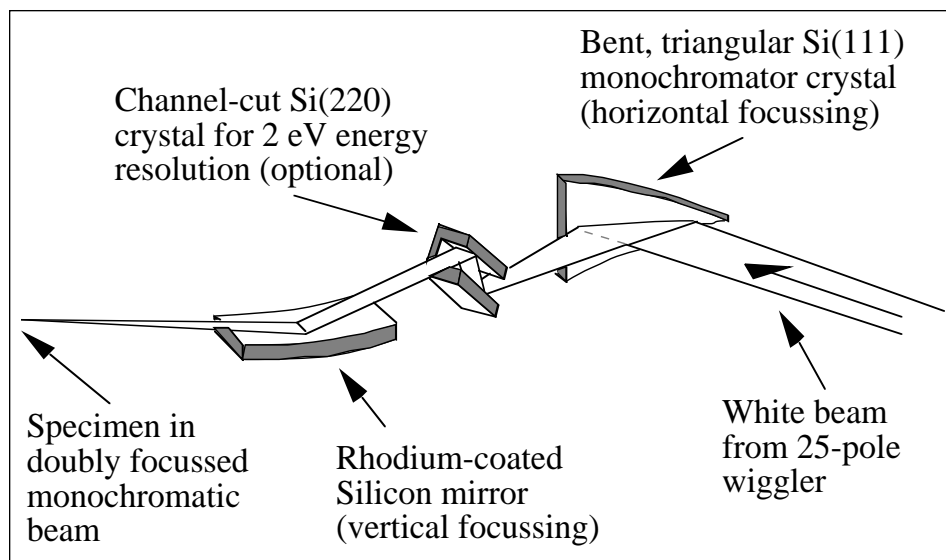


F1 optimized for x-ray crystallography

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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



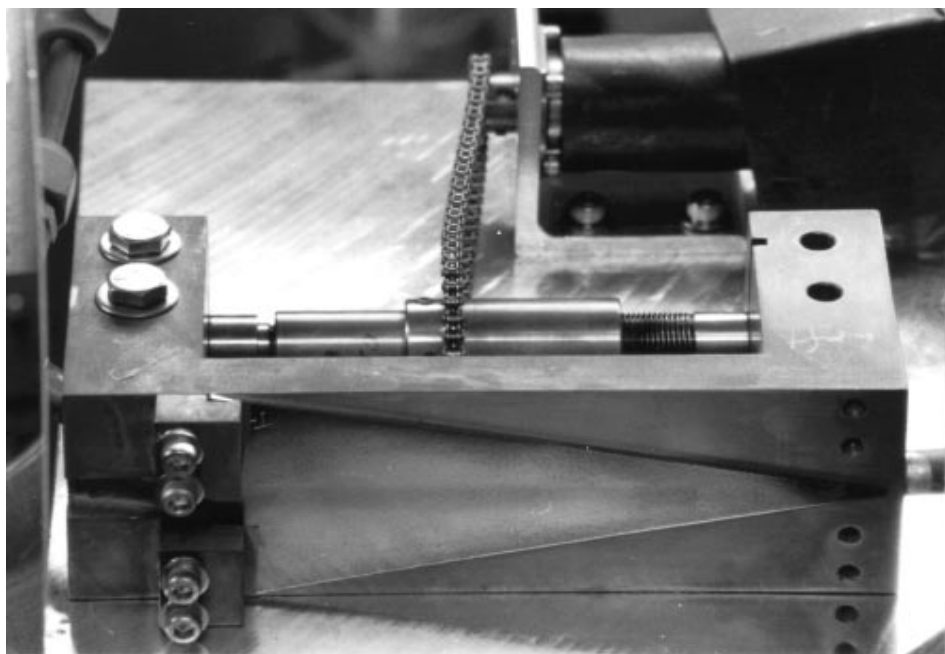
bent rhodium-coated silicon mirror. The typical focal spot size at the specimen for 13keV photons is approximately 2mm horizontal by 0.4mm vertical.

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).