ANL/XFD/CP-93339

CONF-9706157--1

CRYOGENIC HIGH-HEAT-LOAD OPTICS AT THE ADVANCED PHOTON SOURCE*

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June 1997

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INVITED PAPER to be presented at the 10th U.S. National Conference on Synchrotron Radiation Instrumentation, SRI'97, Ithaca, NY, June 17-20, 1997.

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Cryogenic high-heat-load optics at the Advanced Photon Source (Invited)

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Cryogenically cooled silicon monochromators have found wide application at the Advanced Photon Source (APS) and other third-generation synchrotron radiation facilities. Currently, 17 insertion device beamlines at the APS are implementing cryogenic, silicon double-crystal monochromators (DCM) as the first optical element. Recently, several silicon crystal monochromators internally cooled with liquid nitrogen have been tested on the sector 1-ID undulator beamline at the APS. Rocking curves at various energies were measured simultaneously in first and third order from a Si(111) DCM in the Bragg reflection geometry at a fixed undulator gap of 11.1 mm. The crystal exhibited sub-arc second thermal broadening of the rocking curve over a first order energy range from 6.0 to 17.0 keV up to a maximum incident power of 561 W in a 2.5 V x 2.0 H mm^2 beam. It has been demonstrated that cryogenic silicon monochromators can handle the highest power beams from hard x-ray undulators at the APS without significant thermo-mechanical distortion.

I. INTRODUCTION

Great strides have been made over the last several years in developing high-heat-load optics for insertion device beamlines at the Advanced Photon Source (APS). A review of the high-heat-load monochromator development program at the APS is given in Ref. 1. Since commissioning began in early 1996, all of the operational elements necessary for cryogenically cooled optics have been installed and tested on beamline 1-ID. These elements include an internally cooled first crystal, cryogenically compatible monochromator mechanism, and a continuous liquid nitrogen pumping system. Also, a nitrogen gas liquefier has been installed on sector 1. The liquefier collects the boiled-off gas from the pumping system heat exchanger, liquefies it, and returns it to the heat exchanger bath, thus making a completely closed-loop cooling system. Of the 20 sectors currently under development at the APS, 17 have so far opted for using cryogenically cooled silicon monochromators on their insertion device beamlines. The remaining beamlines are employing a water-cooled mirror as the first optical element and/or diamond monochromators.[2] The incidence angle for total external reflection mirrors is very small, typically less than 0.2°. This greatly reduces the heat flux projected onto the mirror allowing it to be water cooled. The heat load on downstream optical components is reduced due to the high energy cutoff of the mirror. Single-crystal diamond possesses a very large thermal conductivity even at

room temperature, the expansion coefficient is smaller, and it absorbs less of the incident x-ray power than silicon making it attractive for high-heat-flux monochromators.[3] However, diffraction-quality diamond of the required size and orientation is difficult to obtain, which has limited its use to date.

It was recognized early in the planning stages for third-generation, hard x-ray synchrotrons that the power emitted by insertion devices, undulators in particular, would probably be the limiting factor affecting the quality (spectral brilliance and flux) of the delivered monochromatic beam. Overwhelmingly, the material of choice for hard x-ray monochromators is single-crystal silicon. Large, highly perfect boules of silicon are readily available due to the extensive use of silicon in the semiconductor industry. Also, fabrication techniques are well understood, silicon is UHV compatible and impervious to radiation damage. Additionally, the acceptance angle for Bragg diffraction (Darwin width) is well matched to the opening angle of radiation at the APS. The Darwin width for the Si(111) reflection at 8 keV is about 36 microradians, which compares favorably with the APS undulator vertical opening angle of about 20 microradians. For these reasons, great emphasis has been placed on developing silicon monochromators that can perform under the enormous power density of an APS undulator. Because of the very narrow acceptance angle, the optical performance of single-crystal monochromators is greatly degraded by thermo-mechanical strain. The most successful

monochromator design has proven to be cryogenically cooled silicon.

Cryogenic cooling of high power density synchrotron x-ray optics was first suggested in 1985 for mirrors[4] and in the following year for crystal monochromators.[5] As was pointed out in those papers, the advantage of operating single-crystal silicon optical components at cryogenic temperatures is twofold: (1) the thermal conductivity, k, increases by nearly an order of magnitude in going from room temperature to liquid nitrogen temperatures, while (2) the coefficient of thermal expansion, α, decreases from its room temperature value of 2.6×10^{-6} K^{-1}, going through zero at about 125 K, and remaining slightly negative. It can be shown that the thermal strain gradient in the scattering plane is directly proportional to the ratio, α/k. Consequently, the thermal gradients and resulting strain are much lower for silicon monochromators operated at cryogenic temperature compared to operation at room temperature. Cryogenic silicon monochromators were first tested on high power wiggler and focused wiggler beamlines at HASYLAB[6] and the National Synchrotron Light Source (NSLS).[7] Cryogenic monochromators have subsequently found wide use at the European Synchrotron Radiation Facility (ESRF).[8]

The goal for the cryogenic optics program at the APS has been to develop cryogenically cooled silicon monochromators that will deliver near theoretical performance over the widest possible functional range of insertion devices.

II. CRYSTAL MONOCHROMATORS

All but two of the insertion device beamlines at the APS utilize an undulator as the primary source. Consequently, most of the development effort has been directed at designing optics for the undulator beamlines. The APS undulator emits a very narrow, well-collimated beam with a very high power density. Accepting just the central-cone of radiation from undulator A yields about 780 W and a peak power density of about 179 W/mm^2 at the monochromator (29 m from the source) at a magnetic gap of 11.1 mm for 100 mA stored current. This corresponds to a first harmonic energy of 3.28 keV.

A double-crystal monochromator is typically the first optical element of most beamlines at the APS. Consequently, it is often exposed to the full central-cone power emitted by the source. The DCM utilizes diffraction to select and pass the desired energy band from the broad incident spectrum. Most of the remaining energy is absorbed by the first monochromator crystal. The quality of the diffracted beam is very sensitive to the perfection of the crystalline lattice. Any thermo-mechanical strain introduced into the crystal degrades the quality of the monochromatic beam. Some of the characteristics that a monochromator crystal assembly must possess are: high radiation resistance, UHV compatibility, and the ability to undergo many thermal cycles. Also, the cooling manifold and piping cannot transmit significant stress or vibration to the optic.

The thermal design considerations for cryogenic monochromator crystals are significantly different than for room-temperature crystals. The two main differences are that the thermal conductivity of silicon is about an order of magnitude larger at liquid nitrogen temperatures and the transport properties of liquid nitrogen are not as good as water, the most common heat transfer fluid. The primary resistances to heat flow from the crystal to the fluid are conduction resistance through the silicon and convection resistance through the solid-fluid interface. For room-temperature crystals, the conduction resistance often dominates. Therefore it is desirable to minimize the thermal path length by placing the coolant channels very close, about 1 mm or less, to the diffraction surface in order to decrease the temperature rise. However, the situation is reversed in the cryogenic case. The cooling channels should be placed much further from the diffraction surface due to the higher thermal conductivity and smaller heat transfer coefficient. Locating the cooling fluid further from the diffraction surface allows the heat to diffuse laterally throughout the crystal, thereby lowering the peak surface temperature and reducing the thermal flux at the cooling channel interface and the likelihood of boiling.

Two general classes of cryogenic crystals have been tested at the APS: thin and thick. Thin crystals are designed so that only a portion of the x-ray power is absorbed. Much of the hard x-ray energy passes unattenuated through the silicon. Roughly speaking, thin crystals are usually < 1 mm thick.

The advantages of thin crystals are:

- Less absorbed power
- Less liquid nitrogen consumption
- Smaller thermal gradients normal to surface

The disadvantages are:

- Heat flow restricted by thin membrane
- Fabrication is more difficult
- More susceptible to mechanical strain

The advantages of thick crystals are:

- Easier to fabricate
- Less susceptible to mechanical strain
- Cooling geometry is not limited
- Beam expansion geometries can be readily used

The disadvantages are:

- Most of the x-ray power is absorbed
- More liquid nitrogen consumption
- Crystal heat exchanger must be larger
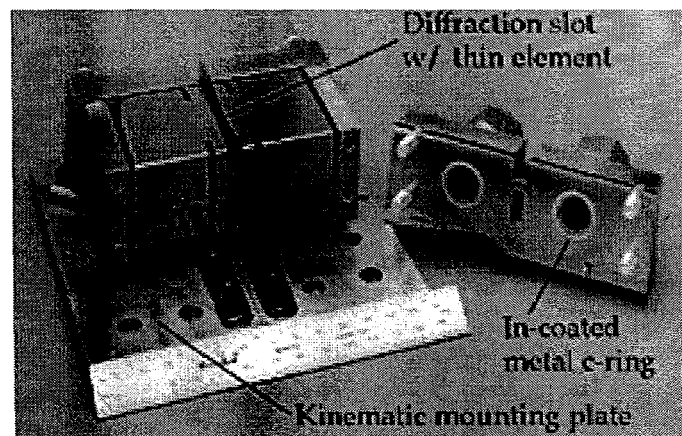- Larger thermal gradients normal to surface

FIG. 1. Photograph of the cryogenic crystal and coolant manifold.

III. EXPERIMENTS

Prior to beamline operations at the APS, cryogenically cooled monochromator crystals manufactured at the APS were tested on a focused wiggler beamline at the ESRF in a tripartite collaboration between the APS, ESRF, and the Japanese synchrotron facility SPring-8.[9,10] In these experiments, a thin, cryogenic crystal was tested up to a maximum incident power of 186 W with a peak power density at normal incidence of 521 W/mm^2. The crystals had slightly more mounting strain than the crystals recently tested at the APS, however the combined thermo-mechanical rocking curve broadening was less than 2 arc seconds at the highest power.

The crystal shown in Fig. 1 was installed and tested in May 1996 on APS sector 1-ID.[11] The crystal consists of a monolithic block of silicon incorporating a thin diffraction element and integral cooling channels. A relatively thin crystal was desired so that a large fraction of the incident beam power would be transmitted, hence reducing the absorbed power in the component. The thin element of the crystal is fabricated by milling slots in the top and bottom faces leaving a region approximately one-half mm thick. A third slot was milled in the downstream face to allow the transmitted beam to pass through. A maximum horizontal beam size of 2.5 mm can be accommodated. The downstream face of the crystal is visible showing the slot that allows the transmitted x-ray beam to pass through. The seal between the Invar manifold and the silicon is made via In-coated metal C-rings. Sealing pressure is maintained by using spring washers on the clamping screws. The mounted crystal assembly is supported on a kinematic plate that allows for unconstrained thermal expansion while preserving the absolute position of the thin diffraction element relative to the x-ray beam. One of the technical difficulties was the development of an ultrahigh-vacuum seal between the coolant manifold and the optical component that is radiation hard, can be thermally cycled, and introduces minimal strain into the crystal. This problem is exacerbated by the fact that the desired thickness of the diffracting crystal is less than one millimeter. The In-coated metal C-rings coupled with highly polished seal faces has proven to be a reliable cryogenic seal.

A similar cryogenic crystal was tested in October 1996. The only difference being that there was no slot fabricated into the top surface of the crystal. The thin element was fabricated by milling the downstream slot about 0.5 mm below the top face. This design is simpler to fabricate and it is possible to polish the diffraction surface. Some thermal performance is sacrificed because the heat exiting the lateral edge of the thin element has less material to diffuse into, but this has not proven to be a problem. One difficulty with the "slotted design" where the diffraction surface is below the crystal top surface as shown in Fig. 1, is that the acid etching process can create a radius on the crystal surface in the diffraction slot perpendicular to the beam direction. This can lead to a deformed beam shape due to the variation of optical path length across the beam footprint.

For the experiments on APS sector 1, the beam emitted by the undulator passed through a temporary commissioning window positioned at 23.5 m and consisting of 0.50 mm of graphite, 0.17 mm of CVD diamond, and 0.50 mm of Be. The fraction of the power absorbed in the commissioning window was about 12 % for a 2.5 V x 2.0 H mm^2 beam at an undulator gap of 11.1 mm. Horizontal and vertical white beam slits were located at 26.75 m, and the monochromator was at 28.5 m. A pair of ionization chambers were placed at 34 m to monitor the diffracted beam intensity.

An Al filter was placed between the ionization chambers so that the first- and third-order reflections could be recorded simultaneously.

The performance of a thick part of the crystal was also investigated. The thick crystal data were taken from the top surface of the monochromator crystal just laterally adjacent to the thin element. Obviously, the cooling geometry for the thick crystal data is not optimum because the heat flows predominantly to only one set of coolant channels; the other set is thermally isolated by the thin element. Rocking curve widths (FWHM) as a function of photon energy are shown in Fig. 2 for a fixed undulator gap of 11.1 mm corresponding to a deflection parameter, K, of 2.57. This situation simulates far worse heat loads than would normally be encountered at higher energies because the undulator gap was kept at 11.1 mm, corresponding to a first harmonic energy of 3.28 keV, for all of the rocking curves and was not opened to track the harmonic as the diffracted photon energy was increased, which would normally be the case. 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 incident power and peak power density are only about 40 percent of that at a gap of 11.1 mm. Consequently, for typical operation in which the gap (i.e., harmonic) is matched to the diffracted photon energy, the monochromator should perform equally as well at much higher currents.

The data for the thin crystal were collected with a 2.5 V x 2.0 H mm^2 and 2.0 V x 2.0 H mm^2 (normal incidence) beam with an incident measured maximum power of 561 W and 313 W, respectively. The beam size for the thick crystal data was 1.9 V x 3.0 H mm^2 with a maximum measured power of 495 W. The storage ring current ranged from 61 to 96 mA for the thin crystal data and from 89 to 95 mA for the thick crystal data. The liquid nitrogen volume flow rate ranged from 6.5 to 10.6 l/min. at a head pressure of 40 psia.

IV. SUMMARY

The cryogenically cooled monochromator and liquid nitrogen pumping system have been successfully tested and commissioned using the undulator on APS sector 1 ID and have provided the highest quality monochromatic beam. The thin, cryogenically cooled monochromator crystal has been tested under worst-case conditions with the APS undulator and displayed a thermal strain of no more than 1.0 to 1.5 arc seconds at the minimum undulator gap and a current up to 96 mA. The rocking curve broadening attributable to the manifold mounting stress was no more than about 1 arc second. The thick crystal performed much better than our expectations, and, due primarily to its lower mechanical strain in the diffraction volume, it exhibited sub-arcsec broadening.

An important benefit of the thin crystal is that it absorbs only a portion of the incident beam power. About 50 percent of the power was absorbed from a 2.5-mm-square beam at an undulator gap of 11.5 mm and a Bragg angle of 19.24°.

The future direction for cryogenic optics research at the APS is to explore cryogenic cooling of other optical components, such as mirrors and multilayers. Additionally, methods necessary to extend monochromator designs to higher storage ring currents (up to a maximum of 300 mA) will be explored. These techniques may include, enhanced heat exchangers using porous matrices,[12] and beam expansion geometries, such as inclined[13] and variable asymmetric crystals.[14]
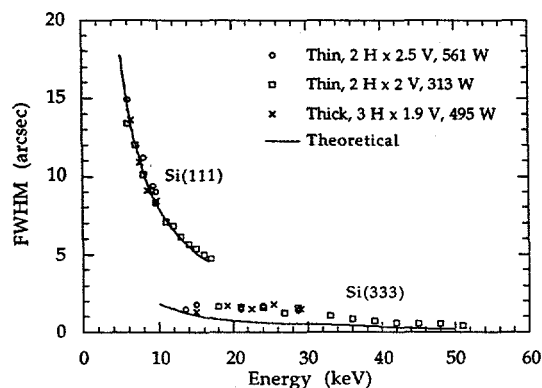
FIG. 2. First- and third-order rocking curve widths (FWHM) as a function of diffracted energy at a fixed undulator gap of 11.1 mm for several beam sizes. The beam incident on the thin part of the crystal measured 2.5 V x 2.0 H mm^2 with a power of 561 W in one case and 2.0 V x 2.0 H mm^2 with a power of 313 W in a second case. The beam on the thick part of the crystal measured 1.9 V x 3.0 H mm^2 with a power of 495 W.

ACKNOWLEDGMENTS

REFERENCES

[1] Lee, W. K., D. M. Mills, L. Assoufid, R. C. Blasdell, P. B. Fernandez, C. S. Rogers, and R. K. Smither, *Opt. Engr.*, **34** (2), 418, 1995.

[2] Yun, W., A. Khounsary, B. Lai, and E. Gluskin, *Argonne National Laboratory Technical Bulletin*, ANL/APS/TB-2, 1992.

[3] Blasdell, R. C., L. A. Assoufid, and D. M. Mills, *Argonne National Laboratory Technical Bulletin*, ANL/APS/TB-24, 1995.

[4] Rehn, V., *International Conference on Insertion Devices for Synchrotron Sources*, R. Tatchyn, and I. Lindau, eds., **582**, 238, SPIE - Soc. of Photo-Optical Instrumentation Engineers, Stanford, CA, 1985.

[5] Bilderback, D. H., *Nucl. Meth. in Phy.* **A246**, 434, 1986.

[6] Joksch, S., G. Marot, A. Freund, and M. Krisch, *Nucl. Instrum. & Meth. Phy. Res.*, **A306**, 386, 1991.

[7] Marot, G., M. Rossat, A. Freund, S. Joksch, H. Kawata, L. Zhang, E. Ziegler, L. Berman, D. Chapman, J. B. Hastings, and M. Iarocci, *Rev. Sci. Instrum.*, **63**, 477, 1992.

[8] Marot, G., and M. Rossat, *High Heat Flux Engineering, Proc. SPIE*, **1739**, 464, 1992.

[9] Rogers, C. S., D. M. Mills, W.-K. Lee, G. S. Knapp, J. Holmberg, A. Freund, M. Wulff, M. Rossat, M. Hanfland, and H. Yamaoka, *Rev. Sci. Instrum.*, **66**(6), 3494, 1995.

[10] Rogers, C. S., D. M. Mills, P. B. Fernandez, G. S. Knapp, M. Wulff, M. Hanfland, M. Rossat, A. Freund, G. Marot, J. Holmberg, and H. Yamaoka, *Rev. Sci. Instrum.*, **67**(9), CD-ROM, 1996.

[11] Rogers, C. S., D. M. Mills, W.-K. Lee, P. B. Fernandez, and T. Graber, *High Heat Flux Engineering III*, **2855**, A. M. Khounsary, ed., SPIE - Soc. of Photo-Optical Instrumentation Engineers, 1996.

[12] Rogers, C. S., D. M. Mills, L. Assoufid, and T. Graber, *Rev. Sci. Instrum.*, **67** (9), CD-ROM, 1996.

[13] Rogers, C. S., and L. Assoufid, *Rev. Sci. Instrum.*, **66**(2) part II, 1996.

[14] Smither, R. K., and P. B. Fernandez, *Nucl. Meth. in Phy.*, **A246**, 434, 1986.