The Development of the GCPCC Protein Crystallography Beamline at CAMD

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Abstract. The Gulf Coast Protein Crystallography Consortium (GCPCC) is developing a beamline at the LSU/CAMD synchrotron. This beamline will be capable of standard macromolecular multiple-wavelength anomalous diffraction (MAD) phasing experiments over an energy range of 7-17.5 keV. The optical configuration uses a vertical collimating mirror, a channel-cut Si (111) monochromator and a focusing toroidal mirror. Built off of the CAMD 7 T superconducting, energy-shifting wiggler, this beamline will deliver a flux comparable to an NSLS bending magnet protein crystallography (PX) beamline. The beamline delivery and commissioning timetable calls for full 24/7 user operations by October 2001. The beamline design, with supporting calculations and ray tracing, is presented to substantiate the expected performance of this beamline for efficiently collecting accurate MAD data from macromolecules.

INTRODUCTION

The Gulf Coast Protein Crystallography Consortium (GCPCC) members include Louisiana State University, Rice University, University of Texas Medical Branch, Baylor College of Medicine, Oklahoma Medical Research Foundation, University of Texas – Austin, University of Houston, and Texas A & M University. In addition to providing access to the consortium laboratories, the GCPCC beamline will allocate 25 % of the beamline time to general users. The beamline is designed to collect protein crystallography MAD phasing data between 7-17.5 keV. This range includes K edges from iron to yttrium and L edges from neodymium to uranium.

The radiation source for this beamline is a 7 T superconducting, energy-shifting wiggler at the Center for Advanced Microstructures and Devices (CAMD) in Baton Rouge, LA [1]. The critical wavelength for the wiggler is 1.18 Å (10.5 keV) when operated at 7 Tesla with a 1.5 GeV electron beam. The X-rays produced by the CAMD wiggler at 1.5 GeV are harder than those from an NSLS bending magnet at 2.58 GeV. This results in a slightly higher flux/mA at the Se K edge for the CAMD source. The wiggler has five poles. The magnitude of the flux produced by the superconducting side poles relative to the central pole is 16% at 7 keV, 3 % at 12 keV, and 0.6% at 17 keV.

OPTICAL DESIGN & RAY TRACING

The optical design is typical of modern beamlines optimized for MAD. A qualitative anamorphic diagram is shown in Fig. 1. The first mirror collimates the beam vertically. Collimation increases the flux transmitted by the slits/monochromator for a given bandwidth. This maximizes the resolution of the monochromator. The mirror also reduces the thermal heat load on the first monochromator crystal by acting as a low pass filter. The wavelength is selected by a Si (111) channel-cut monochromator. Si (111) has a rocking width of 21 mrad at 12.7 keV and this results in an intrinsic energy resolution ($\Delta\lambda/\lambda$) of 1.33×10^{-4} [2]. The experimentally realized energy resolution is lower because of the effects of beam divergence and thermal deformation of the crystal surface. The second mirror focuses the beam horizontally and vertically on the experimental sample.

The program Shadow [3] was used to model the beamline and evaluate the optimal size and placement of the optical elements. Over the small acceptance of the beamline considered here, the variation of the radius of curvature of the electron orbit is negligible. For the purpose of modeling, the source is represented as a simple synchrotron with a bending radius of 0.71 meters. For heat-load calculations, all five wiggler poles are considered.

CP576, *Application of Accelerators in Research and Industry – Sixteenth Int'l. Conf.,* edited by J. L. Duggan and I. L. Morgan

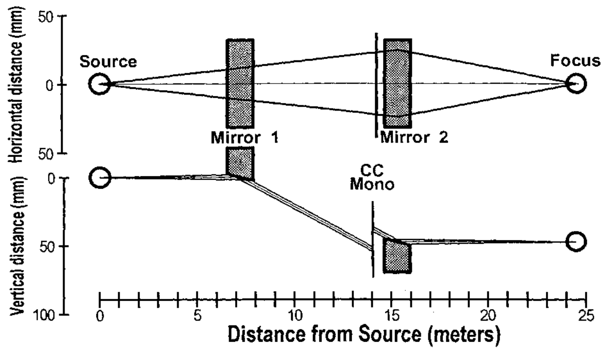

FIGURE 1. Anamorhic diagram of the PX beamline showing the location of the vertical collimating mirror (M1), the channel-cut monochromator, and the refocusing toroidal mirror (M2).

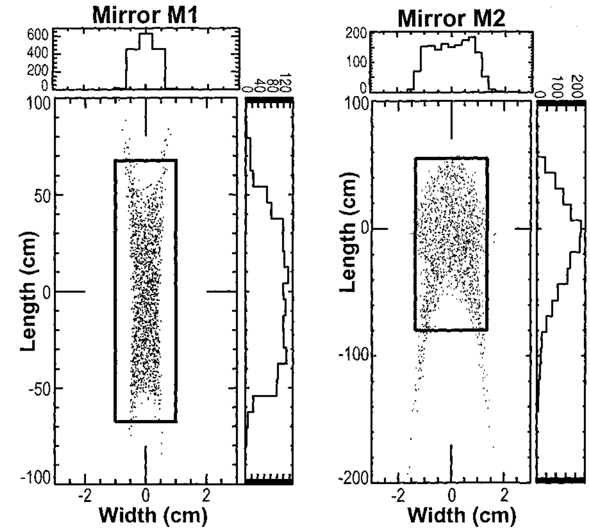

FIGURE 2. Scatter plot showing the location of the backtraced rays (those that made it through the 200 μm sample aperture) on the surface of the two mirrors at 12.7 keV. Note that the 1.4 m long mirrors capture most of the rays making it through the sample aperture. The pole of M2 is not at its center.

The location of the focusing mirror (M2) presents a trade-off between flux and the horizontal divergence at the sample. By de-magnifying the beam horizontally, the flux through a given aperture can be increased but the divergence of the beam transmitted through the aperture also increases. After comparing the flux and beam divergence at the sample for a several different demagnifications, the value of 0.55 was selected. The 0.55 demagnifying optics of the GCPCC beamline results in 2.3 horizontal milliradian divergence at the crystal with good flux. The divergence at the crystal can be reduced with the slits upstream of the monochromator at a cost in flux. This may be useful for some projects with long unit cells. When the horizontal crossfire is limited to 1.8 milliradians, the flux is 15 % less then that at 2.3 milliradians.

After ray tracing the complete beamline with "infinite" optics, that is optics which have unlimited length, rays which made it through the final aperture were back-traced through the system. This allows one to look at the distribution of rays that made it through the aperture at any point along the beamline. The backtraced rays are used to verify the correct dimensions for a mirror. The footprint of the rays on the surface of mirrors is shown in Fig. 2 with a box drawn to represent the size of the optics we will use (1350 mm x 20 mm for M1 and 1350 x 27 mm for M2). These optics are large enough that most of the rays strike the useable surface, although some rays are lost, especially at low energy where the opening angle of the source is larger.

The GCPCC beamline focal spot is shown in Fig. 3. The scatter plot shows rays over a very large area (8 mm x 4 mm). However, if one looks at the contour plot, the peak is centered over a much smaller area (0.4 mm x 1.5 mm). The fact that the spot size is somewhat larger than the sample aperture should make the beamline less sensitive to small fluctuations in beam position.

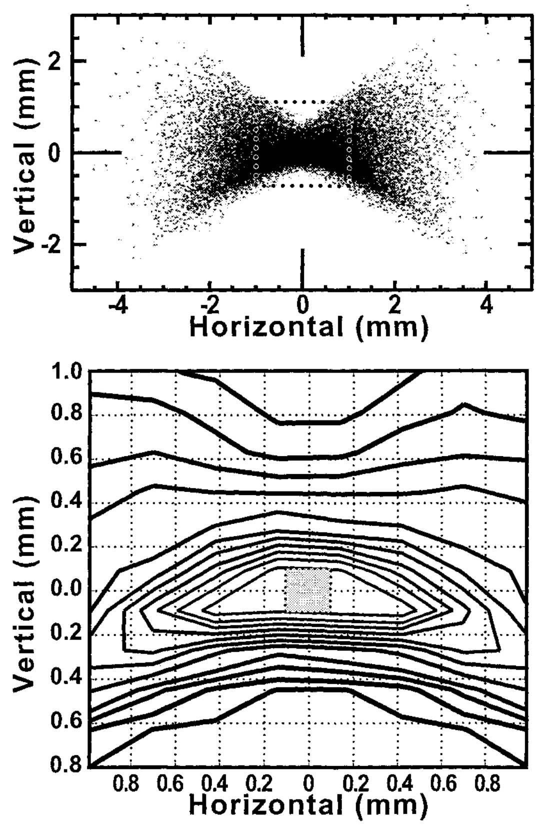

FIGURE 3. Focused spot at sample. Top scatter plot. A 200 μm reference aperture drawn on the lower contour plot.

To provide an estimate of the flux that the GCPCC beamline will deliver, we modeled the NSLS beamline X8C using the same methods as were used for the GCPCC beamline. X8C has optical components that are similar to the GCPCC design. There is a rhodium coated collimating mirror, a Si (111) double crystal monochromator, and a rhodium coated focusing mirror [4,5]. The flux ratio of the two model beamlines is GCPCC/X8C = 0.988. This represents the intensity obtained from the weighted sum of the rays that make it through the 200 micron aperture for the bandpass of the monochromator. At energies higher than 8.75 keV, the CAMD wiggler produces more photons/mA of circulating current at 1.5 GeV than an NSLS bending magnet at 2.58 GeV. Differences in flux will come down to differences in the average circulating current of the storage rings. Intensities measured in 1997 on NSLS bending magnet PX beamlines at 1 Å wavelength under 2.58 GeV operations normalized to 300 mA with a 200 μm square aperture show that the flux at different beamlines varies by a factor of 2-3 (P. Siddons, personal communication). Based on these measurements and calculations, the GCPCC beamline should deliver a flux within the range of existing NSLS bending magnet PX beamlines.

To estimate the heat load, we consider all five wiggler poles and assume that we accept 3 milliradians with an electron beam current of 400 mA. This is a reasonable current for estimating maximum heat load. Using these parameters, the total power output from the source is 160 Watts. The first mirror and beryllium window serve to reduce the heat load on the monochromator crystal. These two elements combine to remove approximately 50 % of the total power output in the 3 mrad fan allocated to the PX beamline. Cooling water will be used to remove heat from the fixed aperture, the collimating mirror, the first beryllium window, the adjustable slits, the first optical surface of the channel-cut monochromator crystal, the white beam stop and photon shutters.

IMPLEMENTATION

Beamline development on the CAMD wiggler is being carried out in two phases (TABLE 1). In the Phase 1 configuration, two beamlines are to be developed: a micromachining beamline and the protein crystallography beamline. The present vacuum chamber in the dipole bending magnet immediately downstream of the wiggler allows 25 milliradians of the radiation fan from the wiggler to be brought out of the accelerator. A new dipole vacuum chamber is being developed with two additional flanges allowing extraction of more wiggler radiation. In both Phase 1 and Phase 2, the two beamlines closest to the centerline of the wiggler fan have a common interface to the dipole vacuum chamber. Additionally, in both configurations, the extreme angular extents of wiggler radiation accepted by these two beamlines are plus and minus 12 milliradians. To limit disruption to the PX beamline during the Phase 2 dipole chamber upgrade, the front-end design was constrained to not require the repositioning of any of the PX beamline optical components. The M1 mirror tank is common to the two beamlines closest to the wiggler centerline.

TABLE 1. Utilization of the Wiggler Fan (Phase 1 & 2).

Fan	Phase 1 Use	Fan	Phase 2 Use
Outside		9 mrad	Lithography
		19 mrad	Gap
9 mrad	Lithography	3 mrad	Beamline #4 [a]
3 mrad	Gap	9 mrad	Gap
——	Center of Wiggler Fan		——
9 mrad	Gap	9 mrad	Gap
3 mrad	PX beamline	3 mrad	PX beamline
		25 mrad	Gap
Inside		3 mrad	DCM beamline

[a] This beamline is currently uncommitted.

Diagnostic screens are located between each optical element to aid in commissioning. This allows the effect of each optical element to be monitored independently without adjusting the other elements. A motor control system is being developed to allow the optical elements to be precisely aligned. The system uses Compumotor 6K series motion controllers and the MX beamline control software [6]. A graphical user interface is being developed to provide the necessary control for commissioning and routine user operations. The motor control system gives the beamline staff control over Mirror M1 – height, pitch, and roll, 4 independent blades of the adjustable aperture, Monochromator – pitch, yaw and detune, Mirror M2 – bend, height, pitch, yaw and horizontal position. In addition, there is a motorized slit unit inside the hutch, which serves to define the beam extents on the sample. The diagnostic screen actuators are also controlled through this system.

Mirrors

Both mirrors are 1.4 m long with useable lengths of 1350 mm. The mirrors are coated with rhodium, which is free of absorption structure over the energy range of the beamline. The grazing angle of 0.20 degrees (3.5 mrad) provides good reflectivity until 18 keV. The first mirror functions to vertically collimate the beam before the monochromator crystal. We are approximating the ideal parabolic figure with a ground cylinder with a radius of 4154 m. To reduce the vertical size of the collimated beam, the mirror is placed as close as possible to the wiggler source (7.25 m). This first optic is fabricated from single crystal silicon and is cooled using cooling blade. This mirror is installed in a "bounce down" configuration.

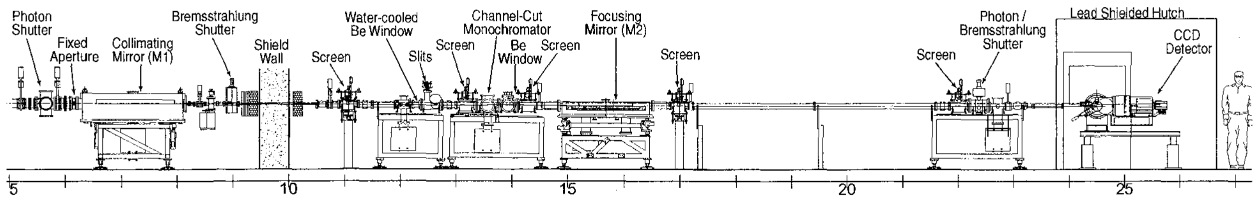

FIGURE 4. Elevation schematic of the GCPCC PX beamline.

The second mirror serves to focus the X-ray beam horizontally and vertically. Its ideal ellipsoid focusing figure is approximated with a toroid. The surface figure is realized by bending a long mirror into which the 3.92 cm sagittal radius had been ground. The tangential radius is adjustable between 4–6 km using a stepper motor driven U-bender. By adjusting the bending radius and the inclination angle, the beamline focus can be adjusted between 24.65 ± 0.15 m. The pole of M2 is located 15.8 m from the source. This mirror is in a "bounce up" configuration and returns the beam parallel to the plane of the ring.

Channel-Cut Monochromator

The geometry of the channel-cut crystal accepts the entire 4.7 mm high incident beam over the energy range from 7 to 17.5 KeV. The monochromator crystal has a flexure that allows the second surface of the crystal to be slightly de-tuned from the first. This provides a mechanism for rejecting higher order harmonics, which is important when working at low energies. At high energy, harmonic wavelengths are removed by the reflectivity cutoff of mirror M1. The small crystal gap (6.5 mm) minimizes the change in beam height upon changing the wavelengths. This is particularly important within a MAD data set where we would like to minimize the need to adjust mirror heights or realign the endstation. For example, a 200 eV change in wavelength at the Se K edge would only change the beam height by 5 microns. Changing between the extremes in the wavelength spectrum would result in a beam height change of 450 μm, which would necessitate changing the height of the second mirror and the endstation. The first crystal reflects the beam up and the second crystal brings the beam parallel to the incident beam but 12.5-12.9 mm higher. The first crystal surface of the monochromator is indirectly cooled via a water-cooled block. Cooling the crystal in this manner is to reduce thermal deformation of the crystal and help to maintain the energy resolution needed for MAD phasing.

Endstation design

We are currently finalizing the endstation design. The endstation will have a large format CCD based detector. The detector will have readout times of 1-4 sec. The goniostat will feature a 2θ offset, and will be mounted on a motorized kinematic experimental table will be with apertures and diagnostics necessary for aligning the system. There will be a fluorescence detector and cyrocolling system typical of modern MAD protein crystallography beamlines.

Manufacture and Installation

The first shipment (November 2000) comprises the collimating mirror system and components through the first screen (Fig. 4). This allows us to commission the first mirror while the remaining items are still in manufacture. The remaining beamline components will be installed in January 2001. The detector and endstation components are expected in Feburary 2001. The beamline delivery and commissioning timetable calls for full 24/7 user operations by October 2001.

ACKNOWLEDGMENTS

We would like to thank Dr. D. Peter Siddons for helpful discussions. Funding for the beamline is provided by the National Science Foundation through the biological infrastructure award DBI-9871464 with interagency matching funds from the NIGMS at the National Institutes of Health and the GCPCC member institutions.

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