Optical Performance of the GM/CA-CAT Canted Undulator Beamlines for Protein Crystallography

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Abstract. A new macromolecular crystallographic facility developed by GM/CA-CAT is operational at the Advanced Photon Source (APS). The facility consists of three beamlines: two lines based on the first "hard" dual canted undulators and one bending magnet beamline. The ID lines are operational, and the BM line is being commissioned. Both insertion device (ID) beamlines are independently tunable over a wide energy range. The inboard ID lines have been upgraded with a new insertion device to provide enhanced performance for MAD phasing experiments near the selenium and bromine K-edges. The ID line monochromators' crystals are indirectly, cryogenically cooled for improved performance and reliability. Focusing is achieved by long bimorph mirrors in a Kirkpatrick-Baez geometry. This paper describes the design of the beam lines and the optical characterization of the mirrors and monochromators.

Keywords: monochromator, bimorph, mirror, crystallography
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INTRODUCTION

GM/CA-CAT was formed by the National Institute of General Medical Sciences and the National Cancer Institute (member institutes of the National Institutes of Health) to construct and operate a user facility for macromolecular crystallography at the Advanced Photon Source (Argonne National Laboratory). The facility (sector 23) was designed and built in collaboration with ACCEL Instruments, GmbH, of Germany and consists of the first two fully independent, tunable canted undulator beamlines and a bending magnet (BM) beamline [1]. The canted design has the potential to double the scientific throughput of synchrotron facilities. Furthermore, the sector has been planned with the needs of cutting edge research in mind. The beamlines are suitable for diverse experiments on crystals with small dimensions, large unit cells, or those that diffract weakly, such as membrane proteins. In addition, a high level of automation is being incorporated into the lines to provide a robust, easy-to-use facility for increased efficiency and high throughput. The ID lines are operational and accepting users and the BM line is being commissioned. Developmental milestones of the project to date are summarized in Table 1.

TABLE 1. GM/CA-CAT Beamline Timeline.

Dual white light delivered to the first optics enclosure	August 11, 2003
Monochromatic light on ID-in delivered to ID-D	June 14, 2004
First diffraction experiments on ID-in	July 8, 2004
Monochromatic light on ID-out delivered to ID-B	February 16, 2005
First diffraction experiments on ID-out	December 21, 2005
Monochromatic light to BM-B	February 23, 2006

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The straight sections at the APS are 5 m long, and typically only one 2.4 m long insertion device is installed. The canted undulator geometry places two 2.07 m long undulators in tandem. Electromagnetic dipoles and radio frequency beam position monitors (RF-BPMs) are located before, between, and after the insertion devices. As the particle beam traverses the straight section, it is deflected outboard by 0.5 mrad and passes through the upstream undulator producing X-rays for the outboard beamline (ID-out). The central magnet deflects the beam inboard by 1.0 mrad. It then passes through the downstream undulator producing X-rays for the inboard beamline (ID-in). Finally, the third magnet deflects the beam outboard by 0.5 mrad returning it to the original trajectory. This geometry results in a 1.0 mrad separation between the inboard and outboard X-ray beams. Independent steering and insertion device gap selection were important design criteria. This is ensured by incorporating the RF-BPMs into the slow and fast feedback systems of the storage ring.

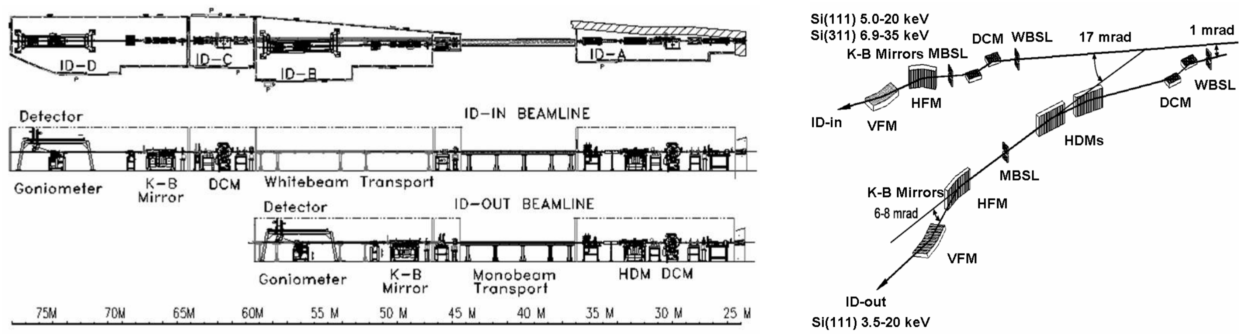

FIGURE 1. GM/CA Sector Layout and Optical Schematic

The layout of the ID beamlines is shown in Fig. 1 (see also Table 2). In the first optics enclosure (ID-A), the beams are initially too close to have separate vacuum chambers so the inboard beam passes through the optical components of the outboard line unperturbed. The first optical components of ID-in are in the ID-C enclosure and include a set of white beam slits (WBSL) followed by a constant exit height, double crystal monochromator (DCM). A set of monochromatic beam slits (MBSL) in the experimental end station (ID-D) defines the beam before the horizontal focusing mirror (HFM) and the vertical focusing mirror (VFM). These “bimorph” mirrors are arranged in a Kirkpatrick-Baez geometry. The design of the experimental endstations will not be described here.

TABLE 2. Component positions relative to the center of the straight section (m)

	WBSL	DCM	IBST	HDM1	HDM2	IBSH	MBSH	MBSL	HFM	VFM	Sample
ID-in	60.313	61.533	62.574			34.650	63.495	64.569	65.800	66.745	72.000
ID-out	27.314	28.900	29.980	31.310	31.970		45.675	46.749	48.000	48.945	54.200

Although the components of ID-out are very similar to those of ID-in, there are a few significant differences. The masks of the WBSLs and integral white beam stop (IBST) have an extra beam path for the inboard beam. The WBSL and DCM are located much further upstream; therefore, the total power and power density are increased on the optics. A pair of horizontal deflecting mirrors (HDMs) sequentially intercepts the outboard beam and deflect it outboard an additional 16 mrad. The energy ranges of the beamlines are indicated in Fig. 1. The lower maximum energy on ID-out results from a compromise between a large separation of the two lines and the high energy reflectivity cut-off of the mirrors. The slightly larger range at the low energy end is due to the different undulators.

Several other optical schemes considered included large-offset, double-crystal monochromators diffracting in the vertical or horizontal plane; pairs of white-beam mirrors; and pairs of vertical-deflecting monochromatic mirrors. Each of these designs were rejected for various reasons such as poor energy resolution, polarization induced intensity loss, mechanical and thermal stability, and a non-homogeneous beam profile due to large distances from vertical reflecting mirrors. The chosen design does not suffer from these limitations, and provides a separation of approximately 500 mm between the beams. This space can accommodate a modern crystallographic end station with a large mosaic CCD detector.

SOURCE INFORMATION

Most ID lines at the APS use the standard Undulator A (3.3 cm period, 72 poles). However, the intensity of the first harmonic falls off rapidly above 12 keV as indicated in Fig. 2. Thus, for Se-Met MAD the experimenter must decide to collect data at reduced intensity with possibly poorer statistics or to switch to the third harmonic which may induce thermal drift in the monochromator due to the almost ten-fold increase in incident power. To avoid this trade-off, a new 3.0 cm device with 69 poles was developed and subsequently installed in August of 2005 on ID-in. This new undulator provides significant first harmonic intensity up to about 15 keV allowing the use of the low power, first harmonic for the biologically important selenium and bromine K-edges.

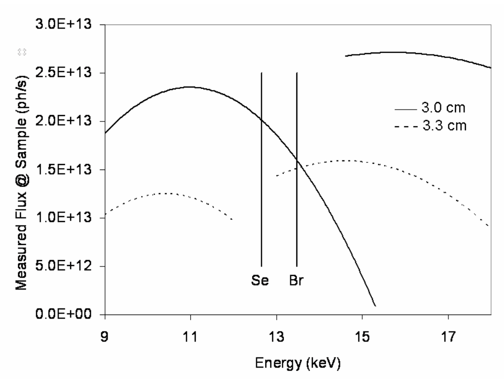

FIGURE 2. Comparison of the 3.3 and 3.0 cm period insertion devices. The Se and Br K-edges are indicated.

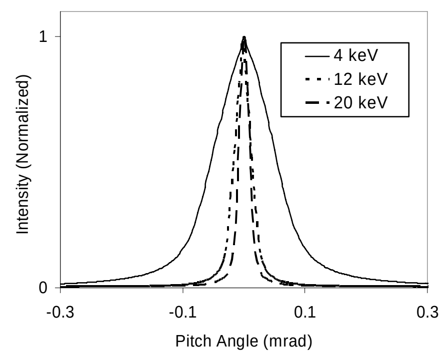

FIGURE 3. Rocking curves at 4, 12 and 20 keV.

OPTICAL COMPONENTS – ID BEAMLINES

The APS currently operates at 100 mA beam current with plans for 200 mA; therefore, the canted undulator front ends [2] and beamlines were designed for 200 mA operation. The first component of each beamline is the white beam-defining slits that are based on the APS L5-92 design [3]. These slits are water-cooled with tapered surfaces to spread the heat load. Temperature probes within 2 mm of the tapered surface indicate temperatures increases less than 5 K under the most extreme conditions.

The monochromator stability is key to the performance of the entire beamline, and a significant effort has gone into both the design and commissioning. The monochromator crystals (Si(111) or (Si(311)) are indirectly cooled by liquid nitrogen. Temperature stabilized shielding around the crystal assemblies capture Compton scattered X-rays providing a high degree of stability under varying thermal loads. The monochromator first crystal has a 1 mm thin web to reduce the amount of power absorbed and Compton scattering produced.

The primary means of evaluating the stability of the monochromator design is to study rocking curves under a variety of conditions. Extreme heat loads may lead to a long-lifetime thermal drift (hours) in the rocking curve centroid, or may broaden the rocking curve. Thermal drift over time does not appear to be problematic. Both monochromators show an initial, rapid equilibration of the in-crystal temperatures in a few minutes. After this, a small long-lifetime drift that does not result in significant beam intensity loss is observed but only under high heat load conditions.

The power limitation of the DCM was evaluated by measuring rocking curve width *vs.* incident power as a function of WBSL opening. The rocking curve width is preserved for incident power less than 600 W. Assuming that the broadening is due to the total power, and that increasing the beam current to 200 mA will double the power through a given aperture, then calculations indicate that approximately 90% of the desired photons can still be delivered by reducing the slit aperture appropriately to limit the power to less than 600 W.

In many double crystal monochromators, scanning the energy results in a loss of intensity as the second crystal detunes relative to the first. At GM/CA, a second crystal counter weight minimizes torque about the rotation axis thereby keeping the crystals aligned. Rocking curves were recorded at 4, 12 and 20 keV with a fixed undulator gap providing first, third, and fifth harmonic radiation. The superposition of the three curves in Fig. 3 demonstrates the success of the design.

Fluctuations in the monochromatic intensity can be indicative of DCM vibrations due to coolant flow or motor movement. EXAFS spectroscopy was used to characterize the noise levels. Figure 4 shows the transmission EXAFS of gold foil recorded about the L3-edge in approximately two minutes. The noise in the incident intensity (I0) was 0.02%, and the noise in the ratio of the transmitted intensity to I0 is 0.004% indicating a high level of stability.

Mechanical inaccuracies or miscalibrations in the motions of the DCM can result in movement of the monochromatic beam as a function of energy. These effects have been minimized through design improvements, extensive characterization and tuning, and were evaluated by varying the energy from 5 to 15 keV (with a constant undulator gap). The measured beam motion at the sample position was < 2 μm in both the horizontal and vertical directions, a figure significantly less then most DCMs.

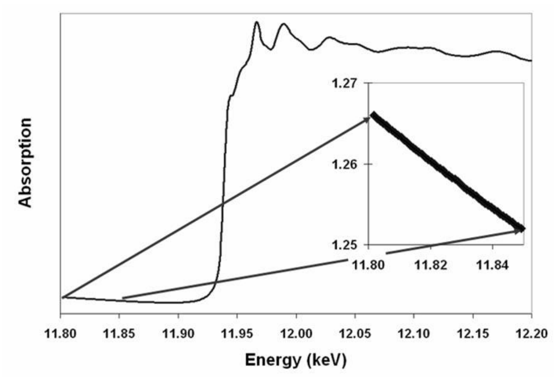

FIGURE 4. Au-L3 EXAFS

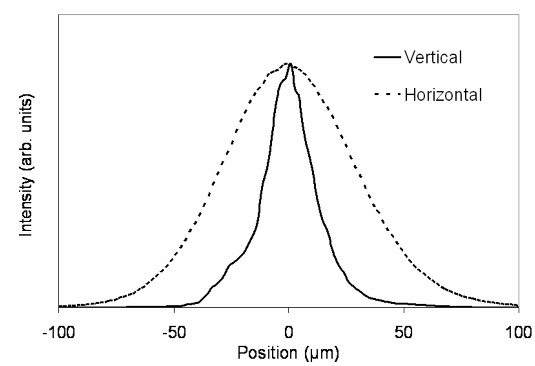

FIGURE 5. Beam profiles at the focal point. The vertical and horizontal beams measured 22.6 and 66.9 μm FWHM, respectively.

The final optical component on both ID beamlines is a pair of bimorph focusing mirrors in a Kirkpatrick-Baez geometry [4,5]. These mirrors also have a choice of surfaces that provide harmonic rejection over an extended energy range. On the first beamline, silica, rhodium, and platinum surfaces are available. The more limited energy range of the second line requires only silica and rhodium surfaces.

The unique advantage of the bimorph functionality is the ability to correct *in situ* the slope error on the mirrors. This allows preservation of the ideal Gaussian beam profile at not only the focal position but also at off-focus positions. Figure 5 shows the smallest, most symmetrical focal profiles that have been achieved to date for both the horizontal and vertical directions as measured with a 10 μm slit scan.

In-vacuum X-ray beam position monitors are located after each optical component and just before the end station goniometery. These provide beam intensity and positional information as well as video monitoring. These BPMs can be used for diagnostics and automated alignment capabilities and have been integrated into feedback systems providing a high degree of stability for macromolecular crystallography experiments.

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