TEST RESULTS OF A DIAMOND DOUBLE-CRYSTAL MONOCHROMATOR AT THE ADVNACED PHOTON SOURCE*

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DISCLAIMER

Test results of a diamond double-crystal monochromator at the Advanced Photon Source (Invited)

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We have tested the first diamond double-crystal monochromator at the Advanced Photon Source (APS). The monochromator consisted of two synthetic type 1b (111) diamond plates in symmetric Bragg geometry. We tested two pairs of single-crystal plates: the first pair was 6 mm by 5 mm by 0.25 mm and 6 mm by 5 mm by 0.37 mm; the second set was 7 mm by 5.5 mm by 0.44 mm. The monochromator first crystal was indirectly cooled by edge contact with a water-cooled copper holder. We studied the performance of the monochromator under the high-power x-ray beam delivered by the APS undulator A. We found no indication of thermal distortions or strains even at the highest incident power (280 watts) and power density (123 W/mm^2 at normal incidence). The calculated maximum power and power density absorbed by the first crystal were 37 watts and 16 W/mm^2, respectively. We also compared the maximum intensity delivered by the diamond monochromator and by a silicon (111) cryogenically cooled monochromator. For energies in the range of 6 to 10 keV, the flux through the diamond monochromator was about a factor of two less than through the silicon monochromator, in good agreement with calculations. We conclude that water-cooled diamond monochromators can handle the high-power beams from the undulator beamlines at the APS. As single-crystal diamond plates of larger size and better quality become available, the use of diamond monochromators will become a very attractive option.

I. INTRODUCTION

The x-ray beams delivered by the undulators at the Advanced Photon Source can have a total power of several kilowatts, with a peak power density of 160 W/mm^2 at 30 m from the source. To preserve the brilliance of these beams, the first optical components of the x-ray beamline have to be designed to work under the extreme power loads without showing significant thermal distortions. Thermal management approaches that have been implemented in the design of the monochromator first crystal include: the use of crystals with internal cooling geometries that are optimized for maximum heat transfer;[1] the use of more efficient cooling fluids, such as liquid gallium;[2] modifications of the symmetric Bragg reflection geometry to reduce the power density on the crystal;[3] and improvement of the thermal properties of the diffracting material, for example by using cryogenically cooled silicon crystals[4] or room-temperature single-crystal diamonds.[5]

At room temperature, the thermal conductivity of diamond is about ten times larger than that for silicon, while the linear expansion coefficient of diamond is two times smaller. For the same absorbed power, cooling geometry and coolant, we expect that the temperature gradients and the thermal distortions in diamond will be considerably less than in silicon. Another advantage is that the absorption of x rays in diamond is less than in silicon: a 0.25-mm-thick diamond crystal will absorb 17% of 8 keV x rays, while a silicon crystal of the same thickness will absorb 97%. Thus, a thin diamond crystal will absorb a smaller fraction of the incident synchrotron beam, with considerably smaller thermal gradients and strain compared to a similarly cooled silicon crystal.[6] Also linked to the low absorption is the possibility of beam multiplexing, i.e., allowing the incident white beam to be used at several experimental stations.[5,7] The use of single-crystal diamonds as high-heat-load monochromators has been implemented at the European Synchrotron Radiation Facility[7] and is planned for beamlines at SPring-8.

While the thermal characteristics of diamond are superior to those of silicon, the opposite is true for the x-ray diffraction performance. For energies above 6 keV, the photon flux delivered by a double-crystal diamond (111) monochromator will be about two times lower than the flux from a silicon (111) monochromator. The reduction in flux is due to the smaller Darwin width and lattice

constant, which result in a narrower energy bandpass. This reduction may be an acceptable trade-off when the ease of cooling a diamond crystal is considered and/or when beams with a narrow energy width are required. Another drawback of using diamond crystals is the current unavailability of perfect single crystals of appropriate size. The largest commercially available plates at this point are 7 mm by 5 mm.[8] These plates are cleaved from synthetic type 1b stones and then are ground and polished to the desired thickness. The plates typically exhibit several arcseconds of mosaic spread and/or strain.

In this paper we describe the test of a high-heat-load double-crystal diamond (111) monochromator in Bragg reflection geometry. The tests were carried out on the Sector 1 insertion device beamline at the Advanced Photon Source, using x-ray beams produced by the 2.4-m-long undulator A. This beamline is operated by the Synchrotron Radiation Instrumentation Collaborative Access Team.

II. EXPERIMENTAL SETUP

Our double-crystal monochromator consisted of two type 1b, synthetic (111) diamond plates. The plates were manufactured by Drukker International, from synthetic stones grown by De Beers, and they were supplied by Harris Diamond Corporation.[8] We tested two sets of plates. Plates in the first set were 6 mm by 5 mm in size; the first crystal was 0.25 mm and the second 0.37 mm thick. Plates in the second set were 7 mm by 5.5 mm in size, and both crystals were 0.44 mm thick. All the plates had a slight asymmetry of the (111) planes, ranging from 0.99 to 3.16 degrees. We assessed the quality of the diamonds by taking x-ray topographs at 8 keV. The data indicate that the crystals are not perfect and that the mosaic spread/strain is of the order of 5 or 6 arcseconds over the full face of the plates. Residual strain in the crystal is due to impurities that can distort the perfect lattice, generate variations in the lattice spacing, and cause dislocations. The mosaic spread arises from the variation of the lattice orientation between the different growth sectors.[9,10] While the added mosaic spread/strain can result in a loss of brilliance in the diffracted undulator beam, it can also increase the bandpass for applications in which the flux is important, thus making up for some of the loss in throughput compared to a Si(111) monochromator.

For our first run (May 1996), the diamond first crystal straddled a 2-mm-wide trough on a water-cooled copper block, which resulted in a 2-mm-wide by 6-mm-long area available for diffraction. For the second set of tests (January 1997), we increased the trough in the copper block to 3 mm; a sketch of the first crystal mount is shown in Figure 1. The thermal contact between the diamond and the copper was achieved by using a thin layer of Ga/In eutectic (80% gallium, 20% indium); the crystals were held in place by the surface tension of the eutectic layer.

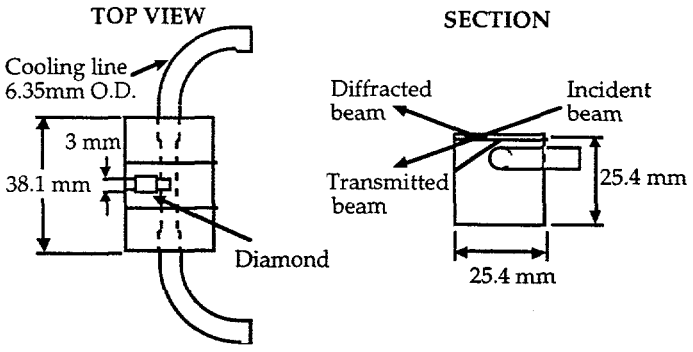

FIG. 1. Top view and section of the water-cooled first crystal mount of our diamond double-crystal monochromator. The copper block was nickel plated to prevent the diffusion of the Ga/In eutectic into the copper. The trough in the block allows the transmission of the incident x-ray beam.

The distance from the undulator x-ray source to the first crystal was 32.6 m for the May 1996 run and 29.4 m for the January 1997 run. White beam slits were located at 26.8 m and defined the beam size at the first crystal position: 1.4 mm horizontal by 1.8 mm vertical for the first run, and 2 mm horizontal by 1.2 mm vertical for the second run. Through these apertures, we accepted 62% and 83% of the undulator central cone of radiation in the horizontal plane, for the first and second runs, respectively, and over 97% in the vertical plane in both cases. An x-ray window was located between the front end and the beamline, and transmitted over 86% of the power produced by the undulator at a gap of 11 mm. The window consisted of a 0.5 mm (May 1996) or 0.3 mm (January 1997) graphite filter, followed by a 0.17 mm diamond window and two 0.25 mm beryllium foils. This window assembly was the only filter in the beam upstream of the monochromator.

Two ion chambers downstream from the monochromator vacuum tank recorded the intensity of the diffracted beam. The first ion chamber was filled with helium at atmospheric pressure; space charge effects from the undulator beam prevented operation in air for this counter. The second detector operated in air, behind an aluminum filter that absorbed x rays from the first order reflection of the (111) diamond crystals. Thus, the second ion chamber recorded the

intensity of the higher order reflections, mainly diamond (333). Figure 2 shows sample rocking curves for the (333) reflection, taken during our first run. The discrepancy between the theoretical and measured width of the rocking curves is due to the mosaic spread/strain of the diamond crystals. The measured width of the (333) reflection is a direct indication of the quality of the region of the crystals being sampled by the beam. For example, from the experimental (333) widths (see Figure 3 below), we deduce that the mosaic spread/strain is 2 to 4 arcseconds for the central 1.4-mm-wide strip of the crystals used in our first run. This measured widening of the (333) rocking curve is consistent with the 5 or 6 arcseconds of mosaic spread/strain observed over the full face of the crystals, as measured with the topography setup.

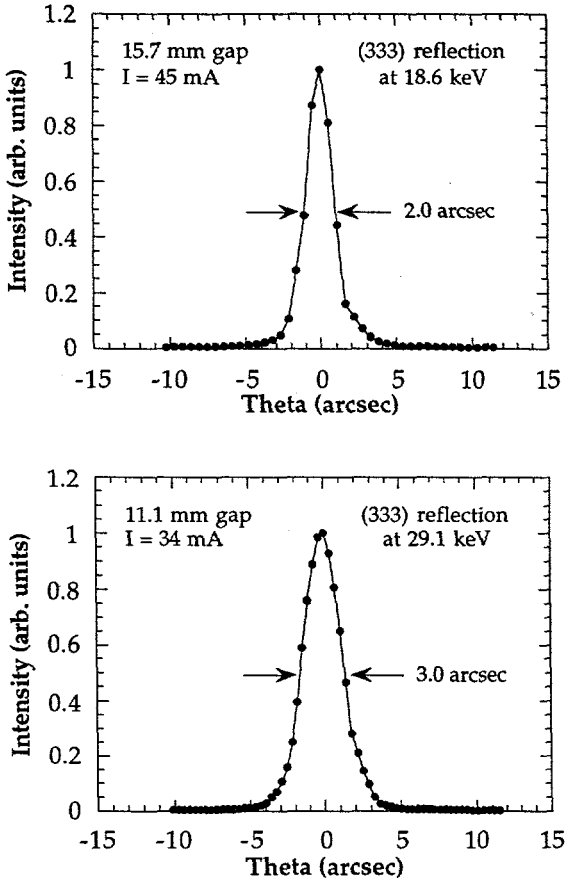

FIG. 2. Sample double-crystal rocking curves for the diamond (333) reflection (May 1996 data). The theoretical widths for the (333) reflection are 0.55 arcsec at 18.6 keV and 0.33 arcsec at 29.1 keV. The undulator gap and the storage ring current are indicated.

III. DATA

The purpose of our experiments was to test the diamond double-crystal monochromator under the high-power undulator beam. We typically gauge the performance of a cooled first crystal by tracking changes in the FWHM of the rocking curve of the diffracted beam as a function of the power incident on the crystal. The incident power can be increased by closing the undulator gap or increasing the storage ring current. Thermal stress and deformation in the crystal will result in an increase of the FWHM of the rocking curve.

We calculated the expected thermal deformation of the monochromator first crystal. We modeled our experimental configuration and used finite element analysis (FEA) to predict the thermal performance of the cooled diamond crystal at E = 17 keV, with the undulator gap at 11 mm and storage ring current I = 100 mA, for the parameters of the January 1997 run. The FEA calculation predicted a maximum temperature difference of 0.4 °C along the tangential direction (along the beam direction) and of 0.2 °C through the thickness of the crystal. The bowing and strains induced in the crystal by these temperature gradients resulted in a maximum tangential slope error (local bending of the diffraction planes) of 0.8 arcseconds. This slope error is much smaller than the theoretical (111) rocking curve width of 4.3 arcseconds, but larger than the calculated 0.23 arcseconds width for the (333) reflection at 51 keV. Because the thermal broadening would add in quadrature to the measured 2 to 4 arcseconds of mosaic spread/strain in the diamond crystals,[5] the FEA results indicated that we should not see a resolvable thermal broadening of the double-crystal rocking curve.

In our first run, we took data at two (111) diffraction energies, E = 6.2 and 9.7 keV, and several undulator gaps, 11.1, 15.7, and 21 mm. The corresponding first harmonic energies at these gaps were 3.3, 6.2, and 9.7 keV. For E = 6.2 keV, we took data on the second harmonic at closed gap (11.1 mm) and on the first harmonic at 15.7 mm, while for E = 9.7 keV, we used the third harmonic at 11.1 mm and the first harmonic at open gap (21 mm). The measured maximum power incident on the first crystal was 200 watts, with a calculated power density of 108 W/mm^2 (normal incidence), at 11.1 mm gap and 90 mA. The corresponding calculated power and power density absorbed by the first crystal were 11.6 watts and 1.8 W/mm^2, respectively, at 9.7 keV. The power incident on the first crystal is largest for the 11.1

mm gap configuration, but the power absorbed in the diamond is largest when the undulator gap is 21 mm. This counter-intuitive effect is due to the spectral distribution of the x-ray beam and to the fact that the diamond transmits most of the photons that have energies above 20 keV. The calculated maximum power and power density absorbed by the first crystal were 14.5 watts and 2.4 W/mm^2, respectively, at 21 mm gap and 96.5 mA, for E= 9.7 keV.

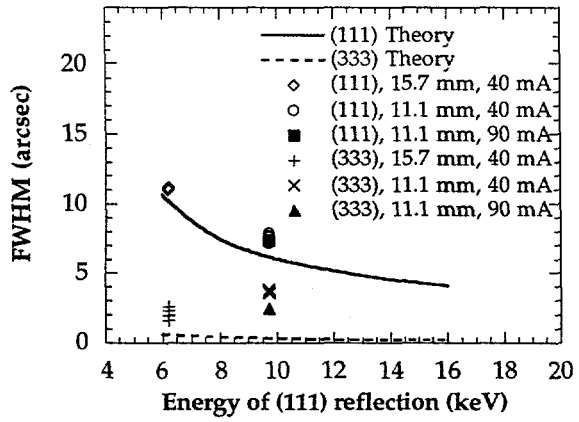

FIG. 3. Measured and calculated values for the FWHM of the diamond double-crystal rocking curve as a function of energy of the x-rays diffracted from the (111) planes (May 1996 data). Data were taken simultaneously for the (111) and (333) reflections; the energy of the x-rays diffracted by the (333) planes is three times the abscissa value. The undulator gap and the storage ring current are indicated.

Figure 3 shows the FWHM of the rocking curve of the diffracted beam as a function of energy, for the (111) and (333) reflections. The theoretical double-crystal rocking curve widths, calculated for perfect single crystals, are also shown. The scatter in the data is due to an unexplained relative motion between the x-ray beam and the crystals; the extreme sensitivity to the position of the beam is due to the less-than-perfect crystalline quality of the diamonds. An increase in the width as a function of increasing incident power (closing undulator gap or increasing ring current) would indicate a worsening performance of the monochromator. The data do not show such an increase. Thermal broadening of the rocking curve, if present, is probably less than 1 or 2 arcseconds.

In our second run, we measured the widths of the diffracted beam as a function of energy for a fixed gap (11 mm), see Figure 4, and we also changed the undulator gap so that the diffraction energy corresponded to either the first or third harmonic of the undulator spectrum, see Figure 5. At some energies, data were taken at two different undulator gaps; the gap is smaller when the energy coincides with the third harmonic. In Figure 5, the first harmonic gaps range from 17 to 26.5 mm, and from 11 to 15 mm for the third harmonic. The maximum power and power density (normal incidence) incident on the first crystal were 280 watts (measured) and 123 W/mm^2 (calculated), respectively, at 11 mm gap and 86 mA. The calculated maximum power and power density absorbed by the first crystal were 37 watts and 16 W/mm^2, at 17 keV (11 mm gap and 82 mA). The scatter in the data in Figures 4 and 5 is again due to a small motion in the incident x-ray beam relative to the imperfect diamond crystals. The data are encouraging from a thermal perspective, because there is not an observable increase in the width of the (333) rocking curve with increasing energy (increasing absorbed power) or decreasing undulator gap (increasing incident power).

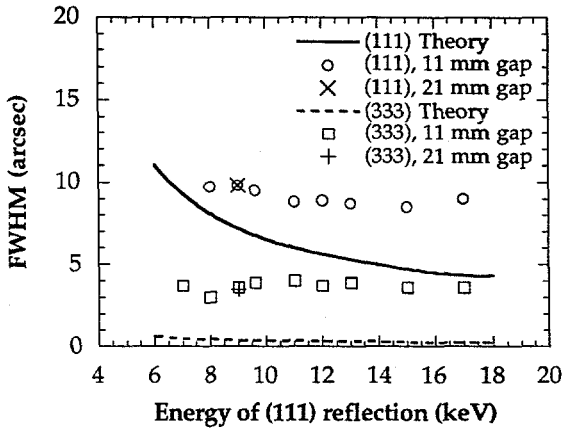

FIG. 4. Measured and calculated values for the FWHM of the diamond double-crystal rocking curve as a function of energy of the x-rays diffracted from the (111) planes (January 1997 data). Data were taken simultaneously for the (111) and (333) reflections; the energy of the x-rays diffracted by the (333) planes is three times the abscissa value. The storage ring current ranged from 87 to 82 mA.

We were also interested in comparing the flux through our diamond (111) monochromator and a silicon (111) double-crystal monochromator. For this comparison, we used a liquid-nitrogen-cooled silicon monochromator during the May 1996 run. This silicon monochromator had been successfully tested at the same beamline and had shown excellent performance under high thermal loads.[11] Using the same beam size and undulator gap, we measured the maximum intensity of the (111) reflection at 6.2 and 9.7 keV for both monochromators. We found the maximum intensity of the silicon monochromator to be 1.9 and 1.4 times that of the diamond monochromator

at 6.2 and 9.7 keV, respectively. The theoretical calculations for perfect crystals[6] predict a ratio of 1.6 at 6.2 keV and 1.9 at 9.7 keV. The reversed trend between the measured and calculated ratio may be explained as follows. At 9.7 keV, the imperfections in the diamond crystals account for a larger fraction of the double-crystal rocking curve width than at 6.2 keV. The reduction in the measured ratio at the higher energy is then due to a larger percent increase in flux for the diamond monochromator at 9.7 keV compared to 6.2 keV. The flux measurements are in reasonable agreement with the calculations and confirm the expected loss in monochromatic beam flux.

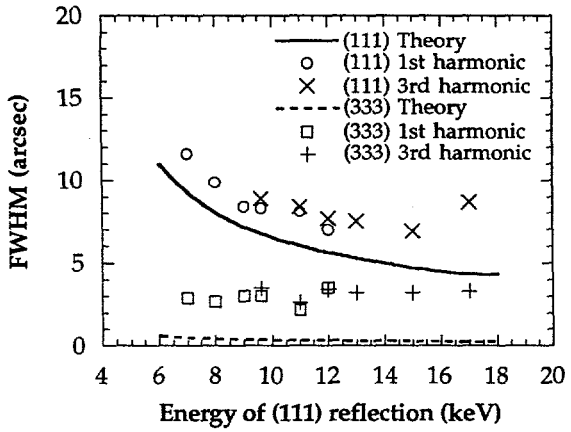

FIG. 5. Measured and calculated values for the FWHM of the diamond double-crystal rocking curve as a function of energy of the x-rays diffracted from the (111) planes (January 1997 data). Data were taken simultaneously for the (111) and (333) reflections; the energy of the x-rays diffracted by the (333) planes is three times the abscissa value. For a given energy, the gap setting of the third harmonic datum is smaller than that of the first harmonic point. The storage ring current ranged from 81 to 77 mA.

IV. CONCLUSIONS

We have installed and tested the first double-crystal diamond monochromator at the APS. Our data indicate that this water-cooled monochromator will perform well even under the highest heat loads on an undulator beamline. The drawbacks are the inherent reduction in flux compared to a silicon monochromator and the lack of absolutely perfect diamond single crystals of appropriate size. Even so, a diamond monochromator might prove a good choice for certain applications, for example, if a narrow energy bandpass or beam multiplexing is desired.

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