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INSTRUMENTATION FOR TIME-RESOLVED DISPERSIVE STUDIES AT
ADVANCED PHOTON SOURCE BEAMLINE 1-BM*

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INSTRUMENTATION FOR TIME-RESOLVED DISPERSIVE STUDIES AT ADVANCED PHOTON SOURCE BEAMLINE 1-BM

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We describe progress in optics and instrumentation at beamline 1-BM, designed in part for time-resolved dispersive XAFS measurements. The key optical element is a horizontally focusing curved-crystal monochromator that invokes a 4-point bending scheme and a liquid-metal cooling bath. The device has been designed for dispersive studies in the 5-24 keV range, with a horizontal focal spot size of $\leq 100 \mu\text{m}$ FWHM. To minimize thermal distortions and thermal equilibration time, the 355 x 32 x 0.8 mm crystal is nearly half submerged in a bath of Ga-In-Sn-Zn alloy, which thermally couples the crystal to the water-cooled Cu frame, while permitting the required crystal bending. Harmonic rejection, focusing schemes and the novel spectrometer positioning system will be described. For microsecond-resolution time-resolved studies, a fast CCD streak camera detector has been developed. Results from commissioning tests of the instrumentation are described.

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INTRODUCTION

This paper is principally a status report on the instrumentation for time-resolved dispersive studies which has recently been installed at the Advanced Photon Source (APS) beamline 1-BM. The design of the liquid-metal-cooled curved-crystal monochromator for this station was described in a recent publication [1]. The curved-crystal optic is used to horizontally focus a portion of the light from the bending magnet source, thereby forming a beam in which photon energy varies linearly with angle. Such an energy dispersed beam will be used to perform absorption spectroscopy measurements, such as various x-ray absorption fine structure (XAFS) techniques [2-5], in addition to novel diffraction measurements including anomalous scattering [6], diffraction anomalous fine structure (DAFS) [7] and continuous energy diffraction spectroscopy (CEDS) [8]. In general, dispersive geometries benefit from the ability to employ a position sensitive detector (PSD) to collect data over a range of energies or wave vectors in parallel. These geometries are therefore particularly amenable to time-resolved studies, where it is desired to make rapid structural measurements *in situ* during structural transformations.

We now highlight key aspects of the instrumentation for dispersive studies[9], which is depicted in Figure 1. The bending magnet source has a measured horizontal size of $A = 350 \pm 25 \mu\text{m}$ FWHM [10]. A 1.2 m long vertical focusing mirror (24 keV fixed cutoff) and a 0.55 m in-station flat harmonic-rejection mirror will be used. (The double crystal monochromator delivers beam to a downstream station, and is generally withdrawn from the beam for experiments in 1-BM-B). The 35 cm long Si(220) dispersive monochromator crystal accepts approximately 3 horizontal mrad from the source and focuses the energy dispersed beam to a focal spot of approximately 100 microns horizontal FWHM for energies in the range of 5-16 keV. Higher energies may be reached using higher order reflections and appropriate absorbers. The energy bandwidth is selectable up to approximately $\Delta E/E = 10\%$. The high-precision 4-point crystal bender includes an active de-twisting mechanism and a liquid-metal bath for high cooling power and fast thermal equilibration of both crystal and bender. A simple coarse-positioning system, based on removable mechanical tethers, allows the optics, spectrometer and detectors downstream of the monochromator to be quickly located at the desired radius and 2θ angle with respect to the monochromator crystal, with a resolution of approximately 1 mm. Motorized fine positioning permits energy scanning over a range of more than 1 keV. A high-speed CCD camera coupled to a precision phosphor can be operated in image mode with 10 ms exposures, or as a streak camera for 60 μs exposures. Each of the in-station instruments will be discussed in turn, giving results from our recent commissioning experiments.

PROGRESS REPORT

We first describe the status of the liquid-metal-cooled curved-crystal monochromator which is shown schematically in Figure 2. Following assembly, installation and alignment, the bender mechanism was filled with liquid Ga-In-Sn-Zn alloy. The motions and focusing capabilities were then tested at an energy of 8.979 keV (the Cu K edge). A temporary quartz harmonic rejection mirror was installed for these purposes. The focal spot size was measured using a phosphor coupled CCD detector (described below) in imaging mode. This was found particularly helpful for adjusting the crystal antitwist to give a clean vertical stripe at the focal plane. The horizontal FWHM of the focal spot changed measurably when adjustments as small as 0.3 microns were applied to the ends of the crystal, such that the 0.1 micron resolution of the bender drives was well utilized. The smallest focus obtained in our commissioning was 150 ± 20 microns FWHM, as shown in Figure 3. After approximately 15 minutes of thermal equilibration, the monochromator focus was found to remain stable over a period of several hours.

The absolute flux at the focus was measured using an N₂ filled ion chamber. With horizontal and vertical acceptance set by upstream beamline slits to be 1.74 mrad and 38 μ rad respectively, the flux was 3.4×10^{10} photons/second at 100 mA. The maximum possible flux, calculated from the source brilliance, assuming perfect dynamical diffraction, and accounting for attenuation losses and polarization factors, is approximately 1.7 times our measured number. We attribute the difference to non-perfect diffraction within the curved crystal.

When the CCD detector was moved downstream to view the dispersed beam behind the focal spot, interesting intensity variations, of approximately 5% relative intensity, were observed across the width of the beam. We now understand these to be due to visible thickness variations in the crystal resulting from gridding during fabrication. We will eliminate this effect by polishing the crystal. In addition to providing a smooth intensity distribution, this may also permit a smaller horizontal focal spot, closer to the design width of <100 microns FWHM. Despite these intensity variations, we were able to make rapid measurements of the Cu K edge absorption spectrum. Figure 4 shows our first near edge structure measurement, obtained using the CCD camera in image mode. The image exposure time was 7 ms and 10 CCD rows have been averaged to produce the plot. The energy resolution is approximately 2 eV.

The in-station harmonic rejection mirror and monochromator exit slits are still in construction, but the experimental station is otherwise complete. So that they may all be re-positioned at once, the

mirror, flight path, sample cell and detectors will all be affixed to the same table as the 4-circle goniometer, which will in turn be centered on the focal spot. The table motorized motions permit the whole apparatus to be rotated on an arc about the monochromator crystal for small displacements corresponding to approximately 1 keV energy shift. Coarse positioning of the table inside experimental station is achieved by lifting the apparatus on its air pads and manually moving it until 3 mechanical tethers between the table and the station perimeter are drawn taught. The length of the removable tethers is calculated to position the center of the goniometer at the desired radius and 2Θ angle from the monochromator crystal, with the harmonic rejection mirror and flight path axis aligned to the exit beam. The tether anchor points and the crystal rotation axis were surveyed in place using a laser tracking system with 100 μm resolution. There is sufficient choice of anchor points that the spectrometer may be repositioned at radii ranging from 0.75 to 4 m and 2Θ angles ranging from 0 to 90° , without interfering with other instrumentation or obstacles in the station. We have demonstrated that this coarse alignment system can effectively position the apparatus to approximately 1 mm resolution, without the encumbrances of permanent rails or large rotation stages.

A VXI based high-speed Charge Coupled Device (CCD) was designed for our time-resolved studies. A detailed description of the camera and its characteristics are described elsewhere [11]. The camera is based on a 512x512 pixel CCD manufactured by Thomson CSF, with 19 μm square pixels. The CCD has a 1:1 fiber-optic face plate bonded to it, which couples the CCD to a 60 μm thick layer of GdOS₂:Tb for x-ray to visible light conversion. Conversion within the phosphor limits the camera resolution to 80 μm . The CCD is read out from two parallel outputs at a maximum pixel clock speed of 15 MHz/output that gives a frame rate of 100 frames/second. Alternatively the device can be programmed as a streak camera with a limited number of rows being read out continuously. The maximum speed attainable in this mode is 40 μsecs per row. The data for both these modes is stored in a custom VXI memory module with 512 MB of DRAM storage. In full frame mode, the memory allows us to store 936 frames before the data must be transferred to disk. In streak mode, the maximum number of linear data images is 479232. The camera is controlled by a custom designed event driven graphical user interface running under Windows 95 on a Pentium PC.

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FIGURES

FIG. 1: Schematic layout of the first optics enclosure (1-BM-A) and the time-resolved station (1-BM-B) at APS beamline 1-BM.

FIG. 2: Schematic representation of the 4-point bending scheme. The positions at which the crystal is constrained are labeled A,B,C, and D. Bending is achieved by pushing on points A and D, while de-twisting is achieved by rotating the clamp at C as indicated.

FIG. 3: Observed focal spot for a dispersed beam with a mean energy of 8.979 keV. The data is a cross section from a CCD image acquired in 20 ms. The measured FWHM of 175 μm shown in (a) is significantly broadened by the camera resolution, which we measured to be 80 μm , by partially blocking the unfocussed beam with a knife edge and differentiating the knife edge scan

(b). When deconvolved from the focal spot measurement, we deduce an actual focal spot size of $150 \pm 20 \mu\text{m}$ FWHM.

FIG. 4: Our first dispersive absorption data, showing the Cu K near-edge structure. The data is an average of 10 rows acquired using the CCD detector in image mode with a 7 ms exposure time.

Fig. 1

Fig. 2

Fig. 3

Fig. 4