mil thick Kapton.

THE HUTCH

The hutch provides space for all experimental equipment. Standard equipment includes the diffractometer, diffractometer support table, special chambers to be mounted on the diffractometer, detectors, and the He-filled detector path.

The Diffractometer

The diffractometer is a 6-circle Huber model. The 6 circles are χ , ϕ , θ (ω), 2θ , and θ and 2θ for the analyzer. The diffractometer is shown in Figure 4.

The six motions of the diffractometer are controlled by the microsteppers. The χ , θ and 2θ motions are geared so that 1 revolution of the motor corresponds to 1° of motion. The ϕ motion is geared so that 1 revolution of the motor corresponds to 2° of motion. The microsteppers are currently splitting up one revolution of these motors into 20,000 steps.

The zeroes of the diffractometer motions are defined as follows. $\chi = 0$ when the ϕ mount is opposite to the θ table and the ϕ axis is parallel to the θ and 2θ axes. $\theta = 0$ when the plane of the χ circle is perpendicular (χ -axis is parallel) to the incoming beam. $2\theta = 0$ when the 2θ detector is receiving the incoming beam.

The sense of these motions is as follows. If we look at the diffractometer with the incoming beam coming from the right, θ and 2θ move in the positive direction when they move clockwise. If we look at the diffractometer with the incoming beam coming towards us, χ moves in the positive direction when it moves clockwise. When $\chi = 0$, ϕ moves in the positive direction when it moves in the opposite direction of θ .

Aligning the Diffractometer

The following is just one way of aligning the diffractometer in χ , θ and 2θ with the beam. It makes use of the Soller slits, which mount on the sample goniometer. Steps 1)–3) ensure that the incident beam hits the center of the diffractometer. Steps 6)–10) ensure that the plane of the χ circle is perpendicular to the beam.

- 1) Mount goniometer and cone on ϕ mount and center in Huber using scope. This is accomplished when the point of the cone remains stationary in the scope when first spinning ϕ and then χ . Note that it is not necessary for the point of the cone to be centered in the crosshairs of the scope, just that it remain in the same place while spinning ϕ and χ . Occasionally the scope gets banged or moved somehow so that the crosshairs are not still centered on the center of the diffractometer. When this happens please notify the management so that this can be fixed. Do not remove or tamper with the scope bracket attached to the χ ring.
- 2) Choose incident beam size with slits and center around hot spot of beam. This is assuming that the mirror has already been focused at the sample position. The slit size, both vertical and horizontal, can then be set and the slits can be scanned both in the vertical and horizontal so that they are centered on the brightest part of the beam. The monitor detector can be used to collect counts as a function of time while doing this. Note that, since the beam is being focused, the beam size at the sample position may not be the same as the incident beam slit size. Measure beam size at the sample position.
- 3) Center cone (and therefore Huber) in beam using using fluorescent screen, x-ray paper and diffractometer table motions.

- 4) Mount ion chamber on 2θ arm and place in position to accept beam (make sure detector slits are open wide enough).
- 5) Mount Soller slits on goniometer so that at $\chi = 0$ the blades of the Soller slits are horizontal. Make sure beam is hitting Soller slits.
- 6) Do a ϕ scan at $\chi = 0$. Move ϕ to where peak is and set $\phi = 0$ there.
- 7) Move to $\chi = 180^\circ$ and do another ϕ scan. If peak is at ϕ' move to $\phi'/2$.
- 8) Do a θ scan. There should be a peak at $\pm\phi'/2$. Move to where θ scan shows a peak and set $\theta = 0$ there. Check at $\chi = 0$.
- 9) Now go to $\chi = 90^\circ$ so that the blades of the Soller slits are vertical. Do a ϕ scan. Move ϕ to where peak is and set $\phi = 0$ there. Move to $\chi = 270^\circ$ and do another ϕ scan. If peak is at ϕ'' move to $\phi''/2$.
- 10) Do a scan with the diffractometer table yaw (rotation about the vertical). There should be a peak at $\pm\phi''/2$. Move to where the table yaw shows a peak and leave it there.

The χ zero can be found in two ways.

- 11) The first way is to just put a level on the ϕ table and adjust χ so that the ϕ table is level. This is then $\chi = 90^\circ$.
- 12) The second way is more complicated. Move to $\theta = 90^\circ$, $\chi \simeq 180$ and $\phi = 0$. Mount the Soller slits in the sample goniometer so that the blades are vertical. Scan χ and find the peak position. Move χ to the peak position and set $\chi = 180$ there. Move to $\chi = 0^\circ$. Scan χ again. If peak is at χ' , move χ to $\chi'/2$ and set $\chi = 0$ there.
- 13) In order to find the 2θ zero first set the detector slits to final size. Scan 2θ to find the peak. Move to the peak position and set $2\theta = 0$ there.

- 14) Set scatter slits a little larger than the detector slits.
- 15) Put x-ray paper before and after these pair of slits. Pictures should match so that these slits are in line with the incident beam slits.

Setting Limits on the Diffractometer

There are two types of limits which can be set on the χ , θ and 2θ motions of the diffractometer, hard limits and software limits (the ϕ motion and the 2 analyzer motions only have software limits). Hard limits (those small cylindrical things) are set to protect the diffractometer motions from running into other things or each other. Software limits should always be set so that they will be hit before a hard limit is hit. Therefore, if the software limits are set properly a hard limit should never be hit.

Hard limits should not be removed completely from the θ or 2θ motions. If it is necessary to move one, just loosen its screw, slide it along its path and then tighten its screw when it is in its new position. The χ motion generally does not have hard limits attached on a permanent basis.

There are two hard limits which are set on the 2θ motion and should not be changed. The screw heads of these are covered with wax. One prevents the 2θ arm from hitting the TV camera in the hutch and the other prevents it from hitting the diffractometer table. There is an additional one which can be set between these two to prevent the 2θ arm from going to zero and thus allowing the detector to see the direct beam. In all cases a software limit should be set to prevent this from happening when either a scintillation or silicon detector is being used. A software limit should also be set just above the highest 2θ value which will be used.

The hard limits on the θ motion are generally set to prevent the χ circle from hitting the incident beam path. If the long extension on the incident beam assembly is being used this has already been set. If the long incident beam path is not attached and this is not a problem then they may be set to just beyond where the χ circle blocks the incident beam. Software limits should be set inside of the hard limits. If negative θ will not be used the lower software limit may be set close to zero. If a chamber or something similar is mounted on the diffractometer then these limits should be appropriately set to prevent collisions.

The hard limits on the χ motion are generally not needed unless a chamber or something similar is mounted on the diffractometer. However, software limits should be used to prevent the χ circle from turning more than 180° in either direction, less if possible. This is to prevent the ϕ motor cable from being pulled too much.

Software limits should be used for the ϕ motion if a chamber or something similar is mounted on the diffractometer to prevent collisions.

The status of the hard limits on the diffractometer and the monochromator (whether one has been hit or not) is indicated on the leftmost module in the top Nimbin in the equipment rack. A red LED will go on indicating which limit has been hit. A motion can be disabled completely by turning off the appropriate switch on this module. This has the effect of both limits on that motion being hit at once so that no motion can take place.

The Diffractometer Table

The diffractometer table supports the diffractometer and provides motions to place the center of the diffractometer in the incident beam. It consists of a top section which

has five motors which provide motions for vertical height, yaw, pitch, roll and horizontal translation, all with respect to the beam. A lower table provides up to a 7 inch vertical translation for raising and lowering the diffractometer when using and not using the mirror.

The top section has a 3-point support, each which can be moved vertically by a combination of a 200 turns per inch mechanical actuator and a stepping motor. This section has a motor which rotates the table about a vertical axis passing approximately through the center of the diffractometer which is also controlled by the same mechanical actuator, stepping motor combination. There is also a motor which translates the table perpendicularly (and horizontally) to the beam. The 5 motions of the diffractometer table do not have hard limits but they do have software limits which have been set by the management and should not be changed.

These motors are used to provide the five motions of yaw, roll, pitch, vertical translation and horizontal translation for the diffractometer table.

1. Yaw - a rotation around a vertical axis passing through the center of the diffractometer.

A positive yaw is in the counter-clockwise direction when viewing from above.

2. Roll - a rotation about a horizontal axis coincident with the beam. A positive roll is in the counter-clockwise direction when looking into the beam.

3. Pitch - a rotation about a horizontal axis perpendicular to the beam. A positive pitch is in the counter-clockwise direction when viewing from the door side of the hutch.

4. Vertical translation - the raising and lowering of the table, with positive being in the up direction.

5. Horizontal translation - a motion perpendicular to the beam with positive to the right when looking into the beam (away from the door side of the hutch).

The Detectors

The beam line has two Bicron and two Harshaw scintillation detectors, two PGT silicon detectors and two ion chambers. The Harshaw scintillation detectors are reserved for use as a monitor detector of the incoming beam (one is a spare). The ion chambers should be used any time very high count rates are to be measured, such as when looking at the direct beam.

The cables from the detectors for high voltage, signal and preamp power can be plugged directly into the panel to the right of the hutch door. The connectors on this panel are connected to the equipment rack, directly into the back of the lower Nimbin (or other) electronics. Other pieces of detector electronics are available in Cabinet 1.

The Bicron scintillation detectors should be used at a voltage of about +700 volts. They are generally connected to an amplifier and then to a single channel analyzer (SCA) (the amp and SCA may be combined into one module). The windows of the SCA should be reset anytime the energy of the x-rays is changed. The output signal from the SCA can then be fed into a counter and a ratemeter. The signal from the amplifier is generally fed also into the oscilloscope for observing the voltage pulses coming from the detector.

The PGT silicon detectors must only be used at liquid nitrogen temperature or damage to them can result. The beam line has a liquid nitrogen dewar for this purpose. **THEY MUST BE COOLED AT LEAST 6 HOURS BEFORE THEY ARE FIRST USED.** Add liquid nitrogen slowly or leaks may develop in the vacuum dewar. If condensation forms on the outside of the detector lower the high voltage slowly to zero and **NOTIFY THE MANAGEMENT IMMEDIATELY.** Fill the detectors with liquid nitrogen at least every

12 hours so that they will not warm up. The silicon detectors are used with the PGT high voltage supply SET SLOWLY TO -600 VOLTS FROM ZERO. Please read the instruction manuals or ask questions if you are not familiar with these detectors so that you will not damage them. They are very expensive to repair.

Other Equipment in the Hutch

He-filled Detector Flight Path

A beam flight path from the sample to the detector is normally mounted on the 2-theta arm of the diffractometer and filled with helium. It is composed of two pieces, one which goes from the detector to the end of the dovetail and one which extends closer to the sample, and which can be removed by the management if it interferes with a sample or chamber mounted on the diffractometer. There is a 1 mil thick Kapton window on each end of the assembly. There is a set of slits mounted to the detector side of this flight path as well as a cowl into which the detector can fit to keep scattered radiation from entering it. The inside of the cowl is lined with plastic to keep it electrically isolated from the detector. A set of slits is usually mounted to the sample side of the flight path for use as scatter slits. The entire flight path is leaktight and can be filled with helium from a Polyflo tube which is hanging from the top of the hutch and is connected to a He tank strapped to the front of the hutch. The pressure of this He is usually kept at 1-1.5 PSI. The flight path can be flushed with He by lifting up on the pressure relief valve on the sample side of the tube.

Analyzer Radiation Shield

A 0.25 inch thick aluminum shield can be attached to the analyzer motion to cut down on background radiation while using an analyzer crystal. It has an attached tube which fits into the He-filled flight path and another tube, which can be set to an angle, into which a detector fits. This shield should be mounted before aligning the analyzer crystal.

Horizontal Scanning Stage

A horizontal scanning stage can be mounted on the 2θ arm in order to scan a detector in the horizontal plane. It consists of a linear motion with an angular motion mounted on top of it. 2θ in the horizontal is set by the linear motion and the angular motion is used to keep the detector and slits pointed at the sample. A detector and slits can be mounted on a narrow dovetail which mounts on top of the angular motion. Both the linear motion and angular motion are driven by stepping motors which can be controlled by the computer by connecting these motors to the analyzer motor cables. At present there is no software which coordinates these two motions so they must be controlled by using an external file scan. There are lead bricks which must be mounted on the opposite side of the 2θ arm in order to counterbalance the extra weight of the horizontal scanning stage. If you need to use this piece of equipment please notify the management prior to your arrival.

Diffraction Accessories

There are pieces of equipment either inside of the hutch or in Cabinet 1 which can be used on the diffractometer. These include:

- 1) Soller slits

- 2) Ge(111) crystal (mounted on sample holder)
- 3) highly ordered pyrolytic graphite (HOPG) (mounted on sample holder)
- 4) Silicon powder
- 5) two large Huber goniometers which fit on the Huber phi mount
- 6) several aluminum sample mounts which fit on the goniometers above
- 7) lead foil for shielding
- 8) extra sets of Huber slits
- 9) goniometer extensions

If you can't find something please ask.

SUPPORT EQUIPMENT

The equipment in this section includes all that is normally located outside of the hutch. This includes motor indexers and drivers, computers and electronics stored in the equipment rack and elsewhere.

Motor Indexers and Drivers

Motors on the beam line are run by either the Superior Electric Modulynx microstepping and normal stepping indexers or digital I/O signals from a PC board (Real Time Devices, Inc., model DG96). The Modulynx microstepping indexers are running the 6 diffractometer motors and the monochromator motor. The Modulynx normal stepping indexers are running the 5 diffractometer table motors. The PC board motor indexer is running the beam line slits motors, the mirror motors and the 4 hutch incident beam slits motors.

The microsteppers have given us few problems in recent years. This is due to several steps which have been taken. The power lines to the driver cards are fused so they will not be able to draw too much power from the power supply. Hard limits are connected on both the diffractometer and monochromator. In addition, there is a master enable-disable switch to the right of the hutch door which, when moved down, will close all the limit switches at once. There is also a module in the top Nimbin on the rack, labeled ' μ STEPPER STATUS', that indicates which hard limit has been hit for the microsteppers

and from which any of the microstepped motors can be disabled individually by turning off the appropriate switch (both red LEDs will go on). Finally, software limits can be set.

A second module to the right of the microstepper module, labeled 'MODULYNX STATUS', indicates the status (green LED for motor motion and yellow LED for direction) of the motors run by the Modulynx normal stepping indexers.

If any of the motors run by the Modulynx indexers refuses to move first check both software and hard limits. If this isn't the cause it is most likely a software problem or a blown fuse, either one in-line to the driver cards or the power supplies for the driver cards located at the bottom of the rack. Please notify the management if this happens.

The signals from the PC board motor indexer first run through a module in the topmost Nimbin in the electronics rack. The module gives information about eight motors. The switch can be used to enable or disable a motor. Both red LEDs will light up when a motor is disabled. The red LEDs will also light up when a hard limit has been hit. IT IS IMPORTANT TO PLACE THE APPROPRIATE SWITCH IN THE UP POSITION BEFORE MOVING THE MOTORS THEY ARE CONNECTED TO, otherwise positions may be lost. Please return the switch to the down position when you are finished moving these motors.

The drivers for the slits motors are mounted on the front wall of the hutch to the left of the TV camera. The drivers for the beam line slits and mirror motors are located in the third rack in front of the hutch. THE POWER FOR THESE LAST DRIVERS MUST BE TURNED ON USING THE SWITCH TO THE LEFT OF THE OSCILLOSCOPE (the blue light will come on) BEFORE MOVING THESE MOTORS, otherwise positions may be lost. When not using these motors for a long period of time please turn off the power

to them.

Electronics

The electronics available at the beam line are listed below. Information about any of this equipment is available in equipment manuals in the beam line filing cabinet. Please realize that at any one time one or more of these may be out for repair. If any of them is crucial to your experiment please call before coming to Brookhaven.

Item	Quantity
Power Designs High Voltage Supply (0-3000V)	2
Ortec 556 High Voltage Supply (0-3000V)	1
PGT Bias Supply	2
GSK Scientific P11-10 (for ion chamber)	2
Tennelec TC246 AMP/SCA	3
Canberra 1718 X-Ray Amplifier Pulse Height Analyzer	1
Canberra 2020 Spectroscopy Amplifier	1
Ortec 672 Spectroscopy Amplifier	1
Ortec 460 Delay Line Amplifier	1
Canberra 2035A Constant Fraction Timing SCA	2
Ortec 550A SCA	1
Tennelec TC525 Ratemeter	2
Canberra 2081 Ratemeter	2

Ortec 974 Quad Counter Timer	2
Canberra 2071 Dual Counter Timer	2
Tennelec TC535P Timer/Multi-Scaler	1
BNC DB-2 Random Pulse Generator	1
Ortec 495 Power Supply	1
Canberra 2000 Nimbin	1
Tennelec TB3 Nimbin	1
Tektronix 2215 60Mhz Oscilloscope	1
Hewlett-Packard HP3478A Multimeter	1

Pumps

We have several pumps available at the beam line. An Alcatel direct-drive rotary vane pump can be used for pumping out the cryostat or other chambers requiring high vacuum. It has a connection for a Kwik-Flange 25 fitting. We have two small Sargent-Welch direct-drive rotary vane pumps for use in pumping on small chambers or anything else not requiring high vacuum. When using these pumps please make sure that your vacuum hoses are securely attached and your chamber is reasonably leaktight. In addition, we have a Leybold turbo pump which can be used for pumping out UHV systems. It should not be used to pump out anything with rubber hoses.

LOW TEMPERATURE STAGE

The low temperature stage is made by CTI and is capable of providing temperatures down to about 10 K. It can be mounted on the diffractometer and is fitted with Be windows which allow x-rays 360° access to the sample. The temperature of the sample holder in the low temperature stage is measured by two silicon diode thermometers, one which has been calibrated by LakeShore Cryotronics. 4-point wire connections are made directly to the temperature sensors so as to eliminate lead contributions. The temperature of the low temperature stage is controlled by a LakeShore model DRC-82C temperature controller which can be controlled by the computer. Sample holders are available and should be requested before coming to use the low temperature stage.

THE COMPUTER

There are two personal computers at the beam line. Each one has an 80386-16 processor, an 80287 math coprocessor, a 70 MB hard disk, a 1.2 MB 5.25 inch floppy drive, a 1.44 MB 3.5 inch floppy drive, a tape backup and VGA graphics with a VGA color monitor. Computer 1 has 1 Meg of memory and computer 2 has 3 Meg of memory. Both are connected to a Hewlett-Packard LaserJet III laser printer. One computer is used to control experiments (computer 1) and the other (computer 2) can be used for any other purpose. All of your work should go into your own directory and only in your own directory.

Information about the XSCAN program is available in help files inside the software. Computer 1 also contains a multi-channel analyzer card which can be used to collect energy spectra from a detector. Both computers 1 and 2 are connected to Ethernet to provide access to the computer networks. We have an account on the NSLS Vax which can be used for e-mail. Please ask for directions on how log onto this computer.

After finishing your experiment please remember to copy your data to bring home. Don't remove your data from the computer's hard disk. As a precaution, we make it a practice to back-up everyone's data onto their own tape cartridge after they leave. However, we only back-up data which we find in your directory on computer 1. So if you have data anywhere else and would like it backed up please move it to this location. These tape cartridges are kept at Brookhaven.

APPENDIX A

Monochromator Calibration

The calibration of the monochromator has been done by measuring the absorption edge of Cu in the transmission geometry with an ion chamber. This scan is shown in Figure A1.

The calibration of the monochromator can be checked by comparing the readout of the up-down counter at the top of the electronics rack with the angle of the monochromator given by the computer. It can also be checked by reading the dials on the monochromator Huber. The dials can be read to about 0.0005 degrees. THE MONOCHROMATOR CALIBRATION ON THE COMPUTER SHOULD BE CHANGED ONLY BY THE MANAGEMENT.

Transmission EXAFS Scan
through Cu Foil

Figure A1

APPENDIX B

Monitor Counts versus Energy

A measurement was made of the monitor counts per second versus the energy of the x-rays using the focusing mirror. This was done by changing the energy, then peaking the intensity by retuning the monitor detector SCA, the second monochromator piezoelectric crystals and the mirror. The counts were measured on a ratemeter and normalized to the ring current. The monitor detector is a scintillation detector looking at the scattered x-rays from a 1 mil thick piece of Kapton at 45 degrees to the incident beam. No attempt was made to correct for the energy dependence of the x-ray scattering from the Kapton. This measurement is shown in Figure B1.

Notice the intensity dying off at about 12 keV, the energy cutoff of the mirror. The large dip at about 8.5 keV is somewhat of a mystery. This dip is not seen without the mirror in place.

This plot is meant to be an aid in choosing an x-ray energy at which to work. If the energy used is not critical and intensity is important, it may be wise to work at an energy which gives the most intensity.

Beam Intensity with Focusing
Mirror versus Energy

Figure B1

Vertical Cross-Section of
Focused Beam at Sample Position

Figure C1

APPENDIX C

Focused Beam Cross-Section

The cross-section of the focused beam is shown in the horizontal and vertical in Figures C1 and C2 and was done in the following way. The ion chamber was mounted on the 2-theta arm with only one set of slits in use, these being mounted at the sample position, and was set to accept the direct beam. With these slits set at 2 mm wide in the horizontal and wide open in the vertical, the mirror angle was adjusted for maximum intensity. The slits were then set at 2 mm high in the vertical and wide open in the horizontal and the mirror bending was adjusted for maximum intensity. After this procedure, pictures were taken before, at, and after the slits position to be certain that the smallest beam, and therefore the focal point, was at the slits.

The slits were again mounted at the sample position. The vertical slit size was set at 0.1 mm, with the horizontal wide open, and this slit was scanned vertically to map out the cross-section of the focused beam in the vertical. The bend of the mirror was adjusted so that the vertical cross-section of the beam was a minimum. The horizontal slit size was then set at 0.1 mm, with the vertical wide open, and this slit was scanned horizontally to map out the cross-section of the beam in the horizontal. The angle of the mirror was adjusted so that the horizontal cross-section of the beam was a minimum. The minimum cross-section achieved (the best focus) in the vertical was about 1.6 mm FWHM and the minimum cross-section achieved in the horizontal was about 1.1 mm FWHM.

Horizontal Cross-Section of
Focused Beam at Sample Position

Figure C2

APPENDIX D

Beam Divergence

The divergence of the focused beam was measured in the vertical and horizontal, at an energy of 4.3 keV, in the following way. A piece of single crystal Si, placed in a holder with a 1 mm diameter hole was mounted at the center of the diffractometer, and in the beam, so that the beam went through the hole. With $\chi = 0^\circ$ and $2\theta = 0^\circ$, ϕ was scanned until the Bragg condition for the (220) reflection was satisfied. The angular width over which anomalous transmission take place is a fraction of an arcsecond so that the width of the peak corresponds to the vertical divergence of the beam. This was repeated at $\chi = 90^\circ$ to measure the horizontal divergence of the beam. These plots are shown in Figures D1 and D2. The measured divergence from the FWHM of these peaks is 0.2 milliradians in the vertical and 0.9 milliradians in the horizontal.

Figure D1

Figure D2

APPENDIX E

XSCAN Default Setup Parameters

The default setup parameters for the XSCAN program are listed in the pages that follow. If you are unsure of the parameters on the computer please refer to this appendix. Bear in mind that these are not the 'right' parameters in all cases. Some parameters might have been changed to suit a particular experiment and the motion and motor positions change all the time. DO NOT CHANGE THE VALUES WHICH ARE IN THE CROSS-HATCHED AREAS. If you have any questions please contact the management.

File Setup Scan Calc Fit Quit

Huber Motion
Table Motion
Table Motors

Huber Motion Setup

name	axis	steps/deg	b_rate	h_rate	accel	l_limit	h_limit	position
------	------	-----------	--------	--------	-------	---------	---------	----------

chi	U	-20000	0.25	2.00	1.00	-90.000	270.000	90.000
phi	V	-10000	0.25	4.00	1.00	-360.000	360.000	0.000
theta	W	-20000	0.25	2.00	1.00	0.000	44.000	0.000
theta	X	-20000	0.25	2.00	1.00	5.000	90.000	10.000
energy	Y	-200000	0.25	4.00	1.00	5.670	29.620	12.721
theta	Z	-20000	0.25	4.00	1.00	-45.000	45.000	0.000

[F]-Help

[Esc]-Exit

File	Setup	Scan	Calc	Fit	Quit
Huber Motion					
Table Motion					
Table Motors					
Aux	Table Motion Setup]				
Aux	name	l_limit	h_limit	position	
MCA					
Tenp	yaw	-2.000	2.000	0.000	
	roll	-2.000	2.000	0.000	
	pitch	-2.000	2.000	0.000	
	vtrans	-10.000	10.000	0.000	
	htrans	-10.000	10.000	0.000	
[Pi-Help] [Esc-Exit]					

File Setup Scan Calc Fit Quit

Huber Motion
Table Motion
Table Motors

[Table Motors Setup]

name axis steps/mm b_rate h_rate accel l_limit h_limit position

rot	U	-3150	1.00	5.00	2.50	-40.000	40.000	0.000
trans	V	-3150	1.00	5.00	2.50	-40.000	40.000	0.000
inup	W	-3150	1.00	5.00	2.50	-40.000	40.000	0.000
indn	X	-3150	1.00	5.00	2.50	-40.000	40.000	0.000
out	Y	-3150	1.00	5.00	2.50	-40.000	40.000	0.000
attheta	Z	-20000	0.25	4.00	1.00	-45.000	45.000	0.000

[F1=Help]

[Esc=Exit]

File	Setup	Scan	Calc	Fit	Quit
Huber Motion					
Table Motion					
Table Motors					
Aux	Aux Motion A Setup]				
Aux	name	l_limit	h_limit	position	
MCA					
Temp	vcenter	-5.000	5.000	0.000	
	vsize	0.000	24.000	1.000	
	hcenter	-5.000	5.000	0.000	
	hsize	0.000	24.000	1.000	
	n_vert	-5.000	10.000	2.040	
	n_angle	0.000	10.000	7.000	
	hblslit	-10.000	10.000	0.000	
	vblslit	-10.000	10.000	0.000	
[F]-Help [Esc]-Exit					

File Setup Scan Calc Fit Quit

Huber Motion
Table Motion
Table Motors

[Aux Motors A Setup]

name axis steps/mm b_rate h_rate accel l_limit h_limit position

vuslit	0	800	0.50	1.00	0.50	-3.000	12.000	0.500
vbslit	1	800	0.50	1.00	0.50	-3.000	12.000	0.500
hlslit	2	800	0.50	1.00	0.50	-3.000	12.000	0.500
hrsli	3	800	0.50	1.00	0.50	-3.000	12.000	0.500
dnirror	4	15748	1.00	4.00	1.00	-10.000	10.000	43.281
unirror	5	15748	1.00	4.00	1.00	-10.000	10.000	39.185
hblslit	6	630	1.00	4.00	1.00	-10.000	10.000	0.000
vblslit	7	630	1.00	4.00	1.00	-10.000	10.000	0.000

[F1=Help]

[Esc=Exit]

**THE MATRIX X-18A
BEAM LINE USER'S GUIDE**

Version 1.0 5/17/93

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The MATRIX X-18A Beam Line at the National Synchrotron Light Source presently accommodates a Principal Research Team from Purdue University, Northwestern University, University of Illinois and University of Missouri, and a host of General Users nationwide. In the interest of better serving the User community, the MATRIX Supervisory staff at BNL have prepared this document to serve as a guide for all Users who require use of the X-18A beam line.

1. ROLE OF MATRIX SUPERVISORY STAFF AT BNL

The resident MATRIX X-18A Beam Line Supervisor and Assistant are responsible for the coordination of any maintenance, design changes, and upgrades to the equipment and software. They are also responsible for enforcing the policies set forth by the MATRIX Executive Committee, those in this document as well as specific policies generated by the NSLS User Administration Office.

Safety is everyone's responsibility. The Beam Line Supervisor and Assistant ensure that their equipment is designed and maintained to provide a safe working environment and that Users receive adequate training for safely conducting their experiments. They also ensure that Users obey applicable NSLS and BNL safety rules and follow proper procedures.

2. MATRIX DOCUMENTATION

In addition to this Guide, Users are responsible for knowing the contents of the following documents:

A. MATRIX X-18A Beam Line Manual

This provides a general overview of the MATRIX X-18A Beam Line by describing the major components of the system and offering some advice as to its use. Beam line specifications are also included here.

B. User Check Lists

Documents to record Users' knowledge and acceptance of MATRIX BEAM LINE USE RULES, and to ensure that equipment is provided and returned in proper working condition (Appendices B and C).

C. Housing Manual

Describes the general policies and procedures when occupying the MATRIX apartment.

A copy of these documents can be obtained by contacting the MATRIX Beam Line Assistant. Documents can be mailed or e-mailed provided that the User supplies a suitable address.

3. BEFORE ARRIVAL

A. Review of MATRIX Documentation

It is important that you familiarize yourself with the MATRIX documentation. Users will be required to sign a check list stating their acceptance of the MATRIX rules before they can begin an experiment.

B. Communication

It is crucial that Users check with MATRIX personnel before departing for BNL. Contact can be made by e-mail, telephone, or U.S. Mail.

i. Trip Confirmation

Users should contact the MATRIX Beam Line Supervisor at least one week in advance to confirm arrival dates. **BEAM TIME IS PRECIOUS! DELAYS OR CANCELLATIONS SHOULD BE COMMUNICATED TO MATRIX PERSONNEL AS EARLY AS POSSIBLE.** The additional time could be used to perform needed modifications or repairs to the beam line, or, it could be allocated to another User.

ii. Equipment Requirements

Not all equipment may be functional at all times. Some may be out for repairs. **USERS SHOULD CONFIRM EQUIPMENT AND BEAM LINE CONFIGURATION REQUESTS WITH THE MATRIX SUPERVISOR AT LEAST ONE WEEK IN ADVANCE.** This allows MATRIX personnel to best accommodate User's needs should a problem arise prior to their arrival. Confirm by e-mail, telephone, or U.S. Mail.

iii. Hazardous Materials

If Users need the use of a chemical hood or special storage space for their hazardous materials, arrangements should be made in advance with the MATRIX Beam Line Supervisor.

4. UPON ARRIVAL

A. Obtaining Beam Line Access

The MATRIX hutch door, cabinets, toolbox, and computer keyboards will be locked until the User notifies MATRIX personnel of their arrival. After the User receives training (if needed) and fills out the User Check-In list, keys for the above items will be issued to the User team leader. (Note: General Users do not have access to the cabinets.)

B. Training

All Users who lack adequate experience on the X-18A Beam Line will require training. MATRIX supervisory personnel will conduct the training session with emphasis on equipment operation, beam line procedures, and safety. A list of trained Users will be maintained by the resident Beam Line Supervisor and will be made available to DOE and NSLS inspectors. The Supervisor may

decide that certain Users require additional training if they fail to demonstrate competency at any time during their appointed stay.

C. Equipment Check

The Management checks the status of the equipment before the User arrives, and after the User leaves. **USERS ARE RESPONSIBLE FOR REPORTING ANY EQUIPMENT THAT IS DAMAGED OR LOST DURING THEIR STAY. Also, USERS ARE NOT TO LOAN OUT ANY EQUIPMENT WITHOUT PERMISSION OF THE MATRIX SUPERVISORY STAFF.**

5. USE OF THE MATRIX BEAM LINE

A. Technical Support

MATRIX personnel are available to provide limited technical support for beam line Users - not to do the User's experiment! MATRIX personnel **MUST** be consulted whenever the User wishes to modify any aspect of the X-18A Beam Line system. This includes mounting sample chambers on the diffractometer, routing electrical cables through the hatch, and moving beam line equipment to accommodate experimental set-ups. Although these operations may seem trivial from a User standpoint, MATRIX personnel would prefer to assist in such tasks. They have a greater knowledge of the equipment and its limitations, and can work around problem spots that may not be apparent to the User.

B. Beam Line Communications During an Experiment

Users can contact the MATRIX Beam Line Supervisor and Assistant by calling them on the phone (they have answering machines) or leaving messages by e-mail on their computers (checked at least once a day). This information is given at the end of this Guide. MATRIX personnel will visit the X-18A Beam Line on a daily schedule to check of the status the equipment and to ensure experiments are performed in a safe and organized manner. Users should state their concerns at this time to receive personalized attention.

When at all possible, Users should supply a phone number or e-mail address where they can be reached in addition to their address so MATRIX personnel can communicate with them. There have been numerous times when important information gathered from weekly NSLS user meetings could not be transmitted to the User because he/she was not available. Updated information on planned outages, maintenance, and operation schedules could be used to optimize beam time at NSLS.

C. Housekeeping and Safety Concerns

Maintaining a safe and clean work area is of utmost importance to NSLS and MATRIX personnel. NSLS has Safety personnel to oversee and monitor safety of operations and experiments, hazardous materials storage and collection, industrial hygiene, and compliance with safety requirements; and a Housekeeping Committee to monitor housekeeping and cleanliness issues. MATRIX personnel are responsible for monitoring User safety and cleanliness at the X-18A Beam Line on a daily basis. The User is expected to abide by the following guidelines as well as those listed in Appendix A.

i. Accidents and Injury

If a User becomes injured while working at the MATRIX X-18A Beam Line, the Beam Line Supervisor should be notified of the occurrence. MATRIX personnel will use this information to determine if the existing hardware and/or Rules need be modified to prevent such accidents from occurring in the future.

ii. Hazardous Materials

All containers of hazardous materials shall be properly packaged, labeled, and stored. Material Safety Data Sheets (MSDS's) should be on hand and available for inspection. An inventory of hazardous materials is made when the User arrives at the beam line. Upon departure, MATRIX personnel will ensure that the User intends to package and ship hazardous materials back to his or her home institution according to DOT regulations.

iii. Radiation Badges

Users must wear valid radiation badges when working at NSLS.

iv. Electrical Safety

All User electrical equipment must be properly grounded. Cables and wires must be secure and out of the way of walkways and passages.

v. Fabrication

The X-18A work table should be used for all electrical and mechanical fabrication including soldering, drilling, and wiring. The User is referred to the NSLS User Shop located in the basement of Bldg. 535C when larger jobs are to be performed. Hazardous material work should be performed under a hood. Arrangements should be made with MATRIX personnel prior to the User's arrival if this is a concern.

vi. Security

The User is responsible for locking the cabinets and tool boxes whenever the X-18A Beam Line is left unattended. MATRIX personnel will check for this when visiting the beam line.

vii. Computer

Users are responsible for all their data and work files in the specified User directory. Other directories should not be altered. MATRIX personnel will delete any User files found in non-specified directories. Computer viruses have been a problem at the X-18A Beam Line. Users should virus check all files read from floppies and networks. Type "virhunt" and follow the instructions.

Parameters in the XSCAN program should not be changed by the User except as stated in the MATRIX X-18A Beam Line Manual. If a problem occurs, refer to the Beam Line Manual for a template of the standard XSCAN parameters.

USER SHOULD SET SOFTWARE AND HARDWARE LIMITS SUCH THAT THE 2-THETA DETECTOR DOES NOT SEE THE DIRECT BEAM.

viii. Food and Drink

All food and drink should be kept away from the computers and the printer. MATRIX personnel will check that the desks are free of foodstuffs, soda cans, coffee cups, and other liquids. Use of the lunch room around the corner is suggested when consuming food and drink.

ix. Emergencies

An intermittent alarm sounding at a rate of approximately 1 per second means that the building must be evacuated. The nearest outside exit to the X-18A Beam Line is located at the loading dock at the Northwest entrance to the building (where the liquid nitrogen tank is kept). There are two way to get there, depending on the circumstances. The first choice would be to walk down the corridor where the lunchroom is, turn left and follow the walkway until you see the exit door on your right (where the radiation badges are kept). The second choice (if the lunchroom corridor is blocked) would be to go through the exit door just to the right of our red toolboxes. This puts you in the Angiography Suite. Walk straight, make a quick jog to the right, and go through a second marked exit door. Turn left and follow the corridor until you see the outside exit door on the right as described above. Once outside, report to the front of the building where an accounting will be made. The nearest fire extinguisher and eyewash station is located around the corner in the hall where the lunchroom is. The nearest fire alarm is located in the corridor leading to the basement of Building 535C. To phone in fire and medical emergencies, dial 2222 anywhere within the Lab.

D. The Hutch and the Equipment Rack

Users Should not disturb the cables in back of the equipment rack. In the hutch, make sure that the cable on the diffractometer table to the 2-theta detector and other cables do not get caught in the 2-theta arm movement.

PLEASE REFRAIN FROM USING TAPE ON THE DIFFRACTOMETER.

E. Property Damage

The User is responsible for reporting any item that is damaged or missing from the X-18A Beam Line during their appointed stay. A thorough equipment check is made by MATRIX personnel after the User leaves, so it is important that Users report damaged or missing items promptly. If MATRIX determines that the damage was caused by negligence or abuse, arrangements will be made for appropriate reimbursement. If it is determined that the loss or damage was not the fault of the User, MATRIX personnel will bear the responsibility for replacing or repairing these items.

F. MATRIX Rules and Regulations

The User is required to abide by the rules and regulations stated in this Guide as well as those listed in the Matrix Beam Line Use Rules in Appendix A. The Rules list is supplied to the User prior to arrival and is also posted on the hutch door.

G. User Rewards and Punishments

Users who follow MATRIX Rules and Regulations will be rewarded. MATRIX will give a conscientious User higher priority when being scheduled for beam time. At the same time, Users who violate Matrix Rules and Regulations will be given lower priority when being scheduled for beam time and may be subject to certain penalties. Greater responsibilities will be afforded to Users who conduct themselves in a responsible and professional manner.

6. UPON DEPARTURE

A. Clean Up

Users are expected to return the X-18A Beam Line to the same orderly condition as when they first arrived. The floor should be swept of debris and any stains and spills cleaned up. Any tape used in the experiment should be removed and the surface cleaned of glue residue. **THERE SHOULD NOT BE ANY TAPE OR GLUE ON THE DIFFRACTOMETER!** Tools should be returned to their respective places and tool sets such as drills and hex keys should be complete and in order. The computer equipment should be cleaned of any stains and the desks should be clean and free of clutter. Users should make a copy of their specified directories. (MATRIX will make its own backup and delete all other extraneous files.) The keys should be returned to the MATRIX Beam Line Supervisor.

B. Completion of User Check-Out List

The User will complete the User Check-Out List and return it to MATRIX personnel. Information will be checked against a MATRIX check sheet after the User has left.

7. GETTING IN TOUCH WITH MATRIX PERSONNEL

A. Prof. Gerald Liedl, MATRIX Beam Line Spokesperson

- i. Phone: (317) 494-4100
- ii. Mail: Purdue University
School of Materials Engineering
1289 MSEE Building
West Lafayette, IN 47907-1289
- iii. E-Mail: liedl@ecn.purdue.edu

B. Dr. Steven Ehrlich, MATRIX Beam Line Supervisor

- i. Phone: (516) 282-7862
- ii. Mail: NSLS, Bldg., 510E
Brookhaven National Laboratory
Upton, NY 11973
- iii. E-Mail: ehrich@bnlx17.nsls.bnl.gov

C. John A. Schwanof, MATRIX Beam Line Assistant

- i. Phone: (516) 282-5635

ii. Mail: NSLS, Bldg., 510E
Brookhaven National Laboratory
Upton, NY 11973

iii. E-Mail: schwanof@bnlls1.nsls.bnl.gov

D. MATRIX X-18A BEAM LINE

i. Phone: (516) 282-5618

ii. Mail: NSLS Person's Name
NSLS, Bldg. 725A
Beam Line X-18A
Brookhaven National Laboratory
Upton, NY 11973

E. MATRIX APARTMENT AT BNL ON-SITE HOUSING

i. Phone: (516) 341-1004

ii. Location: Apartment 2B
York Lane
Brookhaven National Laboratory
Upton, NY 11973

APPENDIX A

MATRIX BEAM LINE USE RULES

- Never load or read software into the computer without virus checking it first. Type "virhunt" in the virhunt directory and follow instructions.
- Create/edit/delete files only in the specified User directory. Any files found in the root or other directories will be erased.
- Do not change parameters in the XSCAN program except as stated in the MATRIX X-18A Beam Line Manual. Refer to the Manual for a template of the standard XSCAN parameters.
- Do not modify any X-18A Beam Line equipment without consulting management first. This includes motors and any hardware with screws that are covered with wax or specially labeled. MATRIX personnel will be present when Users mount their chambers on the diffractometer. If you encounter a problem with the equipment or would like to have something changed, call the Management. Refrain from using tape on the diffractometer.
- Do not change the permanent hard limits on the diffractometer (these are covered with wax).
- Do not disturb the cables in back of the equipment rack and ensure that cables in the hutch do not get caught in the 2-theta arm or on the diffractometer table.
- Make sure all hazardous materials are properly packaged, labeled, and stored. Keep MSDS's on file and ship according to DOT regulations.
- Wear your radiation badge at all times.
- Ensure that all User electrical equipment is grounded; clear cables and wires from walkways.
- All soldering, drilling, wiring, and other electrical/mechanical fabrication should be performed on the work table and not on the computer table. For larger jobs, use the NSLS User Shop.
- Keep food and drink away from the computers and the printer. Use the kitchen around the corner for meals.
- Promptly clean floor, desk, and equipment surfaces of stains, spills, and solder splashes.
- CLEAN UP BEFORE YOU LEAVE. Remove tape and debris from all surfaces and put tools back in their proper locations.
- Never leave the equipment cabinet or tool box unlocked when not present at the beam line.

APPENDIX B

USER CHECK-IN LIST

- [] I have read the MATRIX X-18A Beam Line User's Guide, MATRIX X-18A Beam Line Manual and the Housing Manual, and accept the Rules and Regulations stated therein.
- [] I have read the MATRIX Beam Line Use Rules and agree to abide by them.

USER'S NAME _____ DATE _____

APPENDIX C

USER CHECK-OUT LIST

- [] I did not load or read software into the computer without virus checking it first.
- [] I did not create/edit/delete files only in the specified User directory.
- [] I did not change parameters in the XSCAN program except as stated in the MATRIX X-18A Beam Line Manual.
- [] I did not modify X-18A Beam Line equipment without consulting management first.
- [] I did not change the permanent hard limits on the diffractometer.
- [] All hazardous materials were properly packaged, labeled, stored, and shipped according to DOT regulations. The appropriate MSDS's were kept on file at the Beam Line.
- [] I did thoroughly clean the area by removing tape, debris and stains from all surfaces and returning all tools back to their proper locations.

USER'S NAME _____ DATE _____

DATE

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END