

FURTHER TESTS ON LIQUID-NITROGEN-COOLED, THIN SILICON-CRYSTAL MONOCHROMATORS USING A FOCUSED WIGGLER SYNCHROTRON BEAM*

RECEIVED

C. S. Rogers, D. M. Mills, and P. B. Fernandez
*Advanced Photon Source, Argonne National Laboratory
Argonne, IL 60439*

FFB 16 1996

OSTI

G. S. Knapp
*Materials Science Division, Argonne National Laboratory
Argonne, Illinois 60439*

M. Wulff, M. Hanfland, M. Rossat, A. Freund, G. Marot, and J. Holmberg
*European Synchrotron Radiation Facility
BP 220, Grenoble, Cedex, France*

H. Yamaoka
*JAERI-RIKEN SPring-8 Project Team
2-1 Hirosawa, Wako, Saitama 351-01, Japan*

January 1996

The submitted manuscript has been authored by a contractor of the U.S. Government under contract No. W-31-109-ENG-38. Accordingly, the U.S. Government retains a nonexclusive, royalty-free license to publish or reproduce the published form of this contribution, or allow others to do so, for U.S. Government purposes.

Presented at the SRI '95 APS X-ray Centennial Symposium/Seventh Users Meeting for the APS, Argonne, IL, October 16-20, 1995; to be published in the proceedings as a peer-reviewed volume of the *Review of Scientific Instruments* in CD-ROM format.

*This work supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under contract #W-31-109-ENG-38.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED 35

Further tests on liquid-nitrogen-cooled, thin silicon-crystal monochromators using a focused wiggler synchrotron beam

C. S. Rogers, D. M. Mills, and P. B. Fernandez
Argonne National Laboratory, Advanced Photon Source
9700 South Cass Avenue, Argonne, IL 60439 USA

G. S. Knapp
Argonne National Laboratory, Material Science Division
Argonne, Illinois 60439 USA

M. Wulff, M. Hanfland, M. Rossat, A. Freund, G. Marot, and J. Holmberg
European Synchrotron Radiation Facility
BP 220, Grenoble, Cedex, France

H. Yamaoka
JAERI-RIKEN SPring-8 Project Team
2-1 Hirosawa, Wako, Saitama 351-01, Japan

(Presented on 19 Oct 1995)

A newly designed, cryogenically cooled, thin Si crystal monochromator was tested at the European Synchrotron Radiation Facility (ESRF) beamline BL3. It exhibited less than 1 arcsec of thermal strain up to a maximum incident power of 186 W and average power density of 521 W/mm². Data were collected for the thin (0.7 mm) portion of the crystal and for the thick (>25 mm) part. Rocking curves were measured as a function of incident power. With a low power beam, the Si(333) rocking curve at 30 keV for the thin and thick sections was < 1 arcsec FWHM at room temperature. The rocking curve of the thin section increased to 2.0 arcsec when cooled to 78 K, while the thick part was unaffected by the reduction in temperature. The rocking curve of the thin section broadened to 2.5 arcsec FWHM and that of the thick section broadened to 1.7 arcsec at the highest incident power. The proven range of performance for this monochromator has been extended to the power density, but not the absorbed power, expected for the Advanced Photon Source (APS) undulator A in closed-gap operation (first harmonic at 3.27 keV) at a storage-ring current of 300 mA.

I. INTRODUCTION

Most of the insertion device beamlines at the Advanced Photon Source (APS) will implement liquid-nitrogen-cooled, double-crystal monochromators, a technique pioneered at the European Synchrotron Radiation Facility (ESRF) and recently reviewed by Marot.¹ It has become the *de facto* standard cooling technique for undulator optics at the APS and is a part of its continuing high-heat-load R&D program.² In particular, a thin-crystal feature has been added to the design that further improves the performance of cryogenically cooled Si monochromators because a significant fraction of the power from insertion devices is in the high-energy part of the x-ray spectrum and is not absorbed in the thin crystal.³ Two experimental studies have been made over the past year under APS-like conditions. These experiments indicate that symmetric, Bragg reflection, cryogenically cooled, thin Si monochromators can handle the central cone radiation from APS undulator A up to a storage ring current of at least 100 mA with less than 1 arcsec of thermal broadening of the rocking curve. The first of these experiments was reported in Ref. 4; the results of the second experiment are the subject of this paper.

The APS undulator A emits a total power of nearly 5 kW with a peak power density of 167 W/mm² at a first harmonic energy of 3.27 keV at 30 m from the source.⁵ This

corresponds to the minimum undulator gap of 10.5 mm and the largest heat load. The emitted power and power density decrease as the gap is opened for higher first harmonic energies. For example, when the magnetic gap is adjusted for a first harmonic energy of 8.0 keV, the total emitted power is about 1.2 kW and the peak power density is 80 W/mm². Fortunately, the first and third harmonic photon flux is contained within a small solid angle, the so-called central cone, while the total power envelope subtends a much larger solid angle. Consequently, it is possible to use slits to reduce the total power load, but not the peak power density, on the crystal without impacting the useful flux delivered to the experiment. A pin-hole, measuring 2.0 x 2.5 mm² in the vertical and horizontal directions, allows 84% of the first harmonic (3.27 keV) and 88% of the third harmonic (9.81 keV) flux through while reducing the total power impinging on the crystal to about 732 W. Of this remaining power, only about 231 W is absorbed in a 0.7-mm-thick Si crystal. Figure 1 shows the calculated absorbed power in Si as a function of crystal thickness at several photon energies. The calculations were made for APS undulator A operating at 100 mA with a 2.0 x 2.5 mm² aperture located 30 m from the source. Note that as the gap is opened (increasing the first harmonic energy), the emitted power decreases, but, since the Bragg angle also decreases, a larger fraction of the power is absorbed for a given thickness.

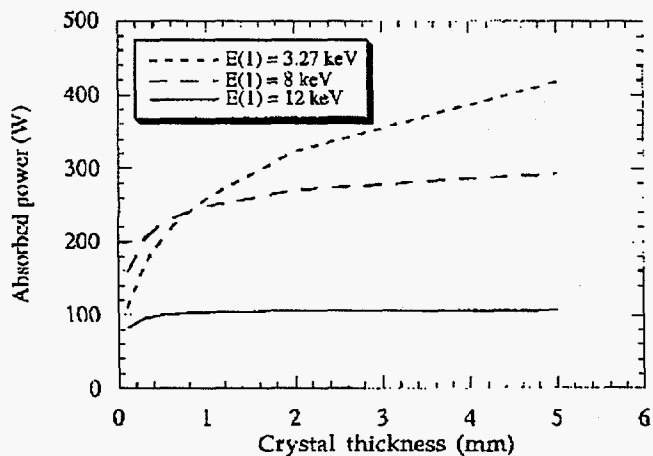


FIG. 1. Power absorbed in Si monochromator as a function of its thickness from APS undulator A for first-harmonic energies of 3.27, 8.0 and 12.0 keV at a current of 100 mA, through a $2.0 \times 2.5 \text{ mm}^2$ pinhole (vertical \times horizontal), ignoring Compton scattering. The total power through the pinhole is 732 W, 327 W, and 112 W, respectively.

Building on the success of the first experiment, a new crystal was fabricated in an effort to improve the characteristics of the previous design. Because most of the total strain in the original crystal was due to the mechanical mounting stress and the force exerted by the fluid seal, an effort was made to make the crystal less sensitive by increasing the thickness of the crystal around the cooling channels and moving the seal area further from the diffraction volume. The crystal was internally cooled in the same manner as before, but the overall dimensions were $35 \times 50 \times 85 \text{ mm}^3$. The diffracting element was about 0.7 mm thick. A photograph of the crystal and its coolant manifold is shown in Fig. 2. Liquid nitrogen enters the crystal through the downstream coolant manifold, passes through one side of the crystal, and is redirected by the upstream coolant manifold through the other side of the crystal. The liquid seal is made by In-coated, metal C-rings captured between the polished faces of the manifold and crystal by a retaining plate. The four strain-relief cuts between the coolant seal area and the diffraction slot were added after the present experiment. The manifolds are held in place with screws set into the side plates, and a constant sealing force is maintained with spring washers. The crystal assembly is mounted on a six-point kinematic/isolation plate. Two truncated tungsten carbide balls are attached to the underside of the upstream manifold and one to the downstream manifold. These balls ride on three sets of rails in the base plate made with truncated cylinders. The travel of the rails is directed toward the center of diffraction so that, as the crystal assembly contracts during cool down, the center of the crystal does not move relative to the base plate and the x-ray beam.

II. EXPERIMENTAL SETUP

As in the previous tests, this experiment was conducted on beamline BL3 at the ESRF using the same experimental

procedure.⁴ The x-ray source was a 44-pole wiggler with the magnetic gap set at 20.1 mm resulting in a deflection parameter, K , of 5.4. A Pt-coated toroidal mirror set at an incidence angle of 2.65 mrad was used to focus the wiggler beam to a spot size of $0.317 \times 1.125 \text{ mm}^2$ in the vertical and horizontal directions onto the crystal. The mirror cut-off energy was about 32 keV.⁶

The (111)-oriented cooled first crystal was placed inside a vacuum chamber and set at a Bragg angle, θ_B , of 11.4° to diffract 30 keV x-rays from the (333) planes. A Si (111)-oriented analyzer crystal was located outside the vacuum chamber, and the diffracted intensity was measured with a Si diode. A total of 1.5 mm of Be (3 windows) and 0.26 mm of C (window pre-filter) were between the wiggler and monochromator. Several different thickness of Al were placed in front of the monochromator to vary the heat load on the crystal. The spectral intensity distribution of the diffracted beam was preserved by maintaining a constant thickness of 2 mm of Al between the source and detector for the various combinations of power filters (e.g., 1.5-mm power filter before the monochromator and 0.5 mm after the monochromator). The remaining beam power after the attenuators was measured with a Cu calorimeter.

III. EXPERIMENTAL RESULTS

Rocking curve measurements were made for the Si(333) reflection at 30 keV. A Si(111) rocking curve at 10 keV was also recorded when all of the Al filters were removed from the beam. The theoretical widths of these rocking curves are 0.5 and 8.0 arcsec FWHM at 100 K, respectively. Rocking curves were taken on both the thin and thick portions of the crystal. After measuring the thin crystal rocking curve, the crystal was raised vertically by several millimeters so that the beam hit the thick part of the crystal but was still located within the slot on top of the crystal. Low beam power, "cold beam", measurements were made for the crystal at room temperature and at liquid nitrogen temperature by placing a 12-mm-thick Al plate in the beam. The cold-beam Si(333)

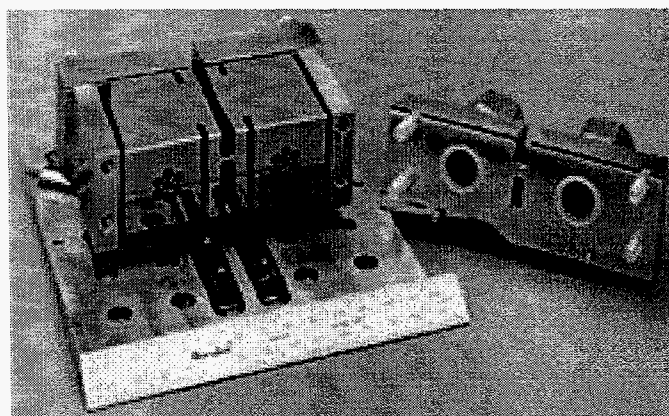


FIG. 2. Photograph of the cryogenic, thin Si monochromator showing its Invar coolant manifold assembly and six-point kinematic base plate.

rocking curve width for the thin section at room temperature was 0.9 arcsec FWHM and increased to 2.0 arcsec when cooled to 78 K. The cold-beam rocking curve width for the thick part at room temperature was 0.9 arcsec FWHM and remained essentially unchanged at 78 K.

Rocking curves as a function of power were taken by placing a series of Al filters in the beam. A summary of these measurements is given in Table I for the thin and thick parts of the crystal. Figures 3 and 4 show the Si(333) rocking curves at 30 keV for the thin and thick parts, respectively, for a cold beam and at the maximum power. Also shown, is the Si(111) rocking curve at 10 keV for a high power beam. Figure 5 shows the rocking curve width as a function of absorbed power density on the surface of the crystal for the thin and thick portion of the present crystal and for the thin part of the previously tested crystal reported in Ref. 4. The indicated power density takes into account the spreading of the incident beam due to the Bragg angle of 11.4° . The performance of the thin portion of both crystals was essentially the same showing less than 1 arcsec of thermal broadening. The mechanical strain in the thin part of about 2 arcsec dominates the contribution from the thermal strain, and therefore, the thermal component is not clearly resolvable. A slight broadening trend of the rocking curve for the thick part of the current crystal is discernible. This broadening is proportional to the power and of the order of 1 arcsec at the maximum power. The rocking curves obtained for the thin part show a different behavior. The width first increases until about 60 W/mm^2 and then tend to decrease. Both thin crystals gave this same result which, although small in magnitude, seems to be reproducible.

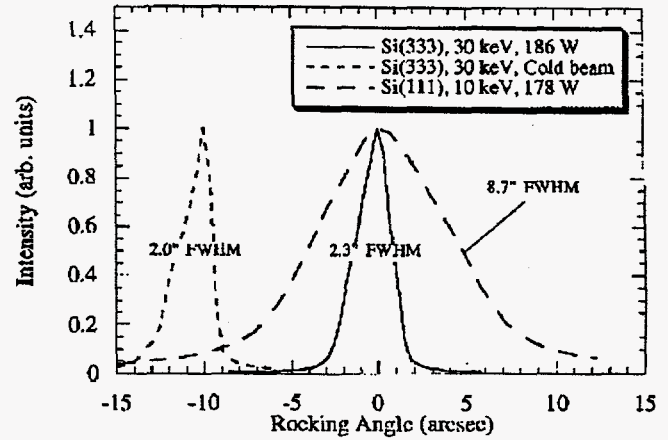


FIG. 3. Si(333) rocking curve at 30 keV for the cryogenically cooled thin crystal for the "cold beam" and at an incident power of 186 W and peak power density of 521 W/mm^2 and Si(111) rocking curve at 10 keV at an incident power of 178 W and peak power density of 500 W/mm^2 . The intensity is normalized to 1.0, and the peak of the cold-beam rocking curve is shifted on the angle axis for clarity.

IV. DISCUSSION

The results of this second series of experiments on heat load effects in cryogenically cooled Si single crystals fully confirm the observations of the first studies. The data presented in this paper extend that obtained in the previous experiment indicating that a thin, cryogenically cooled Si monochromator can operate up to incident power densities

Table I. Measured incident power, normal incidence power density, calculated fraction absorbed, and Si(333) rocking curve widths at 30 keV for the thin and the thick parts of the Si crystal monochromator cooled with liquid nitrogen.

Scan no.	Al filter (mm)	Incident power (W)	Incident power density (W/mm^2)	Fraction abs. in monochromator	Rocking curve width, FWHM (arcsec)
Thin					
148	12	N/A	N/A	N/A	2.0
135	2.0	39	109	0.766	2.0
142	1.5	45	126	0.786	2.1
145	1.0	67	187	0.810	2.3
137	0.5	94	264	0.843	2.4
146	0.25	130	364	0.864	2.6
139	0	186	521	0.896	2.3
Thick					
153	12	N/A	N/A	N/A	1.0
154	2.0	36	102	0.966	1.1
155	1.0	65	183	0.975	1.2
157	0.5	89	249	0.980	1.3
159	0.25	128	358	0.984	1.4
161	0	176	493	0.988	1.7

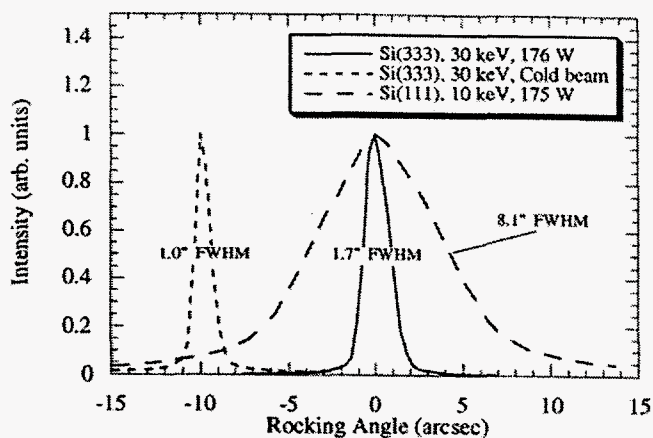


FIG. 4. Si(333) rocking curve at 30 keV for the cryogenically cooled thick crystal for the "cold beam" and at an incident power of 176 W and peak power density of 493 W/mm² and Si(111) rocking curve at 10 keV at an incident power of 175 W and peak power density of 491 W/mm². The intensity is normalized to 1.0, and the peak of the cold-beam rocking curve is shifted on the angle axis for clarity.

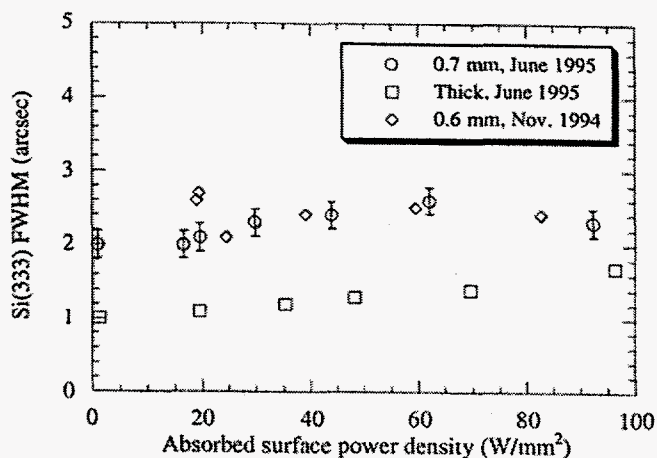


FIG. 5. Si(333) rocking curve widths (FWHM) at 30 keV as a function of absorbed power density on the surface of the crystal for the thin (0.7 mm), and thick (>25 mm) parts of the cryogenic crystal and from the previously tested thin (0.6 mm) crystal.¹ The error bars are the same for all data but only shown for the first data set for clarity.

approaching 520 W/mm² with minimal thermal strain. At first glance, the data suggest that the thick part of the crystal performed better than the thin portion. This is true primarily because the thick part was less affected by the mechanical stress at low temperatures. It should be remembered that this experiment used a mirror with a cut-off energy of 32 keV. For APS undulator A, a significant fraction of the spectral power is at higher energies. The thicker crystal would absorb much more of this power than the thin crystal possibly resulting in greater thermal strain. Additionally, the larger heat load consumes more liquid nitrogen.

The slight decrease of the rocking curve width at the highest power level was consistent with the results of the first study and was typical for the thin part, while the thick part behaved in the opposite way. The difference might be due to the combined effects of mechanical and thermal strain. As described above, the rocking curve width of the thin part increased from 0.9 arcsec to 2.0 arcsec when cooled from room temperature to liquid nitrogen temperature, whereas the thick part was not significantly affected by this stress. It is possible that the thin part buckled during cooling due to compressive forces. The tensile stress produced by heating the thin part in its center with the x-ray beam stretched it (remember that the thermal expansion coefficient is negative below 125 K) and flattened the buckle, which in turn decreased the measured rocking curve width. This effect is a specific advantage of cryogenic cooling of thin crystals and became appreciable only above 60 W/mm² in this case.

The increased thickness at the structural part of the crystal where the coolant channels are located and the increased distance between the seals and the diffraction volume, as compared to the previously tested crystal, did not decrease the mounting strain very much. The topography of the mounted crystal reveals significant localized strain in the vicinity of the seals. This strain propagates to the diffraction volume. The propagation of the sealing stress can likely be reduced by the strain-relief cuts shown in Fig. 2.

ACKNOWLEDGMENTS

These experiments were performed through a technical collaboration between the APS, ESRF, and SPring-8 laboratories. The authors express their appreciation to the staff at the ESRF for their exceptional assistance. This work is supported by the U. S. Department of Energy, BES-Materials Sciences, under contract No. W-31-109-ENG-38.

¹G. Marot, *Opt. Eng.* **34**, 426, (1995).

²W. K. Lee, D. M. Mills, L. Assoufid, R. C. Blasdel, P. B. Fernandez, C. S. Rogers, and R. K. Smither, *Opt. Eng.* **34**, 418, (1995).

³G. S. Knapp, M. A. Beno, C. S. Rogers, C. L. Wiley, and P. L. Cowan, *Rev. Sci. Instrum.*, **65**, 2792, (1994).

⁴C. S. Rogers, 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).

⁵R. J. Dejus, B. Lai, E. R. Moog, and E. Gluskin, Argonne National Laboratory Report, ANL/APS/TB-17, (1994).

⁶ESRF Beamline Handbook (ESRF, Grenoble, France, 1993), p. 37.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.