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EXPERIMENTAL RESULTS WITH CRYOGENICALLY COOLED, THIN, SILICON CRYSTAL X-RAY MONOCHROMATORS ON HIGH-HEAT-FLUX BEAMLINES*

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Experimental results with cryogenically cooled, thin, silicon crystal x-ray monochromators on high-heat-flux beamlines

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ABSTRACT

A novel, silicon crystal monochromator has been designed and tested for use on undulator and focused wiggler beamlines at third-generation synchrotron sources. The crystal utilizes a thin, partially transmitting diffracting element fabricated within a liquid-nitrogen cooled, monolithic block of silicon. This report summarizes the results from performance tests conducted at the European Synchrotron Radiation Facility (ESRF) using a focused wiggler beam and at the Advanced Photon Source (APS) on an undulator beamline. These experiments indicate that a cryogenic crystal can handle the very high power and power density x-ray beams of modern synchrotrons with sub-arcsec thermal broadening of the rocking curve. The peak power density absorbed on the surface of the crystal at the ESRF exceeded 90 W/mm^2 with an absorbed power of 166 W, this takes into account the spreading of the beam due to the Bragg angle of 11.4°. At the APS, the peak heat flux incident on the crystal was 1.5 W/mA/mm^2 with a power of 6.1 W/mA for a 2.0 H x 2.5 V mm^2 beam at an undulator gap of 11.1 mm and stored current up to 96 mA.

Keywords: high-heat-flux optics, cryogenic monochromator, synchrotron radiation, undulator

1. INTRODUCTION

The Advanced Photon Source (APS) at Argonne National Laboratory (ANL) is a dedicated third-generation, hard x-ray synchrotron source.[1] The combination of the small source size and divergence of the storage ring particle beam and the use of insertion devices (IDs) is the hallmark of the APS and other modern facilities, which produce highly collimated x-ray beams of unparalleled spectral brilliance used for research across many scientific and technology disciplines. Nearly all of the ID beamlines at the APS have installed a 2.5-m-long, 3.3-cm-period undulator.[2] The first optical element on most beamlines is a nondispersive double-crystal monochromator (DCM) used to select the wavelength and bandwidth from the incident white beam to be delivered to the experimental station.[3] One of the most technically challenging aspects for beamline optics design is thermal management. The peak heat flux emitted by an undulator is proportional to the peak magnetic field strength, storage ring current, number of magnetic periods, and the fourth-power of the storage ring energy (7 GeV at the APS).[4] The power density of the x-ray beam emitted by the APS undulator can exceed

160 W/mm^2 at normal incidence on the DCM, typically located about 30 m from the source. Often the limiting factor for full utilization of the x-ray beam is the heat load that the beamline optics can handle and still preserve the emittance (phase space volume) and spectral brilliance (phase space density) of the source. Future capabilities of the APS include using 5-m-long IDs and/or increasing the storage ring current from 100 to 300 mA. This can result in a six-fold increase in the beam power; therefore, thermal management of optical components will continue to be a limiting factor in delivering the highest quality beams to the experimenter.

The performance of the monochromator is highly sensitive to thermo-mechanical strain. Absorption of x-ray energy in the first crystal of a DCM causes thermal gradients resulting in strain of the atomic planes. In order for the x-ray beam to be doubly diffracted, the atomic planes of the two crystals must be aligned to within a fraction of the Darwin reflection width. The Darwin width is the angular acceptance of the monochromator crystal, typically several arcsec for silicon. If the first crystal (or the second) is strained, its reflection curve is broadened reducing the theoretically deliverable brilliance. The goal for thermal design of monochromators is to minimize the thermal deformation under the beam footprint. Strictly speaking, one wants to minimize the distortion under that part of the beam footprint that contains the photons that are being diffracted. For bending magnet and wiggler radiation, the spectral distribution is relatively uniformly spread out over the entire beam, whereas the harmonic radiation from an undulator is confined within a narrow central cone in the middle of the power envelope. Typically, the unwanted photons outside of the central cone are removed by the use of an aperture upstream of the monochromator eliminating much of the power incident on the optic. The subjects of high-heat-flux optics and other beamline components have received extensive attention over the past decade resulting in several dedicated sessions at international conferences, including the present one.[5-7] A review article and a comprehensive special journal section on cooled optics have also been recently published.[8,9]

Thermal gradients in the monochromator crystal cause three basic forms of distortion: irradiance mapping resulting in a "thermal bump", overall bowing, and lattice spacing gradients. Usually the mapping error causes the most severe degradation of performance. The thermal mapping error is inversely proportional to the ratio of the thermal conductivity, k, and the coefficient of thermal expansion, α, of the monochromator material. The relative performance of a monochromator material can be quantified by this figure-of-merit (FOM), k/α. The FOM for silicon is greatly improved at cryogenic temperatures. The thermal conductivity increases by nearly an order of magnitude at 80 K compared to room temperature, and the expansion coefficient decreases, passing through zero at 125 K and remaining slightly negative. Rehn proposed exploiting this property of silicon, and other single crystals, for x-ray optics in 1986.[10] A number of papers have since been published on the use of cryogenically cooled monochromator crystals.[11-20]

This paper summarizes the experimental results of several tests of a novel, silicon monochromator crystal cooled with liquid nitrogen and incorporating a thin diffraction element. The thin element is partially transmitting to the high energy part of the white beam, thereby reducing the absorbed power. Two separate tests of this monochromator were performed at the ESRF on a high-heat-flux focused wiggler beamline.[18,19] Recently, the monochromator crystal was tested on the sector 1 undulator beamline at the APS.[20] The APS is composed of 35 repeating sectors each comprising a bending magnet and an ID beamline. Based upon these very encouraging results, most of the collaborative access teams (CATs), groups of researchers from various institutions who develop and operate a beamline(s) at the APS, have implemented a cryogenically cooled monochromator. At present, there are 20 sectors in development. All of the ID beamlines except one are installing an undulator as the primary x-ray source. Of these, 15 have so far opted for using liquid-nitrogen-cooled monochromators with several others yet to decide. The other ID beamline is equipped with an elliptical multipole wiggler and will also use a cryogenic monochromator.[21]

2. EXPERIMENTAL RESULTS

The two crystals tested are shown in Fig. 1. The first crystal (top) was tested on beamline BL3 at the ESRF in November 1994. The second similar crystal (bottom) was tested again at the ESRF in June 1995 and then in May 1996 on an undulator beamline at the APS. The crystals each consist of a monolithic block of (111)-oriented silicon in which a thin diffracting element has been machined into the center of the block by milling out slots in the top and bottom faces leaving an approximately 0.5 to 0.7 mm thick region in the center of the crystal. A slot is also milled in the downstream face to allow the transmitted beam to pass through. The x-ray beam is incident in the center of the slot on the top surface and the transmitted beam passes through a slot fabricated in the downstream coolant manifold. A pattern of seven, 1/8-inch-diameter holes is core-drilled on either side of the diffraction region through which liquid nitrogen is pumped. The seal between the crystal and the Invar coolant manifold is made via indium-coated metal C-rings.[22] The seals have been thermally cycled several times with liquid nitrogen at pressures up to 150 psi with no evidence of leaking when checked with a He mass spectrometer to a sensitivity of 10^{-10} mbar l/s. The silicon and manifold seal surfaces are highly polished to minimize the sealing force and the mechanical stress imparted to the optic. The average surface roughness was 2.4 nm RMS measured with a *Wyko Topo-3D* interferometer at a wavelength of 0.63 µm, and the flatness over the sealing surface was within 0.35 µm measured with a *Wyko 6000* surface profile interferometer at a wavelength of 0.63 µm. The strain relief cuts shown on either side of the diffraction slot were added after the second ESRF experiment and prior to the APS tests. This helped to reduce the propagation of strain from the liquid seals to the thin element. Strain relief cuts will be added along the top and bottom faces of the crystal in the future to further mechanically isolate the thin element from the stress of the sealing gasket while leaving enough material for thermal conduction. The bottom photograph also shows the kinematic/isolation mounting plate that allows for thermal expansion of the crystal mount while maintaining the position of the thin element relative to the x-ray beam.

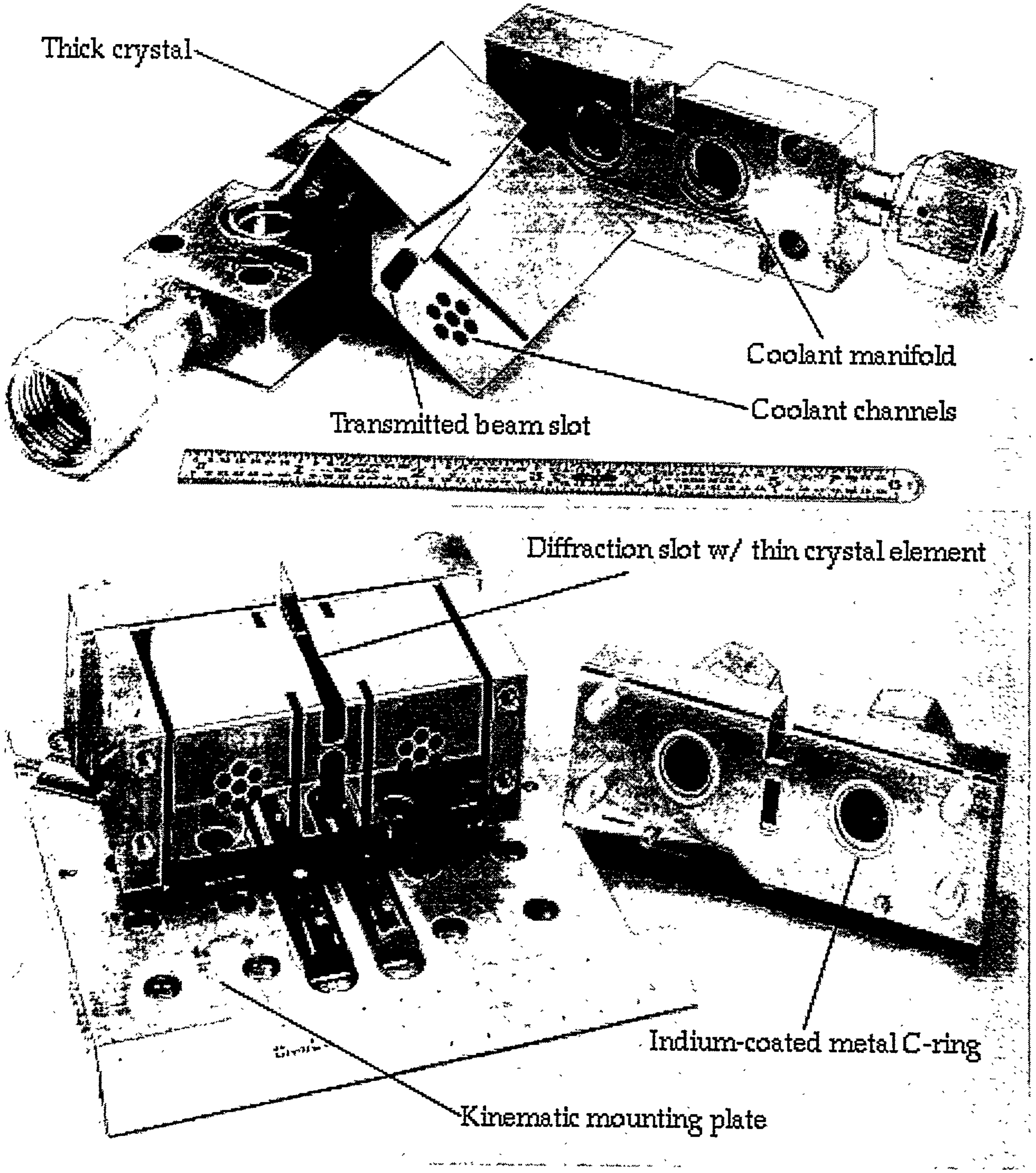

Fig. 1 Photograph of the crystals and coolant manifolds tested on beamline BL3 at the ESRF in November 1994 (top) and tested in June 1995 at the ESRF and in May 1996 (bottom) on an undulator beamline at the APS. Visible in both photographs are the downstream faces, showing the slot that allows the transmitted beam to pass through the crystal, and the coolant channels on either side of the diffraction element.

2.1 ESRF Results

The high-heat-flux tests were performed on beamline BL3 at the ESRF.[23] A Pt-coated, toroidal mirror located 31.3 m from the source was used to concentrate the emitted power from a 44 pole wiggler to a small spot size. The beam emitted from the wiggler passed through a 0.26 mm C (graphite) prefilter and a 0.5 mm Be window. A variable thickness graphite filter was used to adjust the incident power on the mirror. The focused beam measured 0.19 mm (FWHM) vertically and 1.8 mm horizontally on the cooled crystal positioned at 46 m. A variable thickness Al filter downstream from the mirror was used to vary the heat load on the monochromator. The mirror cutoff energy was 29 keV. Therefore, the spectral power envelope incident on the crystal ranged from about 5 to 30 keV; consequently, most of the incident power was absorbed.

Rocking curves were recorded for a fixed energy of 30 keV from the (333) planes as a function of incident power. The thickness of the thin diffraction element of the crystal in the first test was about 0.6 mm, and the thickness for the second crystal tested was about 0.7 mm. Data were also collected from a thick region of the second crystal. Fig. 2 shows the rocking curve widths (FWHM) as a function of absorbed peak power density on the surface of the crystal at a Bragg angle of 11.4°.

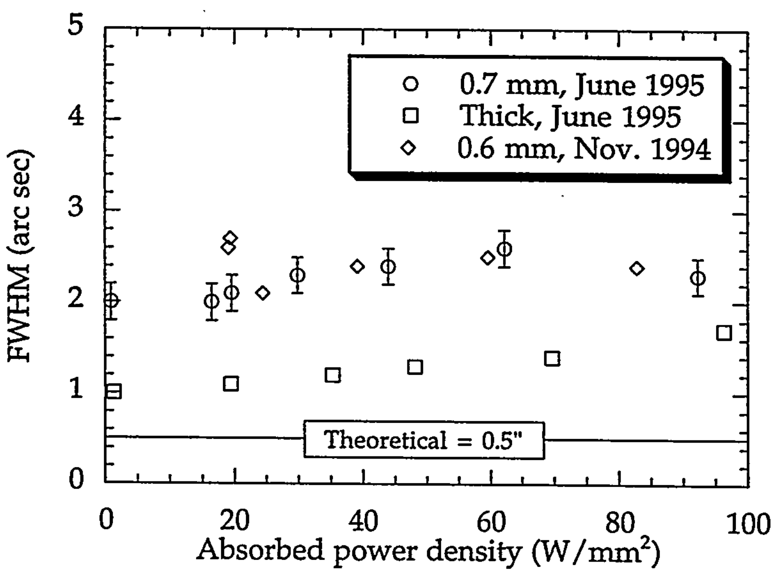

Fig. 2 Rocking curve width (FWHM) for the (333) reflection at 30 keV (Bragg angle = 11.4°) as a function of absorbed power density on the surface for the thin and thick parts of the 0.7-mm-thick cryogenic crystal and for the thin part of a similar 0.6-mm-thick crystal. The theoretical width is 0.5 arcsec.

The response of the thin section was essentially identical between the two thin crystals. The low power rocking curves show a residual strain of about 1.5 arcsec and sub-arcsec thermal broadening up to an absorbed surface power density of 95 W/mm^2. The residual mechanical strain in the thick part was significantly less, only about 0.5 arcsec. The thick crystal also exhibited sub-arcsec thermal broadening up to nearly 100 W/mm^2 with an incident power of 186 W.

2.2 APS Results

Recently, the performance of the same crystal used in the June 1995 ESRF tests was evaluated using the APS undulator on the Synchrotron Radiation Instrumentation Collaborative Access Team (SRI-CAT) sector 1 ID beamline.[24] The x-ray beam emitted from the undulator passed through a temporary commissioning window at 23.5 m consisting of 0.50 mm of graphite, 0.17 mm of CVD diamond, and 0.50 mm of Be. The commissioning window absorbed approximately 12 % of the beam power for a 2.0 H x 2.5 V mm^2 cross-section beam at an undulator gap of 11.1 mm. A set of horizontal and vertical slits at 26.75 m were used to aperture the white beam which was then incident on the monochromator located at 28.5 m. A set of two ionization chambers, I_0 and I_1, was placed at 34 m.

Rocking curves were recorded from both the thin part of the crystal and from a thick part. Fig. 3 shows the rocking curve widths (FWHM) as a function of photon energy. Rocking curves from the (111) and (333) planes were recorded simultaneously by placing 0.09 inches of Al between the two ionization chambers used to monitor the diffracted beam. Data were recorded for a 2.0 H x 2.5 V mm^2 beam on the thin part of the crystal and for a 3.0 H x 1.9 V mm^2 beam on the thick part. The undulator gap was kept fixed at 11.1 mm corresponding to a deflection parameter, K, of 2.57. The storage ring current for the thin crystal data ranged from 61 to 96 mA with a measured beam power of 6.10 W/mA, and the data for the thick part were collected from 89 to 95 mA at a power of 5.51 W/mA. The beam power was measured by recording the time rate of change of the temperature of a Cu block placed in the beam The measured and calculated power correspond to within a few percent indicating that the beam was centered in the slit aperture so that the hottest part of the beam was striking the crystal. The power transmitted through the 0.7-mm-thick crystal was measured for an undulator gap of 11.5 mm at a Bragg angle of 19.24°. For a 2.5-mm-square beam, 52 % of the incident power was transmitted. This value is within 5 % of calculations. These data were collected under much higher heat loads than would normally be the case because the undulator gap was kept at 11.1 mm , 3.27 keV first harmonic, to maximize the power for all of the rocking curves rather than being opened as the energy was increased to track the harmonic. As the gap is opened, the emitted power rapidly decreases. For example, at a first harmonic energy of 8 keV, corresponding to a gap of about 18.3 mm, the power and peak power density incident on the thin crystal are reduced to only about 40 percent of that for an 11.1 mm gap. Rocking curves for the (333) reflection at 15 keV are shown in Fig. 4 for an undulator gap of 11.1 mm for the thin and thick part of the crystal. The power incident on the thin crystal was 506 W at 83 mA and 490 W at 89 mA for the thick crystal. The theoretical width is 1.1 arcsec.

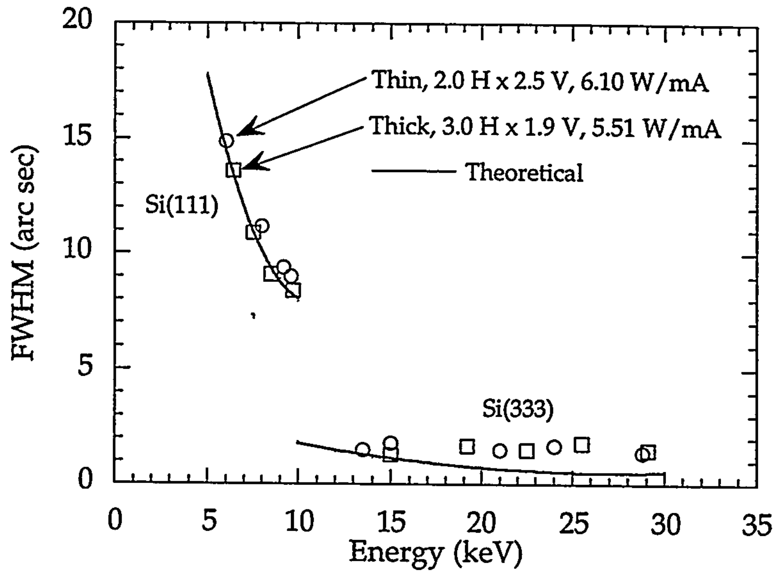

Fig. 3 Rocking curve width (FWHM) for the first and third order reflections from the thin and thick parts of the crystal as a function of photon energy for a fixed undulator gap of 11.1 mm. The beam cross section incident on the thin part of the crystal was 2.0 H x 2.5 V mm^2 with a measured power of 6.10 W/mA, and the beam incident on the thick part measured 3.0 H x 1.9 V mm^2 with a measured power of 5.51 W/mA.

Both the thin and thick parts of the crystal performed about equally well thermally. The thin crystal did exhibit slightly more residual mechanical strain, about 0.5 arcsec, due to the stress at the seal interface and residual fabrication stress. It was noticed during the course of the experiments that the thin crystal surface in the bottom of the slot possesses a curvature in the axis normal to the diffraction plane. This unintended result occurred because of a differential etching rate between the center of the slot and the corners during fabrication. The lateral edges are about 0.6 mm higher than the center. This turns out to be somewhat beneficial from a thermal point of view because less power is absorbed from the central, hottest part of the beam while more material is available at the edges of the thin element to better conduct heat to the coolant channels compared to a completely planar thin element.[25] However, this curvature can also cause spatial aberrations of the diffracted beam due to the unequal path lengths between the center and edge of the beam. The asymmetry in the thin crystal rocking curve is due largely to the curvature of the diffracting surface and some misalignment about the reciprocal lattice vector. The rocking curve broadening is due to a combination of mechanical strain from the coolant manifold, forced vibration from the coolant hoses, and thermal strain. The total amounts to about 1 arcsec. The flow-induced vibration was only evident at very high flow rates and amounted to only a few tenths of an arcsec.

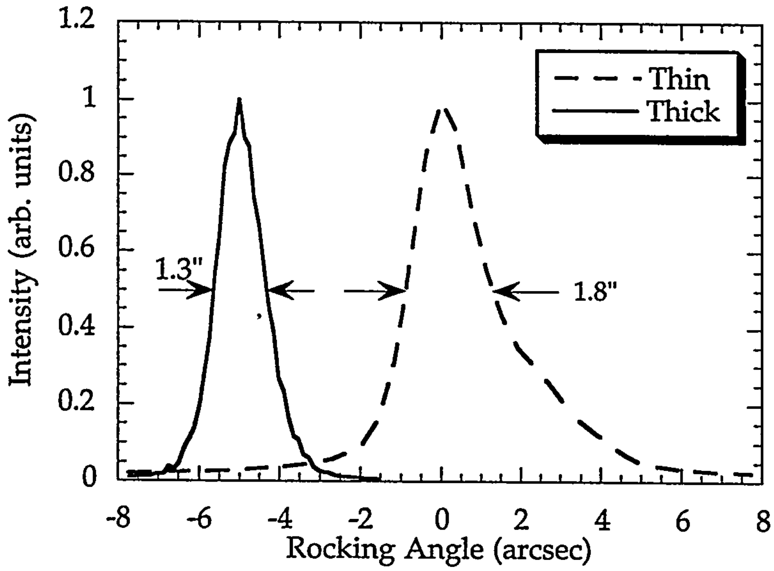

Fig. 4 Normalized (333) rocking curves from the thin and thick parts of the crystal at 15 keV showing the FWHM widths. The beam cross section for the thin crystal was 2.0 H x 2.5 V mm^2, and the beam size for the thick crystal was 3.0 H x 1.9 V mm^2. The angular separation of the rocking curves is for the purpose of clarity only.

3. SUMMARY

It has been demonstrated experimentally that the thin, cryogenically cooled silicon monochromator can handle the worst-case power load from the APS undulator, and other high-heat-flux sources, with minimal thermal strain. Equally important is that the mechanical strain due to the sealing stress and flow-induced vibration has been reduced to the sub-arcsec level, which is very difficult to achieve even in room temperature crystals. The thick crystal also performed very well, even exceeding the performance of the thin crystal, due to the lower mechanical strain in the thick part. It is believed that the mechanical strain in the thin region can be further reduced by adding additional strain relief cuts between the seal faces and the thin element. One of the important benefits of the thin crystal is that it absorbs less power and consumes less liquid nitrogen. The thin crystal absorbed about 50 percent of the incident power from a 2.5 x 2.5 mm^2 beam at a gap of 11.5 mm and a Bragg angle of 19.24°. Continued tests will determine how thin the crystal can be made and still perform acceptably, especially as the x-ray power is increased in the coming months when the commissioning window is removed and the beam current is increased. It is desired to reduce the absorbed power as much as possible. The SRI-CAT sector 1 ID beamline will be installing a nitrogen gas reliquefier with a cooling capacity of 360 W at 77 K. The reliquefier will collect the spent gas from the liquid nitrogen pump heat exchanger, liquefy it, and return it to the pumping system;

thus, making a completely closed-loop cooling system eliminating the need for frequent dewar refills and operator intervention.

This brings to a successful conclusion, at least for the near term, a decade long endeavor to find a suitable solution to the high-heat-flux optics challenge at the APS. Many possible solutions have been proposed and tested by numerous researchers in Europe, the United States, and Japan, all with varying degrees of success and applicability. The implementation of the cryogenic solution at the APS has benefited greatly from this collection of work.

4. ACKNOWLEDGEMENTS

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