

CHESS

a c t i v i t y r e p o r t

Cornell University

Ithaca, NY

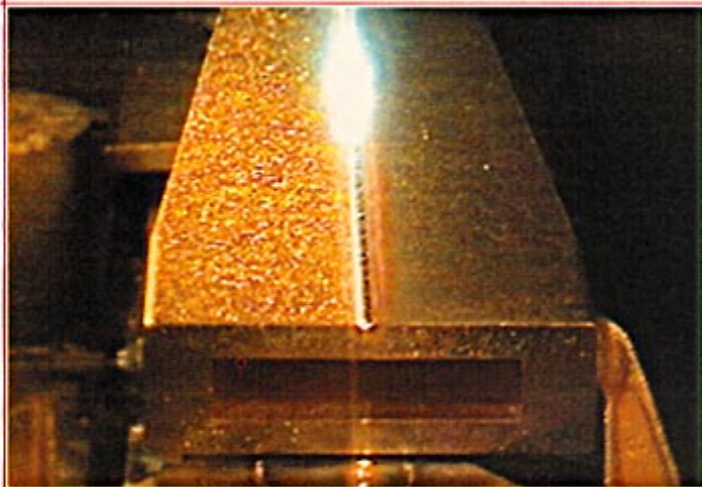
Cornell
High
Energy
Synchrotron
Source

Winter 1993

The new West A-line wiggler



Copper beamstop for new A-line



Announcing a Fall 1993 dedicated CHESS-APS undulator run (see page 15)

Electron-beam welding of the new A-line tapered copper mask

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This is the first illustrated activity report on scientific and technical developments taking place at the Cornell High Energy Synchrotron Source. These pages bear witness to a tremendously active period of development at CHESSE. This report includes general information and announcements, labwide technical projects and developments, and scientific advances under both the MacCHESSE heading and at-large.

The goal of this publication is to familiarize users with the facilities and breadth of science that can be done at CHESSE. In the comfortable and informal CHESSE style, many articles discuss work that is *ongoing*, but are included to raise awareness of the new and future capabilities of CHESSE.

The next activity report will be published following the upcoming CHESSE Users' Meeting (June 22-23, 1993). It will include the scientific and technical progress reported at that meeting, as well as a completely updated facility description for all CHESSE beamlines.

We actively invite (and may sometimes coerce!) users to submit reports of work for publication. For more information, or with comments on this report, call me at 607-255-2959.

 *Ernie Fontes*
Editor & Staff Scientist



CHES

director's message

Bob Batterman

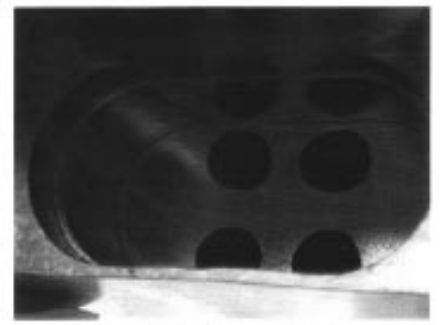
This CHES *Activity Report* begins a more timely information flow concerning developments at CHES. Ernie Fontes joined the CHES staff from Bell Laboratories this past year and is the editor of this communication. Besides informing our users of the capabilities of this laboratory, we hope to encourage feedback so that CHES may develop to its fullest potential as a synchrotron radiation user facility.

The contributed articles show that the scientific endeavors at CHES are as strong and numerous as ever. I bring to your attention several major technical developments taking place within CHES at present. In one way or another, all are affected by the aggressive plans of the Wilson Laboratory to dramatically increase the electron and positron currents stored in CESR. These upgrades are part of a long term project carried out by the High Energy Physics program to explore

the feasibility of building a B-factory, which could ultimately store 1 Amp of current at 8 GeV energy!

In order to deliver more usable x-ray flux from these increased currents, a significant effort is being expended to design accelerator and x-ray optical components that can tolerate the unprecedented heat loads that inevitably accompany the quest for higher currents and photon flux. If the near-term goal of 500 mA current is achieved, the present CHES wigglers will produce ~32 kilowatts of x-ray power! Designing beamstops, x-ray windows, and other beamline containment components for this powerful beam is no simple matter.

Within the CHES West laboratory a significant modernization of the original A-line is currently underway. Our high-field 6-pole electromagnetic wiggler is being replaced by a recently constructed 25-pole permanent magnet device, similar to the one in CHES East. A-line will be totally rebuilt to give us a new tunable doubly-focussed A1 station, similar to the highly successful F1 station. A2 will be a high resolution diffraction station with mirror focussing and a significantly enlarged hutch. With the capability for doubly-focussed wiggler white beam, the A2 station will not only be at the cutting edge of scientific research, but will also provide a premier x-ray heat-

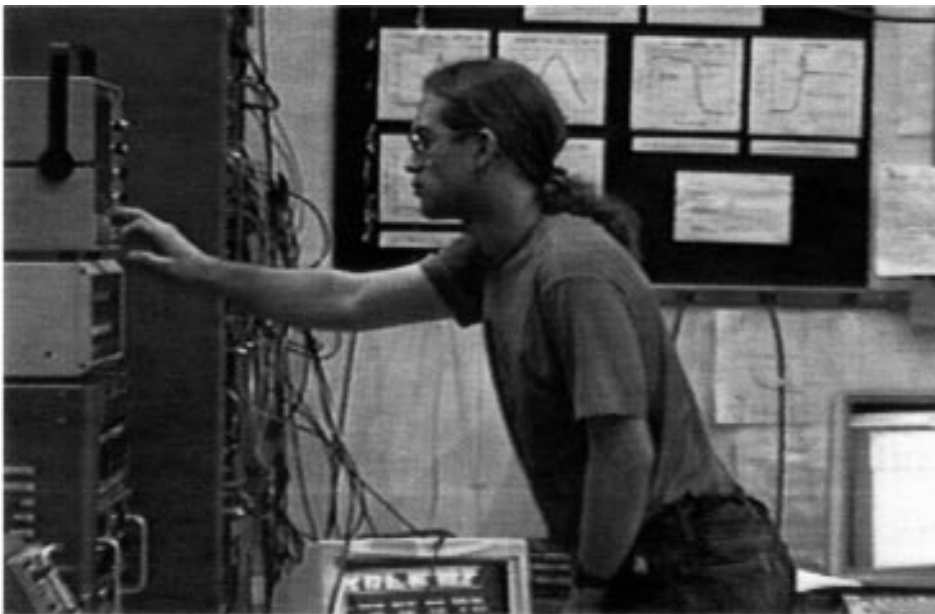


Water channels inside new A-line copper beamstop, before manifold and vacuum seal brazing.

load testing facility useful for the development of beamline components for future x-ray sources. Construction is presently underway, and we anticipate full beamline commissioning in June 1993.

On the MacCHES front, we have recently purchased a Fuji storage phosphor scanner. The successful operation of the F1 station for macromolecular crystallography has raised the level of excitement within CHES as well as increased demand for beamtime. The Fuji scanner is faster than the Kodak system, enabling users to make the best use of their available time. We are requesting funds from NIH to purchase a second Fuji scanner so that we may replace altogether the Kodak system and further increase data collection efficiency.

The development of new beam lines with unprecedented power levels, the upcoming undulator run, and the prospects for exciting new user and staff research produce a high level of anticipation and excitement at CHES.



Bob Batterman

New stations, optics and wiggler

Don Bilderback
Joel Brock
Bill Podulka

We intend to complete a major upgrade of A-line in the spring of 1993. The purpose of the upgrade is to make the stations more powerful and capable, and to handle the much higher CESR currents planned over the next several years. The plan consists of three major parts: a new 25-pole wiggler source, a new UHV beamline, and new stations (A1, A2, and A3) shown below.

Wiggler Upgrade. The current 6-pole, 1.5 T wiggler is being replaced by a 25-pole 1.2 T permanent magnet wiggler (see cover photo). The magnetic design is identical to the F-line wiggler (Rev. Sci. Instr. 63 (1), 305, (1992)), however the A-line device incorporates mechanical features which

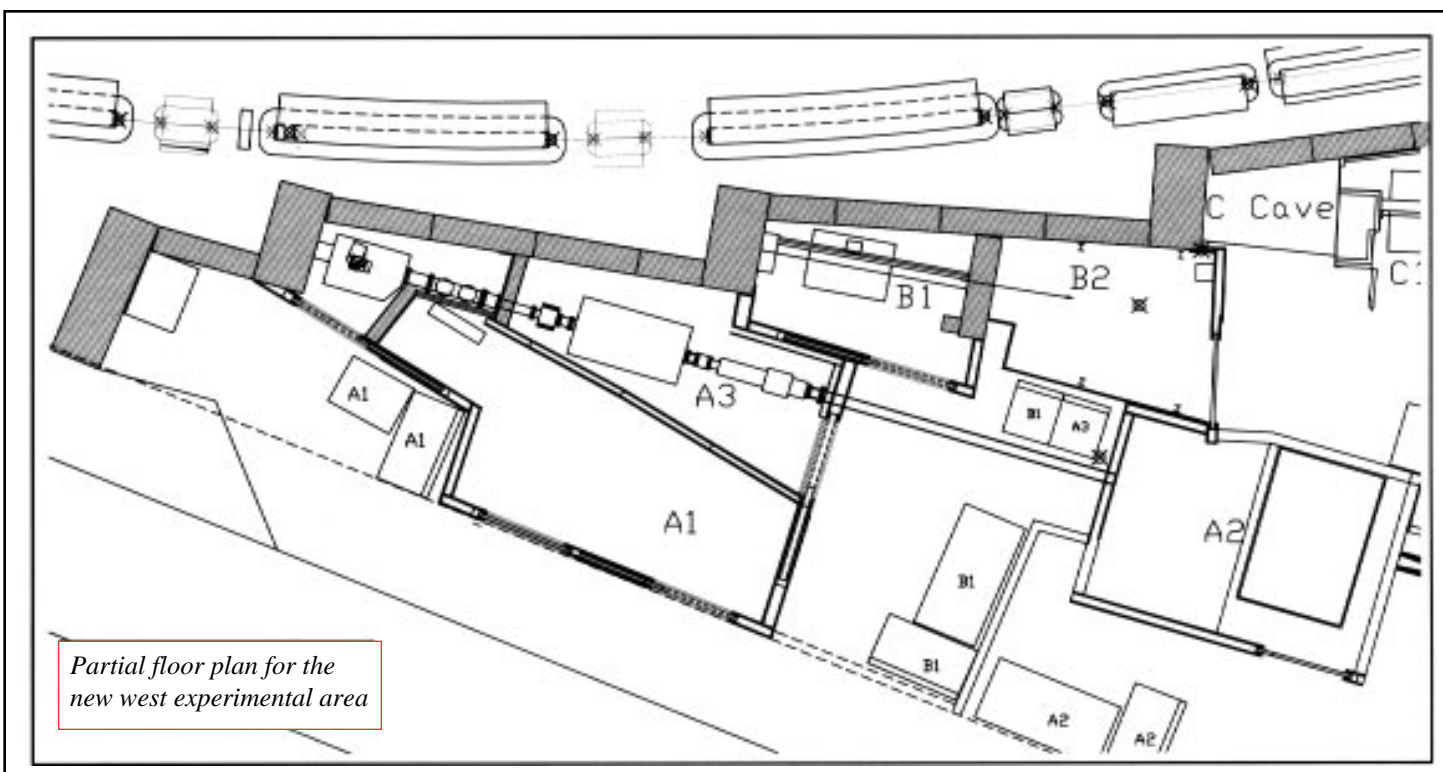
permit fast opening of the pole gap to accommodate storage ring injection. The mechanics were designed and built by Bob Batterman and Stewart Brazil (a Cornell Co-op student), while the magnetic structure is due to Ken Finkelstein. This beam will be shared between the A1 and A2 end stations.

UHV Beamline Upgrade. The present A-line components (beamstops, beryllium windows, etc.) were never designed to handle the power produced by the new wiggler. The new beamline is being engineered to handle the new wiggler to a current of at least 200 mA and, where easily possible, to a 500 mA value, in anticipation of the higher currents planned for in the CESR B-factory upgrade. Consequently, new copper wedge-shaped beamstops are being fabricated with 500 mA capability (see the following article). A new tapered mask (see cover photo) will be installed ahead of the beamstop, and contains a beam position monitor that will always be active. We are considering the interlocking of this position signal to a machine safety control loop. One possibility is that above some power level, detection of syn-

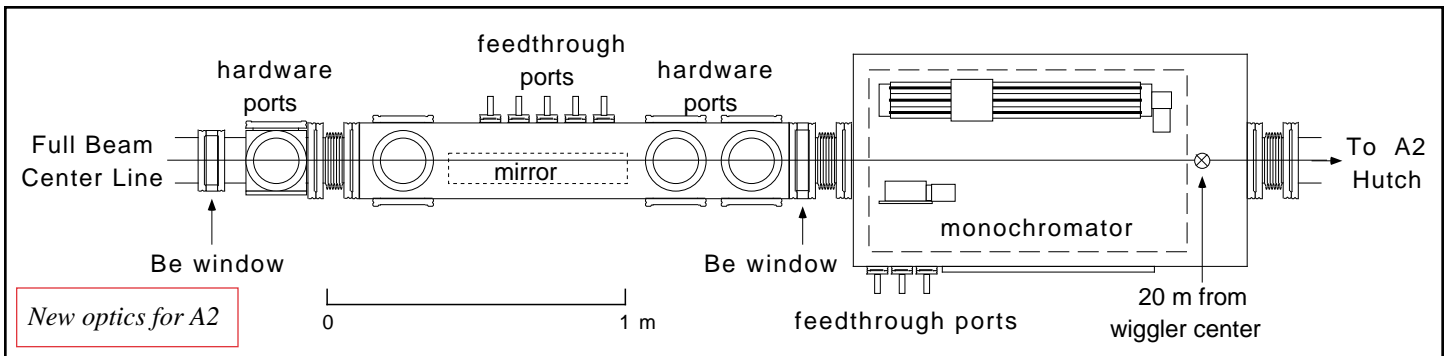
chrotron radiation outside a specified position would dump beam before beamline components could be damaged.

There will also be monitoring of infrared radiation emitted from the downstream side of the first or second beryllium x-ray windows (see separate article). We will be able to monitor the window temperature *in-situ* and sense problems before they cause damage. The new beryllium windows will be patterned after the F-line windows which presently work satisfactorily to 100 mA.

A1 Station Upgrade. The old A1 station, about the size of a telephone booth, is being replaced by a much more spacious room. In addition, the station will now be *tunable* over a range of from 7.0 keV to 14.5 keV. The optics will continue to be a horizontally focussing triangular silicon crystal followed by a vertically focussing mirror. A similar optical arrangement is presently in use on station F1, as discussed in the article on page 8. This doubly focussed line will continue to appeal to the protein crystallography and small angle scattering communities, in general. Our MacCHESS group is heavily involved



Partial floor plan for the new west experimental area



in making this line as good as F1 from a diffraction point of view.

B1 Station Upgrade. See the accompanying article by Keith Brister about the changes to this station (page 17).

A2 Station Upgrade. A complete redesign will let A2 users take full advantage of the new 25 pole wiggler source or dedicated undulator operation, by offering them a tremendous number of beamline configurations ranging from unfocussed white beam to focussed white beam to highly monochromatic, doubly focussed radiation. Joel Brock's group (Applied Physics), Hector Abruna's group (Chemistry) and the CHESS staff are developing a high-resolution x-ray scattering line with funding support from both CHESS and the Materials Science Center at Cornell University.

As shown on the previous page, the old A3 station floor space will become a flexible optics room for the A2 station. The entire A2 beamline will be under vacuum. The various optics areas are separated both from the A1 optics and from each other by beryllium windows. This design will permit A1 operation to continue while A2 optical components are in access. In addition, either the monochromator box or the mirror box may be vented without venting the other, and the upstream/downstream order of the boxes is interchangeable.

A beam position monitor will be permanently mounted upstream of the first x-ray optical element. Another is permanently installed in the front end of A-line. By combining these two position monitors we will determine both the position and

angle of the white wiggler beam. Eventually, we hope to provide these signals to CESR to aid in steering the beam.

The A2 mirror will take advantage of new cooling and bending technology being developed by the APS which is capable of surviving in the intense white wiggler beam. The line will have toroidal and flat mirror options: the toroidal mirror focusses the white beam in both horizontal and vertical planes, and the flat mirror focusses in only the vertical direction. The beamline can also run without a mirror, and the A2 hutch will be white-beam capable. The mirror project is being jointly accomplished with our colleagues from APS (Dennis Mills and George Srajer). The mirror is designed to focus approximately 1/4 of the incident power to an area of a few mm². This will be one of the hottest beams available for hard x-ray heat-load testing and scientific purposes.

The monochromator will be a standard two-axis device. The long travel and adjustable crystal offset permit the use of synthetic multilayer crystals with small Bragg angles. The

second crystal holder will contain the bending hardware currently used by the sagittal focussing crystals at CHESS. Thus, doubly focussed, monochromatic x-ray beams can be produced by combining either the toroidal mirror and flat crystals or a flat mirror and a sagittal focussing monochromator. The variable crystal offset provides the additional flexibility required to use either a back reflection geometry or, using channel cut crystals, a Dumond configuration in the monochromator.

The entire construction project will take place between January and June, with beamline commissioning in early Summer of 1993.



Don Bilderback



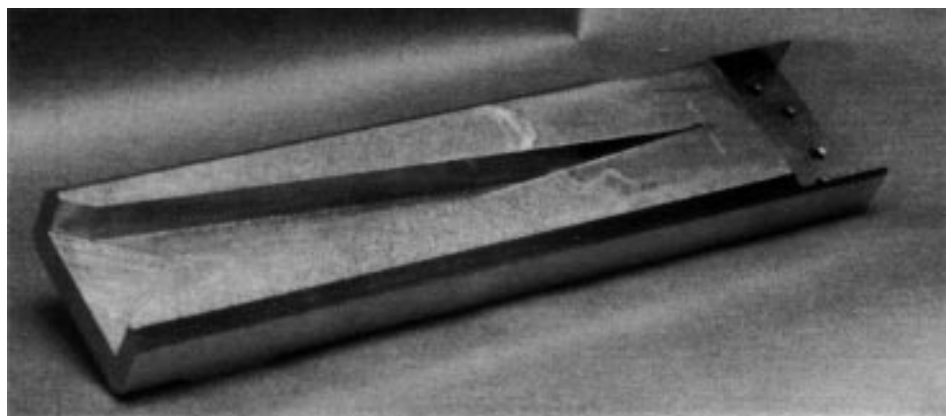
Joel Brock



Bill Podulka

Beamstop for ultra-high heat loads

Qun Shen
 Chuck Henderson
 Matt Marston
 Bob Batterman



The copper portion of the primary A-line beamstop (see also cover photo and page 3).

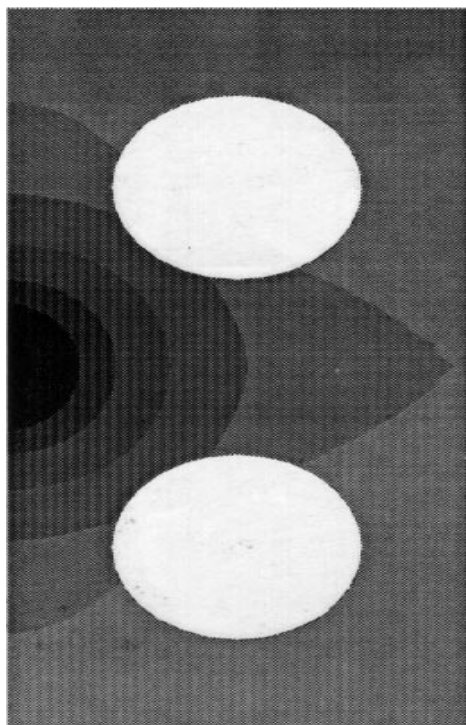
The new A-line 25-pole permanent magnet wiggler can output 32000 Watts of power, with a critical energy of 23 keV, when CESR is running at 5.3 GeV and 500 mA. The beam size at the 10 meter point, where the first beam stop is located, is about 40 mm x 2 mm. This yields a linear power density of 800 W/mm and a surface density of 400 W/mm².

To design a safe beam stop for this hot beam, we have adopted the idea of water-cooled copper absorbers as proposed for the CESR B-factory [K. Smolenski, *et al.*, SPIE Proceedings, (1992)]. Extensive heat load simulation and thermal cycling tests have been performed on a prototype

water-cooled copper piece. The results show that at normal incidence power levels of 40 W/mm and 80 W/mm no damage was observed on the copper surface after several thousand thermal cycles, whereas at 160 W/mm some surface microcracks occurred.

Based on the test results and finite elements analysis, we will use a 5° horizontally-tilted copper surface with ample water cooling (see photo above). The 5° slope decreases the linear power density to 70 W/mm, which is a safe level according to the thermal cycling test. The surface power density is reduced to 35 W/mm², which is about the same as for the present A-line beamstop for the electro-magnet wiggler at 100 mA, and which has survived many years of operation. A finite-element ANSYS calculation on the 5° sloped beamstop shows (at left) a maximum surface temperature of 320°C at 500 mA with a film coefficient of 2 W/cm²-°C. To reduce the overall length, we use a V-shaped design.

Some interesting conclusions can be drawn from our design process. First, the water channel placement is not a crucial factor as long as there are sufficient water channel surface area and copper volume around the channels. This is due to the fact that copper is such a good thermal conductor that any reasonable beam displacement can be compensated for by the conduction. Second, for a typical wiggler beam, tilting of the water cooled surface vertically or horizontally are not equivalent. For example, for a 5° slope, a vertical tilt results in a surface temperature of 900°C instead of 270°C as for the horizontal design shown above. The reason for the very effective side cooling is the matching of a wide horizontal beam with a long horizontal slope, thereby decreasing both the linear and the surface power densities, whereas a vertical slope only reduces the surface power density. Therefore, for a wiggler beamstop, the linear power density should be the dominating design consideration.



Left: Thermal profile of the new A-line beamstop produced by ANSYS calculation. At normal incidence (from left, centered) an x-ray beam produced by the 25-pole CHESS wiggler operating at 100 ma CESR current. Cooling water flows through the two channels. The shading changes density at temperatures of 300, 270, 240, 210, 180, 150, 120, and 90 °C. The cooling water temperature is 85 °F.



Qun Shen

Beryllium windows for wiggler light

Ernie Fontes
Qun Shen

CHASS has an on-going effort to design new and better vacuum chamber x-ray-transmitting windows, an absolute necessity in light of the aggressive plans to increase the CESR current fivefold. At the present 100mA operating conditions, upwards of 7000 Watts of power are produced by the F-line (and new A-line) wiggler, and ~500 Watts are absorbed by the first of three Beryllium windows (typically 0.015 inch thick). Three vacuum-tight Be windows are mounted on water-cooled copper "gaskets" (see photographs) that separate two tee-shaped UHV chambers (see drawing). At only 100mA of current, past windows have shown discoloration, distortions, and in one case, considerable disintegration.

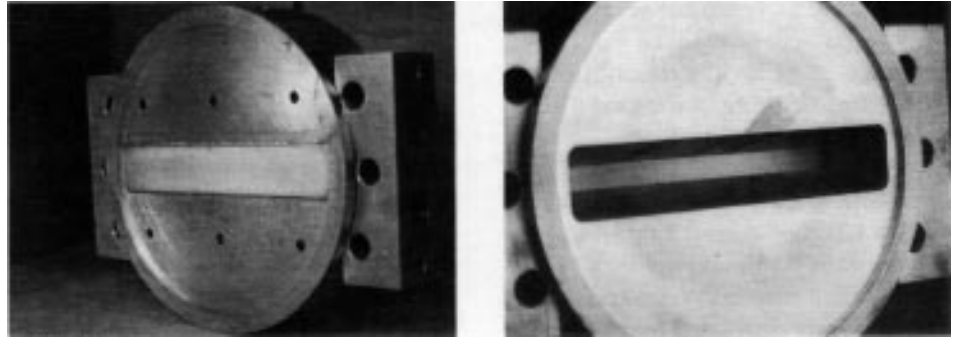
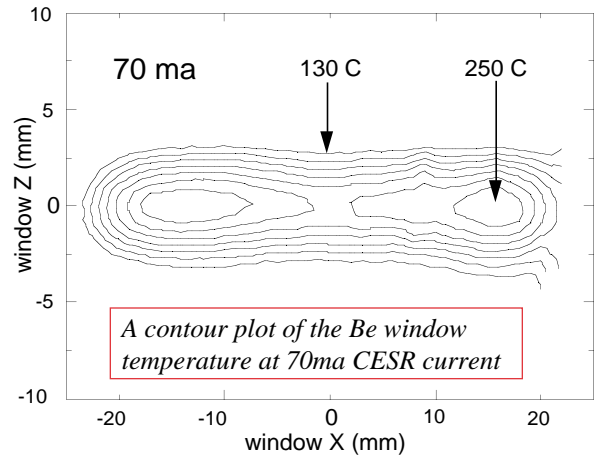
As part of this development project, a 2D scanning Infra-Red camera is being used to monitor the temperature of the Be windows *in-situ*. The IR detector is a thermoelectrically cooled PbSe photodiode, using a CaF₂ optical arrangement to realize a 1mm.² active area. Two computer controlled mirrors are used to scan the entire illuminated area of the downstream side of the first (or second) Be window. An equal-temperature contour plot (above) shows the thermal footprint during stable HEP running. The non-uniformity of the peak temperature is due to optical edge distortions caused by the vacuum chamber.

One and two-dimensional temperature profiles agree very well with ANSYS calculations. Surprisingly, the peak temperatures do not occur during normal beam conditions, but rather, the temperature increases by

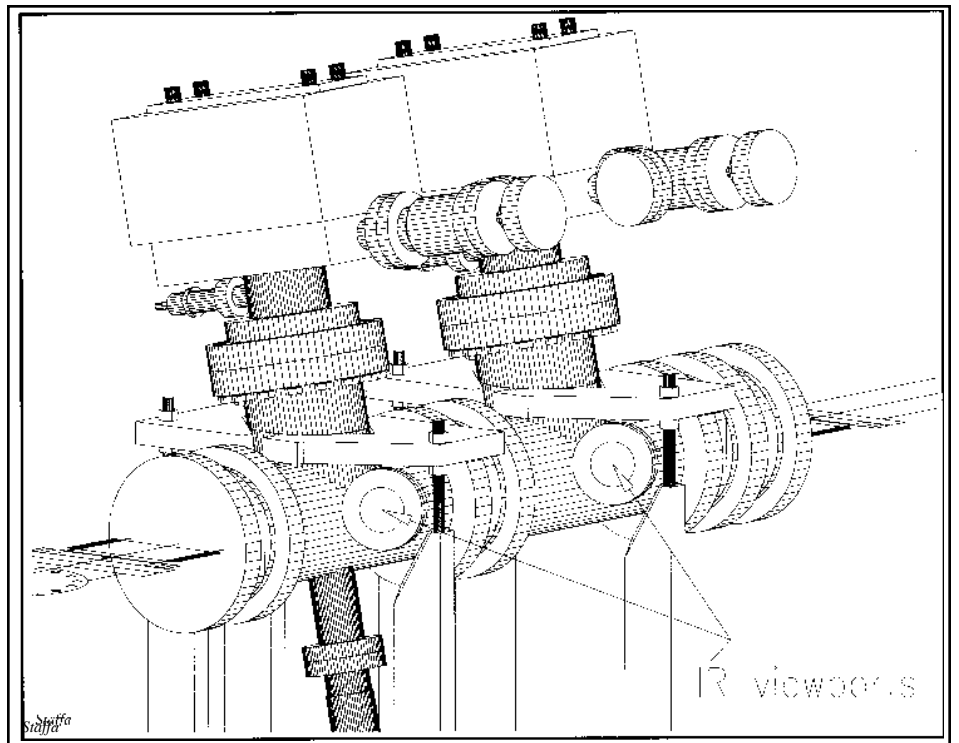
~100 °C during CESR injection and tuning. At those times, the incident x-ray beam comes into contact with the edge of the Be-Cu junction at the window edge. The absorption of large amounts of heat in the Cu and the good thermal contact to Be results in rapid overheating of the window.

In order to increase window longevity, current designs include a modification to the Cu mask so that the Be-Cu junction cannot be directly illuminated with x-rays, and a thin Carbon

filter to reduce the thermal load on the first Be window from 500 to 75 Watts.



(Above) Upstream (left) and downstream (right) views of the Beryllium window gasket assembly, as discussed in text. (Below) An upstream view of the mechanical design of the IR viewports on the two UHV chambers between the three Be window assemblies.

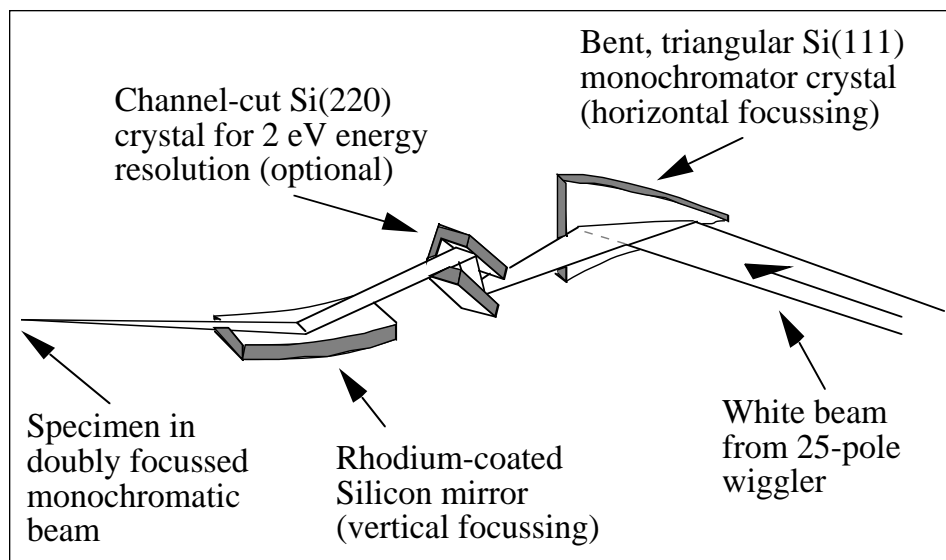


F1 optimized for x-ray crystallography

Bob Batterman
Ken Finkelstein
Sarojit Shastri

This article describes three recent improvements to the CHESS F1 station that make it one of the premier x-ray crystallography facilities in the world. Only a few key elements of the F1 beamline are presently discussed: note that a fully documented F1 facility description will be forthcoming in the next CHESS *Activity Report*.

The source of radiation for F1 is a 25-pole permanent magnet wiggler built at CHESS. About 2 milliradians of this beam is intercepted by a cylindrically bent silicon crystal and simultaneously diffracted and focussed in the horizontal plane (see figure above). An optional channel-cut crystal can be used to increase the monochromaticity to 2 eV energy resolution. This beam is then focussed vertically by a cylindrically



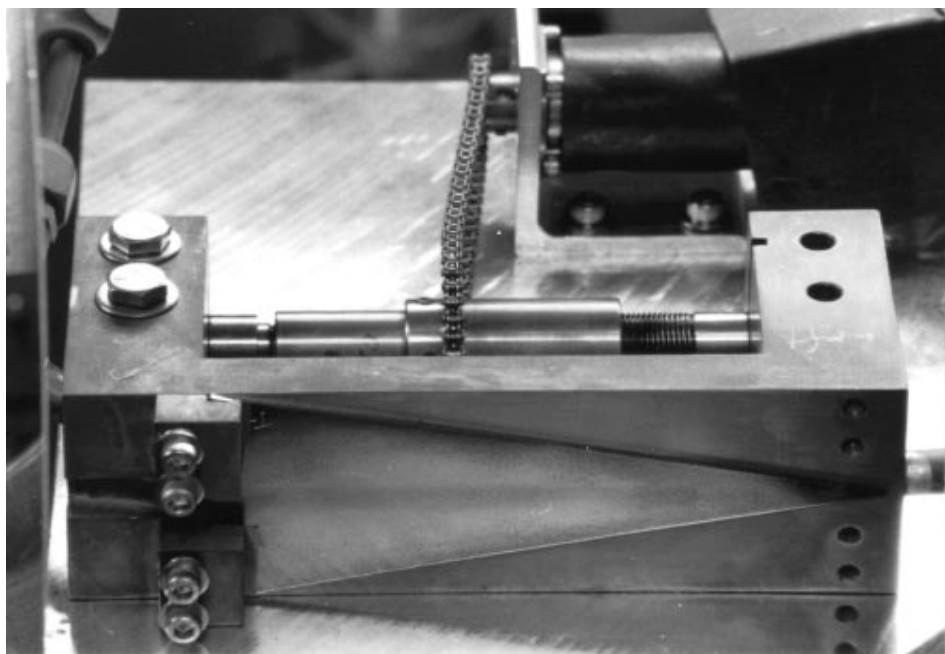
bent rhodium-coated silicon mirror. The typical focal spot size at the specimen for 13keV photons is approximately 2mm horizontal by 0.4mm vertical.

The most recent upgrades to the beamline include: 1) modification to the bending mechanism for the first triangular silicon crystal to withstand the high heat loads produced by the wiggler and yet maintain tunability over a wide range of energy, 2) the addition of a channel-cut monochromator to provide high energy resolution simultaneously with double focussing, and 3) the modification of the bent silicon crystal to optimize its

acceptance properties to match that of the synchrotron beam and thereby improve the delivered intensity by, in some cases, up to a factor of 4.

Because of the enormous heat flux produced by the wiggler, the cooling of a first optical element is of primary importance to delivering a high flux beam to the experiment. In our fixed wavelength A1 station, the bent silicon crystal has a fixed radius. We accomplish cooling by matching the bent crystal surface to a water-cooled copper block, machined with a matching fixed curvature to maintain intimate thermal contact with the crystal. However, if the primary monochromating crystal is to be tunable, the radius of curvature must be variable. The challenge, then, is to design a cooling device that can accommodate a changing crystal bend radius.

The photograph at left shows how this was simply accomplished. A copper block in the shape of an elongated "C" is machined flat. The triangular crystal is attached only at its base. As a differential screw spreads the two ears of the C-shaped copper block, the copper surface is curved, thereby bending the crystal. What is not obvious, but essential, is that the figure of the crystal is determined by the elastic properties of the triangular crystal and the copper block merely acts as a bending device. Since the copper block only



holds the base of the crystal and pushes at its tip, the triangular shape of the crystal guarantees a nearly perfect cylindrical figure. This shape provides the best focussing of x-rays. The added benefit of the current design is that as the copper block bends into an approximate cylinder, its close proximity to the crystal facilitates cooling for any radius of bend.

The second recent innovation utilizes a channel-cut crystal inserted between the triangular crystal and the rhodium-coated mirror. This crystal selects a very narrow band of the incident photons and yet maintains, simultaneously, both horizontal and vertical focussing. In this way the F1 station can be used for MAD crystallography (multiwavelength anomalous diffraction) with an energy resolution of a few eV yet tunable over hundreds of eV. Having both the high intensity in a focussed beam, and tunable energy, the anomalous scattering properties of certain elements can be utilized even with small specimens. In certain cases, MAD crystallography circumvents the need for isomorphous substitutions and can directly get phase

information from the parent crystal itself.

Finally, to maximize the x-ray flux into the specimen, we take full advantage of the diffracting properties of the monochromator crystal and its dependence on crystal perfection. It is well known that an imperfect crystal diffracts more integrated intensity than a perfect one. In general, most of our x-ray optics have been done with perfect silicon crystals. It is possible, however, to create a slight amount of imperfection in a crystal such that the energy spread increases modestly and at the same time gain a corresponding improvement in photon throughput.

By carefully grinding the surface of the bent silicon crystal with fine emery paper, it was possible to increase the throughput of the system by almost a factor of 4, with less than a factor of 2 increase in the energy width passed. To calibrate the flux gain, we use a 0.3mm collimator at the sample position. After grinding the crystal surface, the flux at the sample increased by a factor of 3, while simultaneously increasing the energy bandpass from 13 to 17eV.

The success of the roughening is due to the fact that the F1 beamline has optical aberrations (i.e. source size, imperfect focussing) much larger than the intrinsic diffraction width of the perfect unground monochromator crystal. Increasing the energy bandpass of the crystal is analogous to the removal of a flow constricting aperture. The diffracted intensity onto the specimen is maximized by better matching the selectivity of the monochromator crystal to the overall beamline optics.

The figure on page 11 summarizes the successful performance of the F1 station for macromolecular crystallography: a previous x-ray diffraction pattern, requiring 10 minutes on the original A1 station, can now be collected in 10 seconds at the F1 station.

Storage phosphors (image plates) have replaced film as the detection medium of choice for accurate collection of diffraction data on high flux beam lines such as A1 and F1. Storage phosphors have good spatial

resolution, digitizing an 8" x 10" area into approximately 2000 x 2500 100x100 micron² pixels. They also have a very high dynamic range of 10⁴, and do not suffer from count rate limitations as do multiwire proportional counters. Also, the 10 megabyte image is immediately available for evaluation on the computer screen.

For some years we have been operating a prototype image plate scanner built by Kodak. It is capable of accurate data collection but is relatively slow (~5 minutes per image) compared to more recent devices. This spring we took delivery of a Fuji scan-



New Fuji BAS 2000 scanner

Bill Miller

ner that is presently installed in the F1 station. This is a much more compact unit and has faster throughput than the Kodak scanner (less than 2 minutes per image). This minimizes the turn-around time per image making better use of the high flux available at F1.

For more information on the scanner system, contact Bill Miller (607-255-7163).

This story was written by William Holder of the Cornell News Service for publication in the Cornell Chronicle on December 3, 1992.

Fighting AIDS: Scientists seek new drug design route

The Cornell High Energy Synchrotron Source (CHESS) is the centerpiece of a new effort by Cornell University scientists to find a new way to design drugs for AIDS and other diseases.

Armed with a \$3.31 million grant from the National Institutes of General Medical Sciences, a branch of the National Institutes of Health, four faculty members whose laboratories have a high speed electronic link for sharing graphic images of complex molecules and other data will work on an approach to structure based drug design.

Structure based drug design is an ongoing effort in universities and pharmaceutical companies to design drugs on computers, based on a detailed knowledge of the geometry of a target molecule that is crucial in a disease. So far, however, the technique has failed to deliver on its promise. At fault, at least in part, are the complexities of accurately determining the geometry of drugs as they bind with their targets and limitations on computer algorithms, according to Steven Ealick, a Cornell professor of biochemistry who is leading the new effort.

The Cornell team is using a multidisciplinary approach in its search for a new route to drugs for treating AIDS. They will use the Cornell synchrotron, one of the world's most intense sources of X-rays, in an effort to speed up by orders of magnitude the process of acquiring geometric information through X-ray crystallography. CHESS has the only biological isolation facility in the world where crystals of hazardous viruses and proteins can be routinely subjected to intense X-ray beams to determine their three-dimensional structure.

The National Supercomputing Facility at the Cornell Theory Center



Steven Ealick, professor of biochemistry, molecular and cell biology, mounts a film plate in the macromolecular crystallography facility at CHESS, the centerpiece of a \$3.3 million effort by four faculty to develop new drugs for AIDS and other diseases. (Photo: Chris Hildreth/University Photography)

is furnishing the resources for a related effort to more accurately compute how strongly candidate drugs bind to their targets.

Bruce Ganem, a Cornell professor of chemistry, will head the experimental thrust of the program, consisting of chemical synthesis of potential drugs. His work, he said, will provide a "reality check on structure-based design by showing how well candidate drugs actually bind to target molecules.

Other members of the team are David Shalloway and Andrew Karplus, faculty members in the Section of Biochemistry, Molecular and Cell Biology.

"Our approach to AIDS," said Ealick, "is to look at proteins that are related to disease but not produced by the virus." Specifically, they have selected PNP (purine nucleoside phosphorylase), an enzyme present in red blood cells and other tissues that recycles substances required for normal cell growth. Unfortunately

for AIDS therapy, the enzyme also destroys DDI, a newly approved AIDS drug.

"We have good unpublished observations that suggest DDI may work much better in the presence of a supplemental drug that inhibits PNP. Developing such an inhibitor is one of our goals," he said.

Ealick and Ganem hope to take advantage of a new understanding that the old "lock and key" model of interactions between drugs and receptors is inadequate.

Scientists now believe that they need to know what the lock and key look like when the key is inserted and turned, according to Ganem. More technically, they seek a molecule that will mimic the geometry of the interaction between DDI and PNP at a more advanced stage (the transition state) in the reaction that destroys DDI. Their hope is to find a substance that provides a better fit with PNP and therefore leaves DDI to go about its business.

Practitioners of structure based drug design have found the task more complicated because the geometries of both the target molecule and the drug often undergo substantial changes when the two interact. A detailed knowledge of what each looks like by itself may say little about the more important drug-receptor complex.

The team expects its techniques to be applicable to many medical problems. Karplus, for instance, is seeking an inhibitor of an enzyme produced by the parasite that causes sleeping sickness and other widespread and serious disease in tropical countries. A crucial component of this project, one that also has bearing on AIDS, is determining how the presence of water molecules alters the geometry of the drug-target interaction. Karplus expects the synchro-

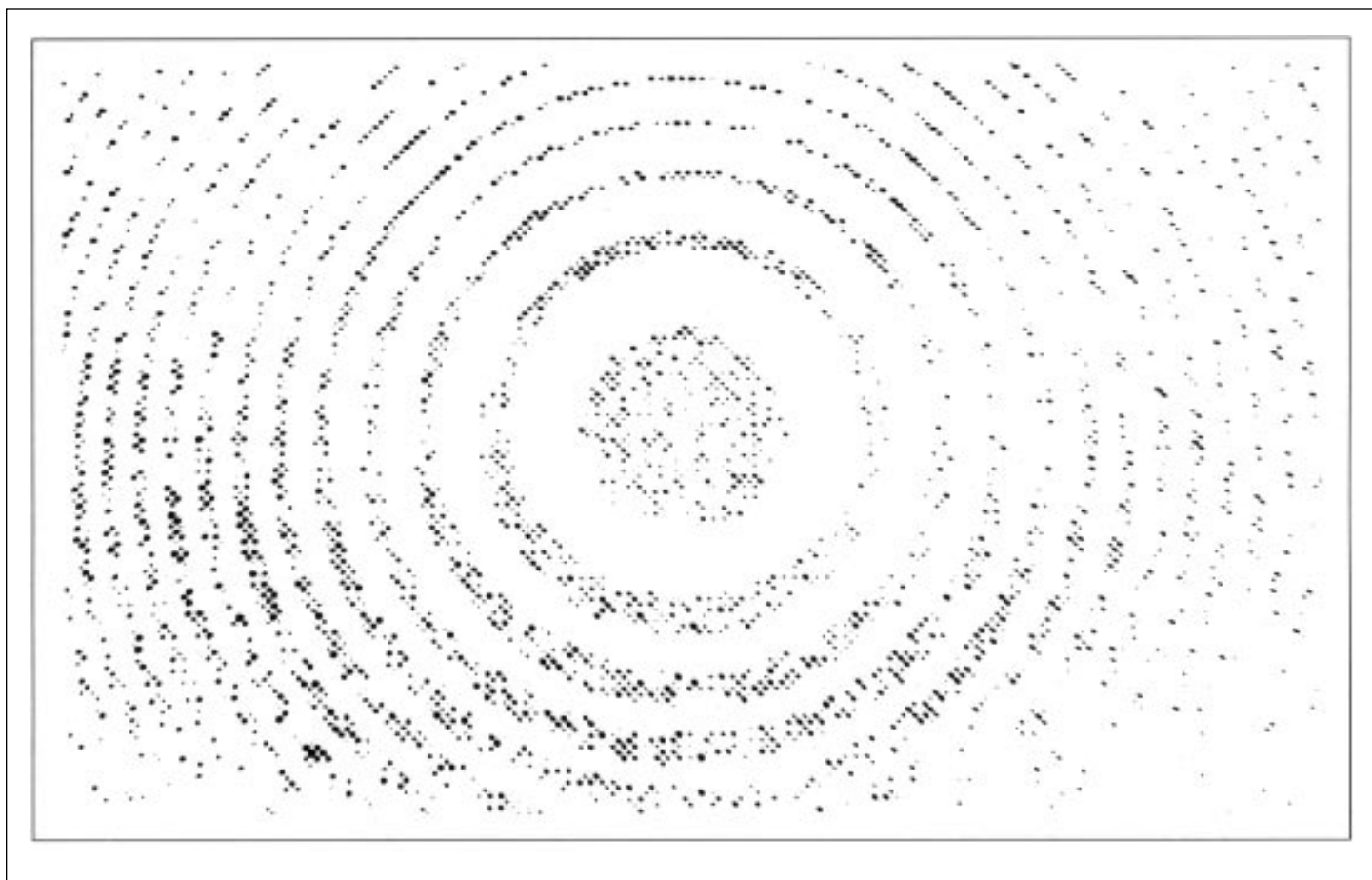
tron to help provide him with the amount of X-ray data he needs to understand that interaction.

One of the most ambitious aspects of the project is an effort, headed by Shalloway, to improve the ability of scientists to compute the strengths of the binding interaction between drugs and targets. More than anything else, it is deficiencies in computational algorithms that are holding up progress in structure based drug design, he said.

Part of the problem is that computations must take into account the mountains and the molehills of molecular motions. These motions vary from the large and slow vibrations of the whole protein, which take place on the microsecond scale (a millionth of a second or longer), to the ultrafast vibrations of chemical bonds, which occur in femtoseconds (a millionth of

a billionth of a second). Accounting for such disparate scales in the same computer model is a formidable challenge. Shalloway is approaching the problem using techniques he acquired in earlier training as a theoretical physicist.

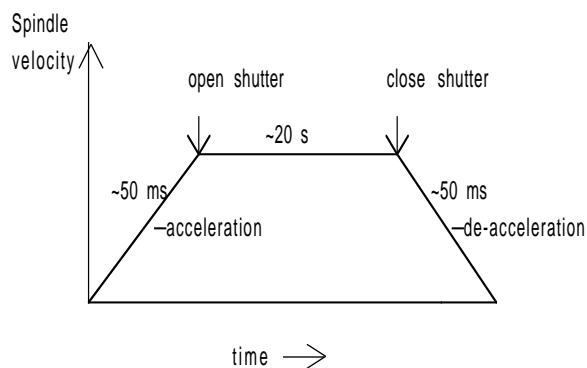
CHESS is uniquely suited for the experimental component of this work. The X-rays used in this facility are produced by the Cornell Electron Positron Storage Ring, a one-half-mile ring in which counter-rotating beams of electrons and positrons are collided at energies of about 5 billion volts for the study of subatomic particles. The rotating beams produce short bursts of X-rays lasting about one ten-billionth of a second and having an intensity about 1 million times that of a chest X-ray.



Central portion of an HRV16 x-ray diffraction pattern taken on the A1 station ($\lambda=1.56\text{\AA}$) at CHESS (Rossmann, Purdue University). The crystal-to-film distance was 100mm, and the exposure time was 10 minutes. The oscillation angle was 0.3° . A similar pattern taken today on the F1 (or new A1) beamline using a Fuji imaging plate and a crystal of about 0.3mm diameter would require about a 10 second exposure time.

Oscillation camera control for crystallography

Jim Laluppa
Tom Irving



The mechanical requirements of oscillation photography are quite simple: the crystal must move over a well defined angular range at a constant speed. The practical problem is that a stepping motor needs a significant amount of time to speed up and to slow down, and one must compensate for the distance traveled during this time in setting up the oscillation range and in controlling the opening and closing of the shutter.

In a recent F1 upgrade, the program *immed* has been replaced by *oscam*, a macro package that performs oscillation scans within the lab-standard diffractometer program *spec*. *Oscam*, and camera control program *osc*, mimic the internal functions of *immed* while providing a more user-friendly interface. *osc* is menu-driven, and editing oscillation parameters has been simplified. Rather than specify motor speed, acceleration time, and shutter opening point, then creating loops to do multiple passes, the user now simply provides the range, total exposure time, and sample advance. *osc* calculates the optimal motor speed and number of passes.

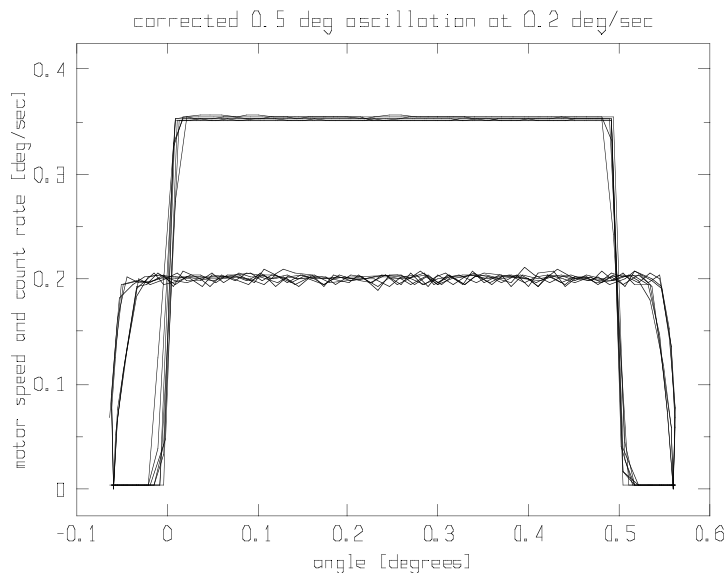
The most important improvements are internal, however. *oscam* allows us to quantify the performance of the entire system, which could not be done under *immed*. We discovered flaws in the basic camera behavior, among them that the E500 motor controller did not guarantee the same rate for accelerating and decelerating motors. Since the shutter had been controlled by motor speed, the nonuniform rate resulted in angu-

lar passes which did not perfectly overlap.

We found, however, that software corrections, such as delay and offset loops, could not guarantee precision in all experimental situations. Hardware modifications were needed. The shutter is now controlled by an accurate optical angle encoder mounted on the spindle motor and an electronic timing generator, ensuring pinpoint accuracy of the oscillation range. The **figure below** shows both the accuracy and precision achieved. At the start of an oscillation, the spindle is backed off a distance and the motor starts moving. When the spindle passes the starting angle, at full speed, the shutter is opened. After the specimen has com-

pleted the full oscillation range, the shutter is closed, and the motor begins to decelerate. At all times the exact position of the spindle encoder is known, and the spindle is always travelling at a constant speed while the shutter is opened.

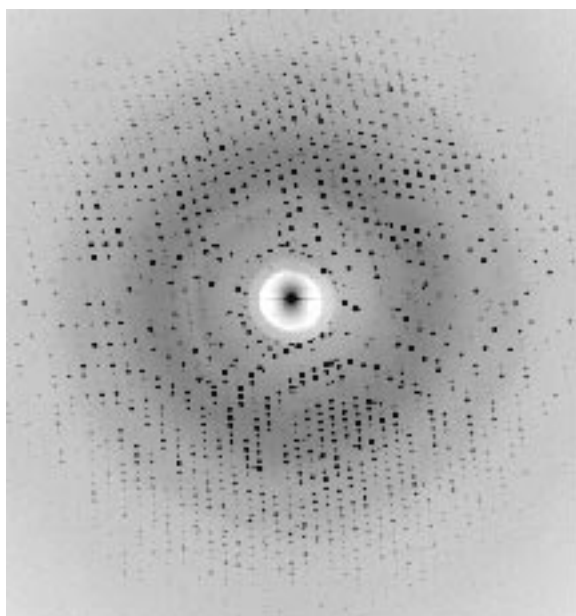
Other features of *osc* include precise reporting of the measured range and time of the exposure, graphical depictions of speed and count rate, and automated record keeping to file and printer. The current oscillation camera control system is intended to evolve along with the needs of the crystallographic community, so users should feel free to make suggestions for improvements. Direct your comments to Jim Laluppa or Tom Irving (607-255-7163).



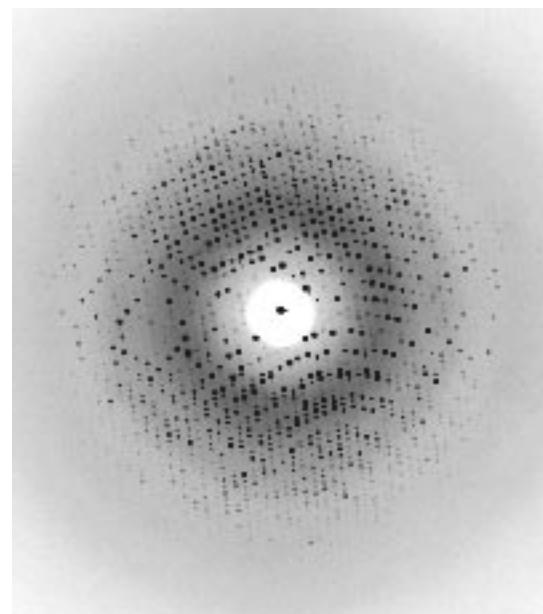
The bottom curves show the instantaneous velocity of the camera spindle for 3 complete oscillation cycles (six passes). The velocity fluctuations are due to timing errors in reading the E500 motor controller. The top curves show the opened/closed state of the shutter: the specimen is only exposed to x-rays when the spindle velocity is constant.

Protein crystallography at high energies at F2

Steve Ealick
Tom Irving
Qun Shen



Lysozyme oscillation frames measured at F2 with wavelengths of 0.5 Å (left) and 0.34 Å (right). Both patterns were recorded using unfocussed radiation at station F2, yielding good statistics to 1.5 Å resolution.



For many years, most synchrotron stations for crystallography, such as CHESS A1, used X-ray sensitive film as the primary detector and x-ray radiation at about 8 keV (1.5 Å). Two years ago CHESS constructed station F1 on a wiggler line that is tuneable from about 7-14 keV. In practice, this station operates with a standard energy of 0.91 Å. The benefits of using higher energies soon became apparent in terms of improved X-ray diffraction data that resulted from decreased absorption, decreased sample decay and lower background scatter.

The crystallographic results from this station have been spectacular, with experimenters reporting much better data using fewer crystals. Because of the high energy operation of CESR, the critical energy of the F line wiggler is about 24 keV or 0.5 Å, and a useful flux to the sample can be obtained at 0.3 Å or less. Therefore, it is currently possible only at CHESS to determine whether or not the benefits derived from lowering the X-ray wavelength from 1.5 Å to 0.9 Å can be extended to the high energy range (0.3-0.5 Å).

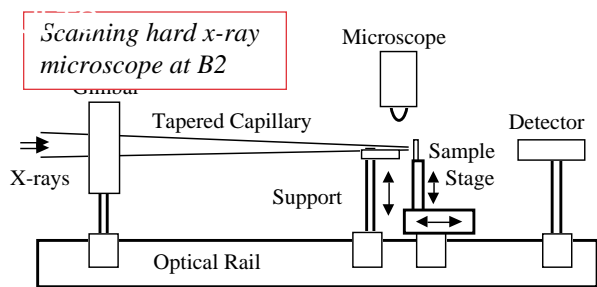
In order to test these ideas we have performed preliminary experiments on station F2 using lysozyme and nucleoside deoxyribosyl trans-

ferase, an enzyme with high sensitivity to radiation damage at room temperature. Test lysozyme data was measured using the oscillation method and image plate detectors at the critical wavelength of 0.5 Å and just above and below the Ba absorption edge at 0.34 Å. The experiment near the Ba absorption edge was to determine whether or not the sensitivity of the image plate, which contains Ba, could be enhanced by measuring data above the absorption edge.

The figures shows a typical lysozyme oscillation frame measured at wavelengths of 0.5 Å (top) and 0.34 Å (bottom). Both patterns were recorded using unfocussed radiation at F2, over an oscillation angle of 1.0 degree. The data is somewhat noisy because of insufficient shielding and/or collimation, but the data were easily processed at all three energies and gave good statistics to 1.5 Å resolution.

These experiments showed that data collection from macro-molecular specimens at these energies is perfectly feasible. The Ba edge experiment was inconclusive although the higher energy data showed significantly more background and therefore poorer overall signal to noise.

The data from transferase showed some but as yet inconclusive evidence for enhanced crystal lifetime at 0.35 Å. These preliminary experiments are encouraging and MacCHESS personal will continue to systematically explore the use of high energies with a wider range of examples and conditions.



malized transmission change as the gold stripe is scanned. The absolute intensity change was 2%, taken at 12.3 keV, using a multilayer monochromator, where the incident flux exiting the pipette was 10^6 photons/sec.

A two-dimensional image can be formed by monitoring absorption as the 2-dimensional pattern of the gold lines described above is scanned across the tip. Since this type of 2D raster scan requires long periods of data collection, these experiments have proven to be very stringent tests of the stability of the x-ray beam position and angle. We are excited about the possibility of doing microprobe imaging experiments on the submicron scale.

The application of these condensing and confining devices to Laue diffraction from proteins was tested in a collaboration with Steve Ealick. Using a 100 μm thick lysozyme crystal and a 5 μm diameter x-ray beam, very nice Laue photos were obtained in 10 second exposures. With the attribute of enhanced intensities, microbeams from single tapered glass capillaries may greatly boost our ability to determine three-dimensional protein crystal structures that are too small for the present technology.

A new addition to our group, Dan Thiel who has just finished up his Ph. D. work at Cornell, has been instrumental in the making and profiling of pipettes, while Steve

Hoffman has been heavily involved with the Laue diffraction and imaging experiments. Aaron Lewis (Hebrew U.) and Ed Stern (U. of Washington) also continue to contribute to the collaboration.

Submicron-sized x-ray beams

Don Bilderback
Steve Hoffman
Dan Thiel

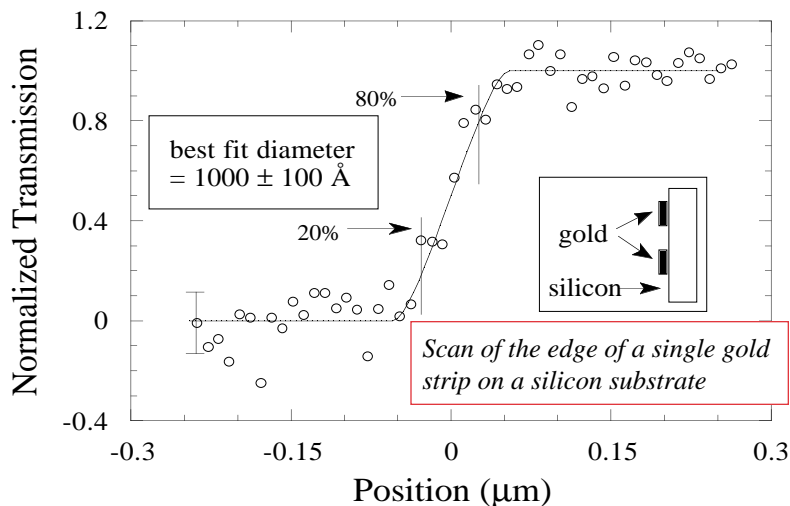
Submicron diameter x-ray beams have been made using single hollow tapered capillaries. X-rays are concentrated into a smaller cross-section (becoming more divergent) by totally reflecting from the inner walls of tapered glass pipettes. The smallest beam to date had a 0.1 μm diameter and a gain (intensity/area) of 960 at 6 keV.

Beam sizes are confirmed by scanning the edge of a 1000 \AA thick gold stripe, lithographically deposited onto a silicon wafer, across the tip of the capillary. The figure above illustrates the experimental set-up, while the figure below shows the nor-

Call for MicroScience X-ray Station at CHESS

The microscience group at Cornell is interested in new applications of microbeams to science. For instance, we plan to look at fibers of a few microns in diameter in the near future with Material Scientists (David Grubb, Chris Ober, Wade Adams, etc.) using wide angle x-ray scattering to determine the local structure differences from the skin and core, for instance, of strong man-made composite fibers. We also plan to map the strain, microstructure, and composition of tiny polycrystalline crystals (Slade Carghill, Jeff Noyan) using high spatial resolution with microdiffraction and microfluorescence measurements. Many biological experiments are also in the conceptual stage.

We are also in the process of raising funding for specific equipment to outfit a CHESS beamline for these kinds of investigations. The biological work has support from MacCHESS, but we hope to generate additional support in the Material Science community. One possibility is to complete a first round of preliminary experiments of the type described above and then to approach the Division of Materials Research at the NSF to fund the creation of a specialized beamline over a 2 yr. period of time, including a person to help construct and oversee collaborative experiments (miniature monochromators, translation stages, fluorescence detectors, tiny 2-d CCD detectors, etc.). We need to raise about \$200,000 to adequately fund these efforts. Additionally, we would like to attract visiting scientists to come and join us in these efforts for some period of time. If you are interested in contributing in time, effort, or even funding for these activities, please contact Don Bilderback immediately at Ph: 607-255-0916 or Bitnet: bilderback@crlnches.



Fall 1993 dedicated undulator run

Don Bilderback

CHES is now accepting proposals for its third hard x-ray undulator run of one month duration, due to take place somewhere between September and November, 1993. During this run, CESR is reconfigured to optimize the performance of the undulator. Since the CHES undulator is the sole user of CESR during this time, the NSF provides additional funding (\$0.5M per month) to cover the operating expenses.

Below is a list of publications that resulted from the successful running of the 3.3 cm period, 123 pole undulator¹⁻³ in 1988 and 1991 by a joint team of scientists from Cornell

University and Argonne National Laboratory. This insertion device produces x-rays with energy tunable from 4.7 to 8 keV in first order and 14.1 to 24 keV in third order. We anticipate operation at 5.43 GeV, 80 to 100 milliamper currents with 6 bunches of electrons or 35 milliamperes in a single bunch timing mode.

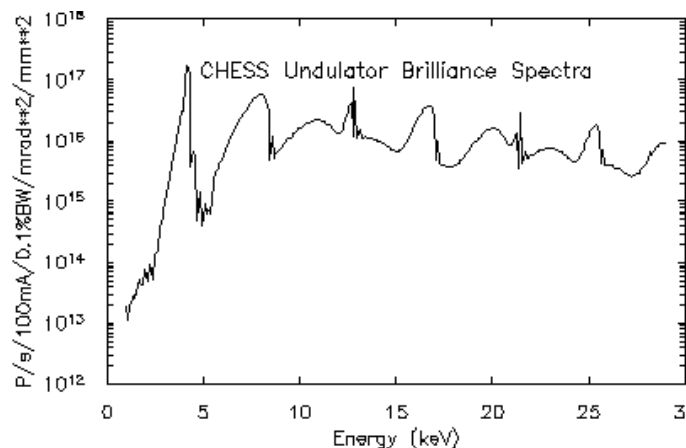
Proposals for experiments (including machine physics, x-ray optics, and x-ray science) should be submitted to CHES in the form of a two-to-four page description of the desired experiment and its significance to synchrotron radiation research by Monday, April 19, 1993.

Proposals will be reviewed by a review committee.

In the 1988 running period, successful experiments included: heat loading tests of monochromator optics^{4,5}, x-ray standing wave study⁶ on a membrane-aqueous interface, x-ray fluo-

rescence microprobe study of minerals⁷, measurement of emittance from the undulator radiation itself⁸, 120 picosecond Laue diffraction^{9,10}, and a beam position monitor study¹¹. The 1991 run included a successful test of an inclined monochromator¹²⁻¹³, beam position monitor¹⁴, cryogenic cooling of monochromator crystals, time-resolved Laue crystallography, nuclear Bragg scattering, intensity fluctuation spectroscopy, and an x-ray microprobe experiment¹⁵.

For further information about the upcoming undulator run, please contact Don Bilderback or Bob Batterman.



1. "Performance of a Hard X-ray Undulator at CHES", D.H. Bilderback, B.W. Batterman, M.J. Bedzyk, K. Finkelstein, C. Henderson, A. Merlini, W. Schildkamp, Q. Shen, J. White, E.B. Blum, P.J. Viccaro, D.M. Mills, S. Kim, G.K. Shenoy, K.E. Robinson, F.E. James, and J. M. Slater, *Rev. Sci. Instrum.*, Vol 60 (7), p. 1973, 1989.
2. "Development of an Undulator Radiation Source for the Cornell Electron Storage Ring", E.B. Blum, Doctoral Dissertation, 1989.
3. "Operation of CESR as a Low Emittance X-ray Source," E.B. Blum, *Proceedings of the 1989 IEEE*
4. "Undulator Heat Loading Studies on X-ray Monochromators Cooled with Liquid Gallium", D.H. Bilderback, C. Henderson, J.White, R. Smither, G. Forster, *Rev. Sci Instrum.*, Vol. 60 (7), p. 1973, 1989.
5. "Liquid Gallium Cooling of Silicon Crystals in High Intensity Photon Beams", R.K. Smither, G. Forster, M.J. Bedzyk, D. Bilderback, K. Finkelstein, C. Henderson, J. White, L. Berman, P. Stefan, T. Oversluizen, *Rev. Sci. Instrum.*, Vol. 60 (7), p 1486, 1989.
6. "Diffuse-Double Layer at a Membrane-Aqueous Interface Measured with X-ray Standing Waves," M.J. Bedzyk, G. M. Bommarito, M. Caffrey, and T.L. Penner, *Science* 248, 52-56, (1990).
7. "X-ray Fluorescence Microprobe Imaging with Undulator Radiation", M. Rivers, S.R. Sutton, and B.M. Gordon, *Materials Research Society Symposium Proceedings*, Vol. 143 *Synchrotron Radiation in Materials Research*, E.R. Clarke, J. Gland and J.H. Weaver, P285-290, Materials Research Society, Pittsburg, PA, 1989.
8. "Emittance Measurements of CESR Using the Emitted Radiation from a Short Period Undulator", D.M. Mills, P.J. Viccaro, A. Merlini, Q. Shen, and K. Finkelstein, *Nuclear Instruments and Methods in Physics Research*, A291, p. 481-486, 1990.
9. "120 Pico-second Laue Diffraction Using an Undulator X-ray source", D.M.E. Szebenyi, D. Bilderback, A. LeGrand, K. Moffat, W. Schildkamp, and T.Y. Teng, *Transactions of the American Crystallographic Association*, Vol 24, p. 167-172, 1988.
10. "Quantative Analysis of Laue Diffraction Patterns Recorded with a 120 Picosecond Exposure from an X-ray Undulator", D. M. E. Szebenyi, D. H. Bilderback, A. LeGrand, K. Moffat, W. Schildkamp, B. Smith Temple & T. Y. Teng, *J. Appl. Cryst.* Vol 25 (1992) 414-423.
11. "Beam Position Monitor for Hard X-ray Undulator Radiation", W. Schildkamp, submitted to NIM.
12. "High Heat Load Performance of an Inclined Crystal Monochromator with Liquid-Gallium Cooling on the CHES-ANL Undulator", A. T. Macrander, W. K. Lee, R. K. Smither, D. M. Mills, C. S. Rogers, and A. M. Khounsary, *Nucl. Instrum. & Meth.* A319(1992)188;
13. "Thermal, Structural and Diffraction Analyses of a Gallium-cooled X-ray Monochromator", C. S. Rogers, A. T. Macrander & D. M. Mills, *SPIE workshop on Optics for High-Brightness Synchrotron Radiation Beamlines*, to be published in *SPIE Proceedings* Vol. 1740, (1992).
14. "The APS X-ray Undulator Photon Beam Position Monitor and Tests at CHES and NSLS", D. Shu, B. Rodricks, J. Barraza, T. Sanchez and T. M. Kuzay, *Nucl. Instrum. & Meth.*, A319(1992)56-62.
15. "Focussing Optics for a Synchrotron-Based X-ray Microprobe", A. C. Thompson, K. L. Chapman, G. E. Ice, C. J. Sparks, W. Yun, B. Lai, D. Legnini, P. J. Vicarro, M. L. River, D. H. Bilderback & D. J. Thiel, *Nucl. Instrum. & Meth.* A319(1992) 320-325.

First microsecond time-resolved XAFS

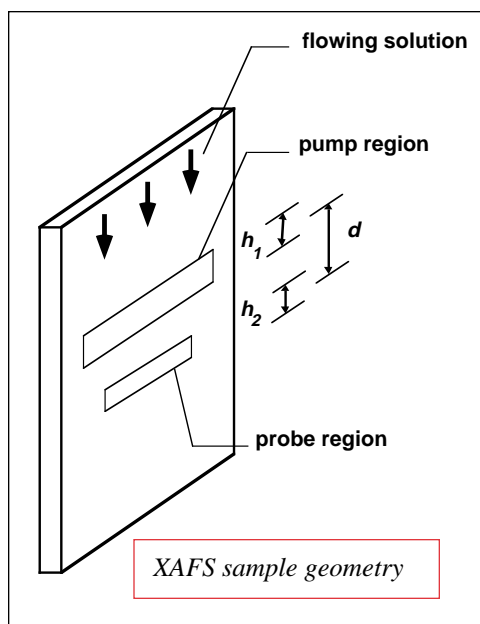
Dan Thiel

We have recently completed the first microsecond x-ray absorption fine structure (XAFS) measurement, which is the best temporal resolution ever obtained with this technique. This experiment required both a new experimental method as well as a new method of XAFS data analysis. In collaboration with P. Livins (Western Washington Univ.) and E. A. Stern (Univ. of Washington) and A. Lewis (Cornell and Hebrew Univ.), we studied the triplet excited state of the inorganic complex $\text{Pt}_2(\text{P}_2\text{O}_5\text{H}_2)_4^+$. For the first time, a direct structural method has been used to determine the molecular structure of an electronic excited state, thereby allowing us to obtain unprecedented structural detail of a transient excited-state molecule. The 1- μsec resolution of the technique has broad applications to chemistry and biology since many molecules display interesting photo-induced changes on such a time scale.

Our technique combines XAFS and rapid-flow laser spectroscopy. Flow spectroscopy has been used in Raman spectroscopy but never combined into an XAFS measurement. In our technique, the solution containing the molecule of interest is passed through a nozzle to form a free-standing jet, similar to that of a dye laser. The flowing sample (see figure) is excited by focussing the laser onto the jet, and the x-ray beam is subsequently directed at the jet downstream of the laser focus. The spatial separation of the laser beam and the x-ray beam d translates directly into a time delay $t=d/v$ with the velocity of the flowing sample given by v .

To achieve microsecond time resolution, both the exciting laser beam and the probing x-ray beam

must have small spatial dimensions along the flow direction, denoted by h_1 and h_2 . These values are typically $10\mu\text{m}$ in order to limit the time in which the flowing molecules reside in the beams. This condition on the x-ray beam puts a premium on maximizing the available x-ray intensity (flux/unit area). Our data was collected on the A2 station using a sagittally focussing monochromator to focus the synchrotron beam horizontally. Due to restricted space, no vertical focusing was used. Although the A2 station is relatively large, space was restricted primarily due to the installation of a high-power (25 W in the visible / 5 W in the ultraviolet)



argon ion laser inside the hutch, along with the various optical components.

While flowing the molecules at a velocity of about 15 m/sec, or 32 miles/hour, we excited the sample with 0.75 W of laser light which excited about 18% of the illuminated $\text{Pt}_2(\text{P}_2\text{O}_5\text{H}_2)_4^+$ molecules into the triplet state. By reducing the time delay to 0.8 μsec , we easily observed the triplet state which had a 4- μsec lifetime in our aqueous glycerol solution.

The fluorescence x-rays from the Pt L_{III} absorption edge were collected at a count rate of 2400 cps.

The analysis begins by comparing the excited state to the ground state (no laser) data. We subtracted the post-edge background using a procedure developed by the U. of Washington group. We found it beneficial to use the results of the proven computer code FEFF developed by the group of J. J. Rehr (U. of Washington) to calculate the XAFS amplitudes and phases.

From our analysis, we have been able to determine the change in three of the molecule's radial parameters upon excitation to the triplet state. This determination indicates that the molecule undergoes an overall contraction when excited. Our technique has allowed us to determine the contracted values with a precision as small as 0.01 \AA although the errors in our parameters are large, typically 20%.

By accomplishing the first μsec XAFS measurement, we have obtained the first XAFS data from an electronic excited state. We expect many more interesting results will emerge from this powerful technique especially in regard to biological molecules. Finally, we are developing better x-ray focusing devices such as tapered glass capillaries to improve this type of measurement.



Dan Thiel

High pressure

facility at B1

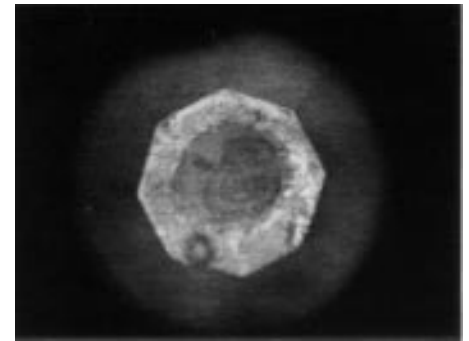
Keith Brister

The CHESS High Pressure Facility exists to provide experimental support for high pressure experiments with diamond anvil cells. In these cells, pressures from around 0.1GPa (1kbar) to well over 100GPa (1Mbar) can be generated. The facility has two stages for mounting diamond anvil cells. One is permanently installed in the B1 hutch while the second apparatus can be used in any of the larger stations (A2, A3, D1, F1, or F2). In addition there is a microscope and some software support for preliminary data analysis. Energy dispersive diffraction is generally used at B1 although some Laue experiments have also been done.

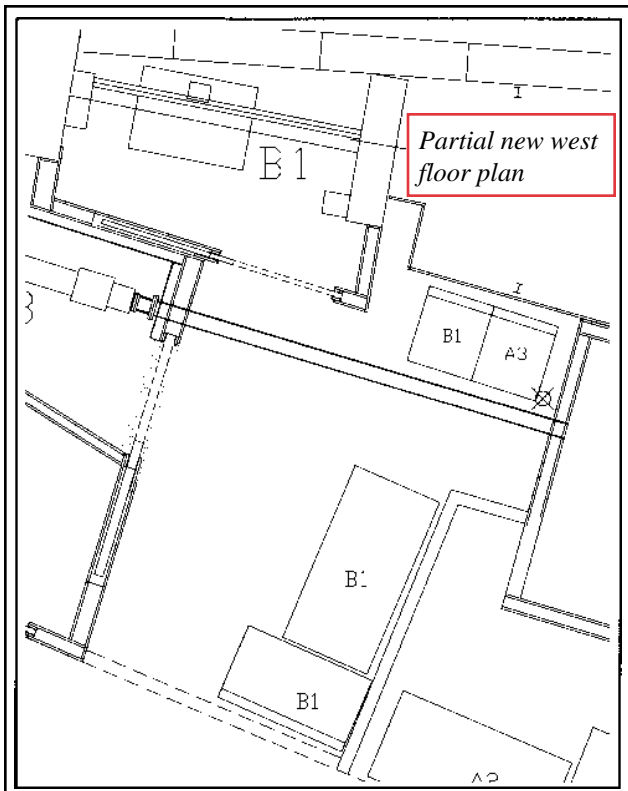
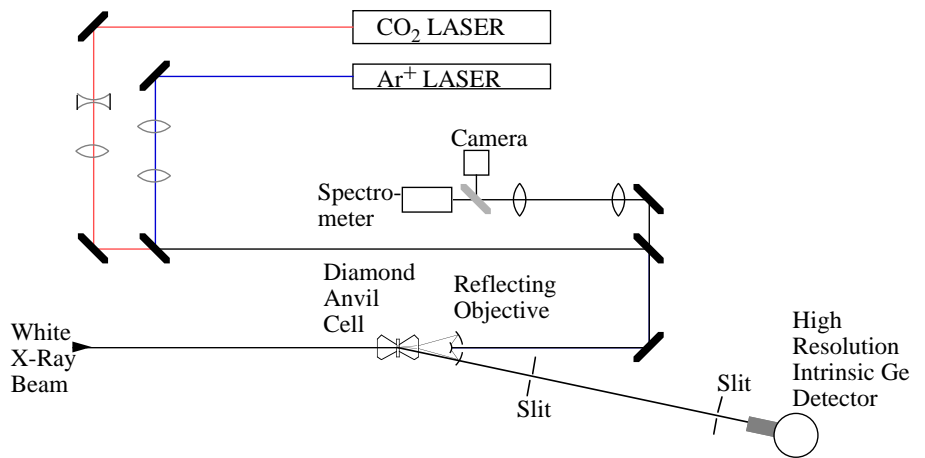
A schematic of the diffraction and optical system in the B1 hutch is shown above. Currently the Ar⁺ laser is used for ruby and Sm-YAG pressure measurements. The first version of the optics, installed in February 1992, were difficult to align and use, however, recent improvements should make the setup very easy: Beam-steering mirror mounts are being replaced with kinematic mounts with precision adjustments and several new degrees of freedom have been added to make it easier to direct the argon ion laser light.

Experiments using the CO₂ heat-

ing laser are planned for as soon as the A-Line construction is complete enough to allow B-Line to operate.

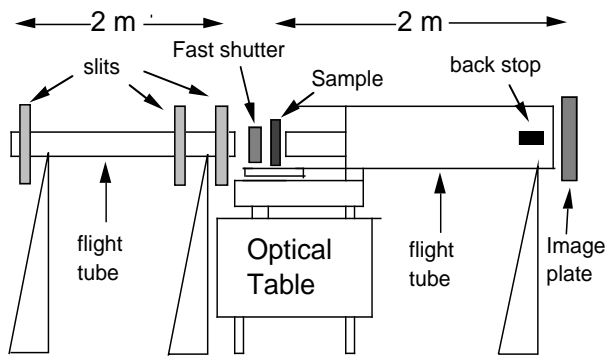


Sample in diamond anvil cell after heating to 2300K with CO₂ laser.



The second apparatus has been used in A3 for energy dispersive diffraction and in A2 for collecting Debye-Scherrer data as well as for collecting XAFS data.

The new A-Line reconstruction will affect the High Pressure Line at B1 in several ways. First, much of the reconstruction cannot take place while B1 is operating, so B1 will be down from the beginning of January until utilities can be restored to B1 and the dust has settled a bit. Second, the B1 hutch will be enlarged somewhat and the door will be replaced by the "east" EZ-Slide style (see figure). Third, the floor plan will be changed significantly: The computer will be placed much closer to the experiments and the laser curtain will be placed so that all the High Pressure Line equipment and computer will be accessible while the curtain is closed. The entire B1 floor plan will be under the 8foot ceiling and a stud wall will be placed between B1 and A2 so the noise and traffic problems that currently plague the B1 users will be eliminated. Since A3 will no longer be a working experimental station during normal running, the B1 users will not have to share the space around the B1 door with the A3 users. Although the changes will require B1 to be off-line for two or three months, the new facility will be a much better place to do experiments.



Small-angle muscle diffraction facility for F1

Tom Irving

X-ray diffraction is the only technique that can obtain high resolution structural information from muscle in the living, hydrated state. X-ray diffraction of muscle make unusually high demands on X-ray sources and optics. Muscles diffract relatively weakly, have many components with similar long spacings (thus diffraction is at very small angles) and the changes of physiological state in muscle and the accompanying structural changes occur in a time scale of milliseconds or a few tens of microseconds.

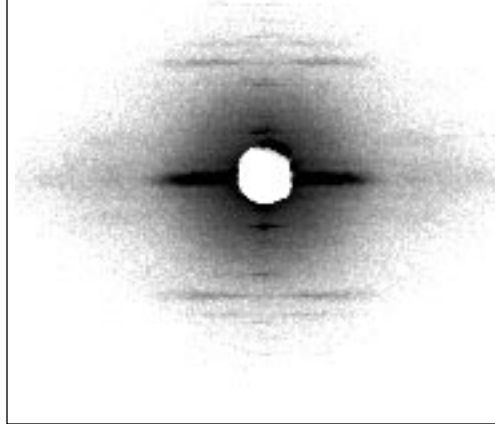
The promise of synchrotron radiation as a source of high intensity, well collimated X-rays for time resolved studies of muscle was recognised as early as 1971 by G. Rosenbaum and colleagues. This promise can be realized in studies that produce full two-dimensional diffraction data at high time resolution during rapid mechanical transients, thus establishing a link between muscle structure and muscle physiology.

The major impediment to reaching this goal so far has been lack of x-

ray flux. All synchrotron small angle diffractometers that have been used for muscle studies to date are on bending magnet beam lines. Here we report preliminary results from, and characterization of a small angle diffractometer built (in collaboration with H.E. Huxley, Brandeis University) for the F-1 station, an exceptionally high flux wiggler beam line.

The camera that was ultimately constructed (see above) was first tested in August of 1992. The collimation system consists of three sets of horizontal and vertical slits connected by a 1.8 m long evacuated flight tube with mica windows. The four jaws of each set of slits are individually stepper motor controlled.

50 ms exposure from sartorius muscle



Downstream of the mica window is a solenoid shutter capable of ~40 ms opening times which incorporates an ion chamber for beam intensity monitoring. This can be used in conjunction with a chopper type shutter to yield millisecond or submillisecond exposures. The sample is mounted on a two-axis vertical stage that can be used to move the sample in a raster pattern in the beam to reduce the radiation damage in any given part of the sample.

Downstream of the sample position is an evacuated flight tube, adjustable in length from 80 cm to 2 m.

mounted at one end to the X-95 rail and at the other to a stand with motorized x-y motions to align the beam stop. The focal spot size is about 3 x 0.3 mm FWHM at the 5 m point in the hutch, containing as much as 10^{13} photons/s at 13.65 KeV (0.91 Å) and 85 mA ring current, a factor of 20 greater than competing beam lines at Daresbury and the Photon Factory.

The middle figure shows a diffraction pattern from an isometrically contracting whole sartorius muscle from bullfrog muscle taken in 50 ms using a Fuji image plate with the Fuji BAS2000 scanner. Since it is possible to get over 100 tetanic contractions from a frog muscle, this rapid exposure time means that any of the features visible on this image can be resolved to 1 ms with a single specimen and with good spatial resolution. Order to order spatial resolution is (on image plates) in excess of 2500 Å.

Clearly, the F-1 small angle camera will be an excellent tool for time resolved studies of muscle, with particular application to the weaker diffraction features such as the actin layer lines. The short exposure times make it possible to obtain two dimensional diffraction data with millisecond time resolution in combined mechanical and X-ray experiments on single muscle specimens. For more information, contact Tom Irving at 607-255-2174/3607.



Tom Irving

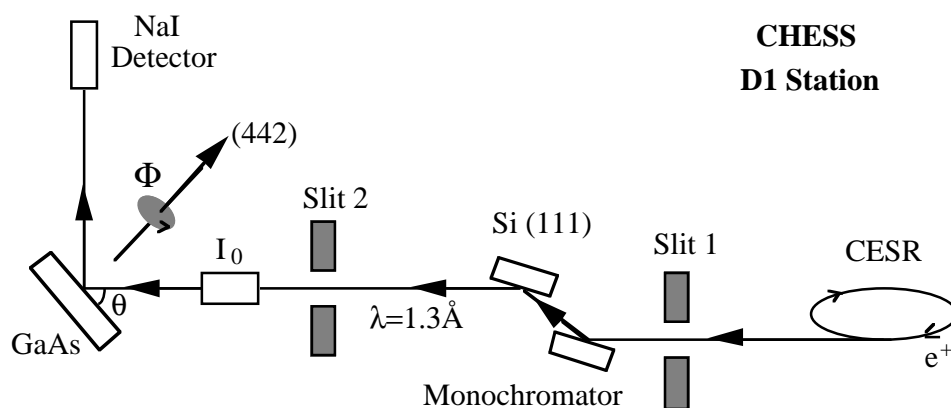
New analyzer for circularly polarized x-rays

Qun Shen
Ken Finkelstein

A unique type of circular polarization analyzer for x-rays has been tested at CHESS [Phys. Rev. B45, 5075 (1992)]. Its potential areas of application range from the characterization of synchrotron radiation from bending magnets and special insertion devices to analyzing polarization in magnetic and/or resonant scattering experiments.

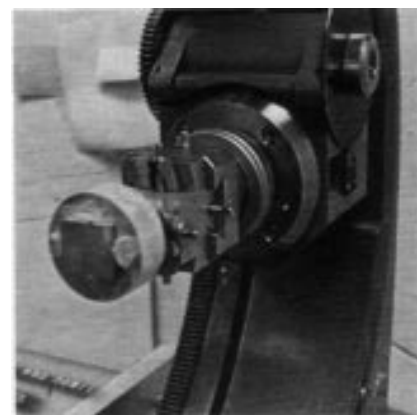
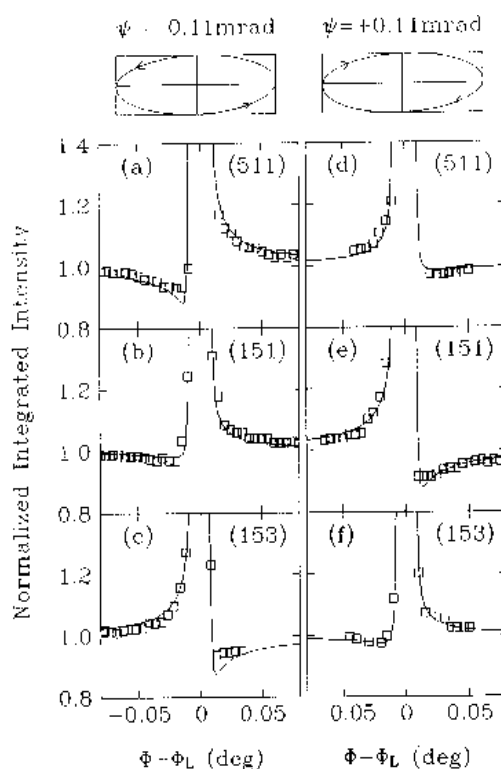
The method utilizes multiple-beam Bragg diffraction from a single crystal. Two main effects that can happen in a multi-beam diffraction process are: 1) phase sensitive interference among the multiple Bragg beams and 2) polarization state mixing because of the double Thomson scattering. The two effects combine to yield an interference intensity in the primary reflection which depends on the magnitudes of the *s*- and *p*-components of the *E*-field and also on their relative *phase*. Thus by measuring the interference intensity as the crystal is rotated through a multiple reflection one can obtain the purity of the circularly polarized component in the incident beam.

With the data in the figure we demonstrate that a complete characterization of a general, elliptical polarization of x-rays is possible. The experiment was performed at CHESS D1 station which receives bending magnet radiation from positrons. In the experiment we measured three independent multiple-beam diffraction profiles on GaAs using the radiation 0.11 mrad above and (separately) 0.11 mrad below the CESR orbit plane. Presumably the only difference between the radiation selected at



these two positions is the handedness of the elliptical polarization. As one can see from the figure, the intensity profiles are asymmetric. For each reflection the difference in this asymmetry for the two incident beams is a demonstration of the sensitivity that multiple-beam has to circular polarization. By fitting the data to a perturbation theory which includes the multi-beam effects [Shen, Acta Cryst. A42, 525 (1986)], we were able to obtain the Stokes-Poincare polarization parameters $(P_1, P_2, P_3) = (0.77 \pm 0.18, 0.05 \pm 0.40, 0.57 \pm 0.07)$ above and $(0.77 \pm 0.18, 0.05 \pm 0.40, -0.57 \pm 0.07)$ below the orbital plane.

Because it utilizes Bragg reflections from crystals, the new multi-beam diffraction technique has the advantages that it can provide strong signals and can be applied in a wide energy range. We believe it will be especially useful for energies below 20 keV where the magnetic Compton scattering has a weak cross-section. It would be very interesting to use this analyzer in a magnetic scattering experiment where it would provide direct information on x-ray polarization due to the magnetic interaction.

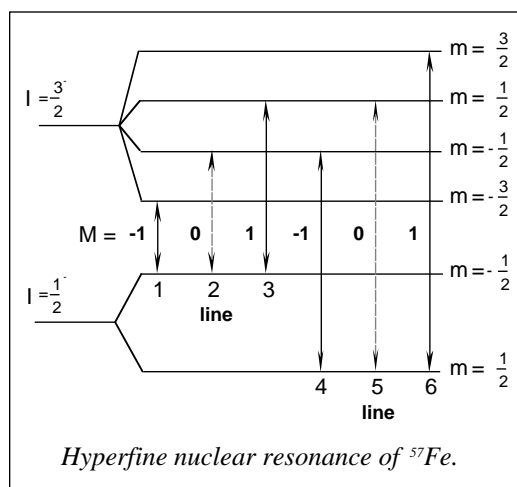


Goniometer on Picker 4-circle

(left) Multiple-beam interference profiles measured on the GaAs(442) main reflection. Data in (a)-(c) were taken using the synchrotron radiation emitted at 0.11 mrad below the positron orbital plane and those in (d)-(f) were using radiation at 0.11 mrad above the orbital plane. The solid curves are theoretical calculations fit to the data, as discussed in text. The polarization ellipses determined are shown in the top insets.

Coherent nuclear resonance scattering

Sarvjit Shastri



The coherent X-ray scattering by atomic nuclei is practically unobservable due to the large nuclear mass. However, certain nuclei, Mössbauer isotopes with low-lying excited states, exhibit resonant scattering when the incident photon energy is tuned to the energy difference between the excited and ground states (see **figure**). The scattering resonance width is small ($\sim 10^{-9}$ eV at 14.4 keV for isolated ^{57}Fe) but is of considerable strength so that one is able to see the effect. This scattering phenomenon is not only anticipated to serve as a useful ultrahigh resolution monochromator ($\Delta E/E \sim 10^{-11} - 10^{-14}$) for subsequent scattering studies, but is also of fundamental physical interest due to the numerous peculiar quantum mechanical effects manifested. The most striking effect is the delayed nature of the coherent scattering after synchrotron pulse excitation. This delay in the appearance of the diffracted photon serves as a way to discriminate the nuclear scattering from the much stronger, prompt electronic scattering and fluorescence background.

The F2 wiggler station is often used for nuclear resonance scattering studies. John Arthur (of SSRL) and coworkers have used (Phys. Rev. Lett. **69**, 699 (1992)) the nuclear Bragg diffraction from ^{57}Fe -enriched yttrium-iron-garnet to illustrate the rotation-induced phase shift imparted to the quantum state of a particle (photon). J. Tischler and B. Larson

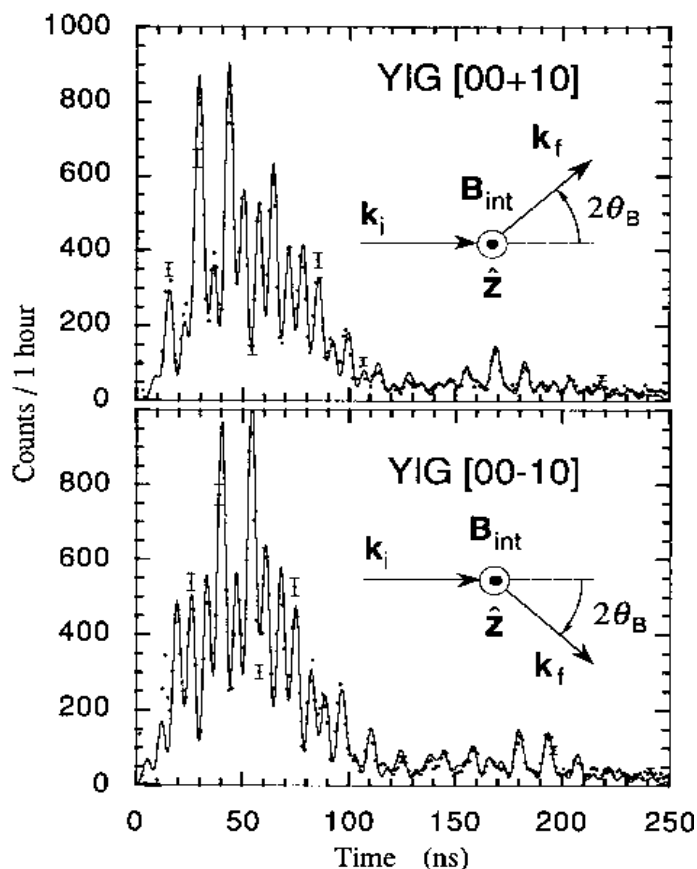
(ORNL) have been using the resonantly diffracted beam from $^{57}\text{Fe}_2\text{O}_3$ to look for quasi-elastic scattering in other non-resonant samples that undergo soft-mode structural phase transitions (eg. BaTiO_3).

Until recently, practically all work has been with ^{57}Fe -enriched samples (^{169}Tm diffraction from Thu-

lium-iron-garnet has been observed by W. Sturhahn and co-workers at HASYLAB). However, E. Alp (ANL) and coworkers have seen, at F2, resonant scattering for the first time from ^{119}Sn (resonant energy 23.87 keV). They observed nuclear diffraction from a $^{119}\text{Sn}/^{120}\text{Sn}$ multilayer as well as the nuclear reflection suppression of the electronic reflectivity at some angle of incidence.



Sarvjit Shastri



Time beat patterns for right- (top) and left- (bottom) handed scattering. Solid curves are dynamical diffraction theory calculations, including the angular phase. Because some of the interfering transitions share identical M values, the shifted beat patterns are modulated with an unshifted beat pattern of a longer period, giving different heights to corresponding intensity peaks in the [0010] and [00-10] patterns.

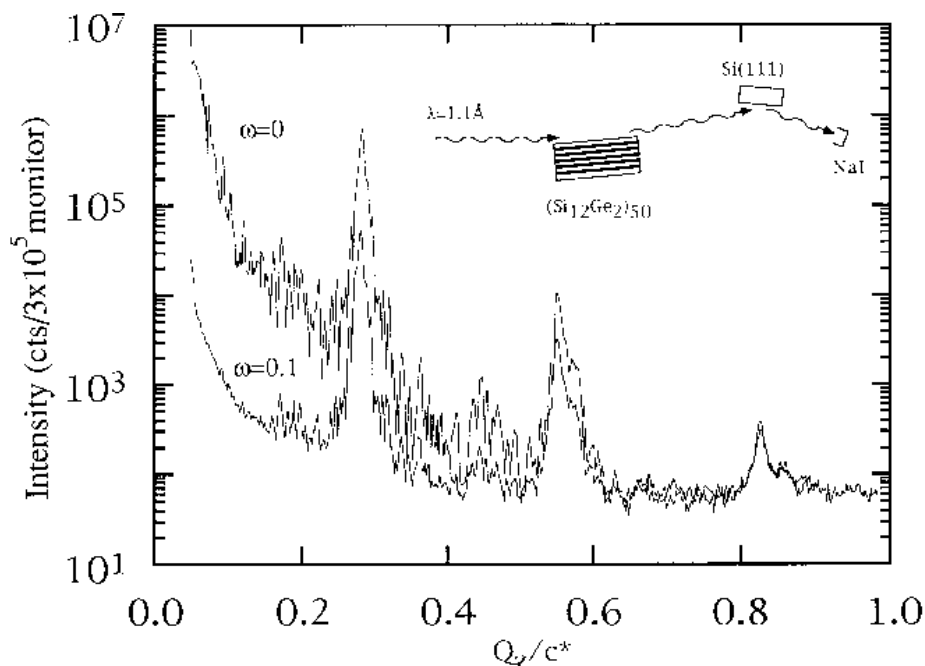
High resolution diffraction facility at F3

Randy Headrick
Peter Quigley

The F3 station is a relatively new experimental station providing focussed monochromatic beam to a semi-permanently installed high resolution spectrometer. The station is optimal for a number of experiments such as general purpose diffraction, low angle scattering, and anomalous scattering with NaI scintillation counters or position-sensitive detectors. The philosophy is to provide a facility for a wide range of experiments using a standard configuration in order to minimize setup time.

Focussing is achieved by a bent, slotted Si(111) crystal monochromator producing $>10^{11}$ ph/sec for typical experimental geometries. The table below shows the performance of F3 and other comparable stations at CHESS (comparable mean 2-3eV energy resolution at 10-13keV):

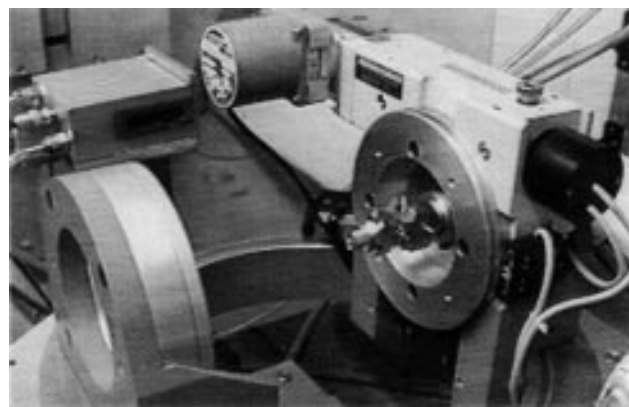
The F3 spectrometer is a Huber 424 two-circle goniometer with a Huber 511.1 Eulerian cradle, also known as a small Huber 4-circle. A motorized analyzer assembly has been added for a 6-circle configura-



tion. This spectrometer with analyzer has been used for a number of applications including: to separate closely spaced features in diffracted beams, to reject Compton scattering from diffracted beams, and to reject the incident beam for low-angle scattering experiments.

The figure above shows an example of data that was obtained recently at F3 from a $(\text{Si}_{12}\text{Ge}_{250})$ superlattice. Scans with different settings of the analyzer crystal were used to separate the true specular reflected beam ($\omega=0^\circ$) from diffuse scattering caused by roughness ($\omega=0.1^\circ$). The $\omega=0^\circ$ curve is a q-2q scan showing three orders of superlattice Bragg reflections. The period of the superlattice is 14 monolay-

ers giving rise to the Bragg peaks at $L=4/14, 8/14,$ and $12/14$ for the first three orders. The $\omega=0.1^\circ$ curve is an "offset" q-2q scan of the diffuse scattering. Resonant features, when Q_z matches the reciprocal superlattice spacing, and closely spaced satellites in the diffuse scattering match the satellites in the specular scattering. This data has been used to determine



CHESS sagittal focussing summary

| station | source (W=wiggler) | magnification | energy range (keV) | brightness @10keV (ph/s/100mA/mm ²) |
|---------|-----------------------|---------------|-----------------------|--|
| A2 | W | 0.59 | 5-25 | 1×10^{11} |
| C2 | HB | 0.27 | 5-12 | 2×10^{11} |
| D1 | HB | 0.32 | 5-15 | 1.5×10^{11} |
| F2 | W | 0.32 | 5-22 | 6×10^{11} |
| F3 | HB | 0.33 | 5-20 | 1.5×10^{11} |

the structure of roughness at the interfaces of the superlattice.

The F3 station is available for feasibility studies, express mode proposals, and also for regular (long term) projects. For more information call Randy Headrick, (607-255-0919)

Operations and scheduling

Jeff White
Lana Walsh

Along with the advancements on the beamlines and at the stations that have been discussed by others, there are other changes taking place to increase the productivity of the available beam time. As of this past summer, with the agreement of the accelerator physicists and the High Energy Physics experimenters, we have instituted a new machine schedule to have every third week of running be *uninterrupted* by machine studies. This not only helps users that need a lengthy amount of beam time but also improves on the day to day running of CESR. Starting in December, as another aid to improving day to day running, we have begun to test a machine schedule that reduces the daily accesses to the ring from three per week to two.

To assist long-range planning, we include (at right) an approximate schedule for the coming calendar year.

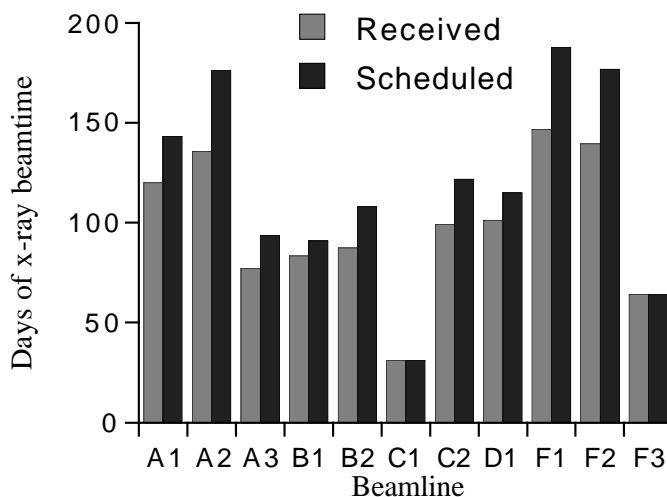
Tentative 1993 CHESSE operations summary

- January Normal operations, except A-line shutdown Jan. 11.
- February CESR shutdown Feb. 1.
- March Normal operations after CESR startup. Proposal submission deadline.
- April Normal operations.
- May Normal operations.
- June CESR shutdown. CHESSE user's meeting June 22-23.
- July Normal operations after CESR and new A-line startup.
- August Normal operations.
- September Normal operations.
- October CESR shutdown. Dedicated undulator run. Proposal submission deadline.
- November Normal operations.
- December CESR shutdown.

Lana Walsh (left) and Jeff White



During the period 10/91-10/92, CHESSE provided a total of 1300 user-days of beamtime, or 3.55 user-years! The graph at right shows a comparison, by beamline, of the number of days scheduled versus received: the difference is due to unscheduled machine maintenance and/or equipment failure. With newly upgraded equipment and a redistributed schedule for machine maintenance and studies, we plan to deliver considerably more x-ray user-days in the upcoming year.





Contacting CHESS staff

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 FAX: 607-255-9001

CHESS/MacCHESS Staff

| | |
|------------------|-----------------|
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| Steve Ealick | Peter Quigley |
| Ken Finkelstein | Dana Richter |
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| Steve Hoffman | Qun Shen |
| Randy Headrick | Mike Sloand |
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Phone extensions: all numbers are (USA) 607-255-XXXX

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| | <i>Associate director, MacCHESS</i> | |
| Virginia Bizzell | <i>Administrative manager</i> | -0922 |
| Lana Walsh | <i>Proposal administrator</i> | -7163 |
| Jeff White | <i>Operations manager</i> | -7163 |

For technical information about specific beamlines or scheduled experiments, please refer questions to the appropriate beamline coordinator:

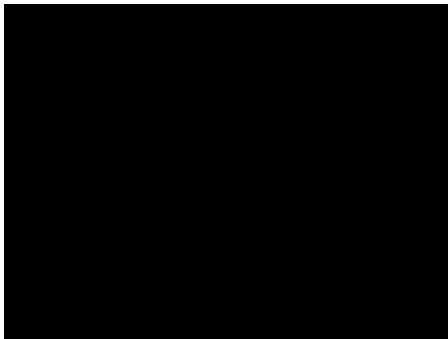
| | | |
|-------|-----------------|------------|
| A1/F1 | Don Bilderback | -0916/0643 |
| A2/F2 | Qun Shen | -0923 |
| B1 | Keith Brister | -0920 |
| B2/C2 | Ken Finkelstein | -0914 |
| D1 | Ernie Fontes | -2959 |
| F3 | Randy Headrick | -0919 |



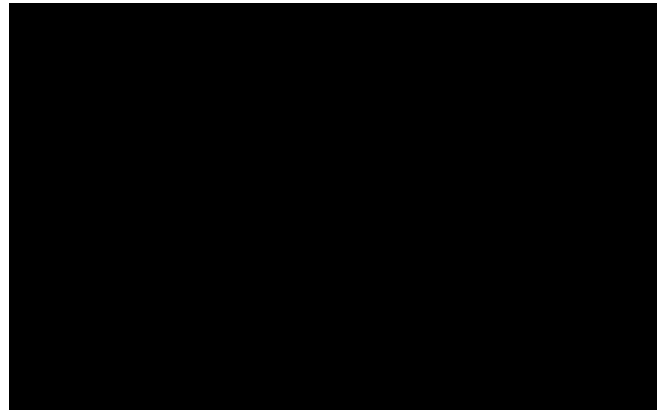
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CHES T-shirts are available, contact Virginia Bizzell



*Stewart Brazil, Cornell
Co-op student and a co-
designer of the new A-
line wiggler*



**Cornell High Energy
Synchrotron Source**

Wilson Laboratory
Cornell University
Ithaca, N.Y. 14853-8001

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