First High Heat Load Experience with a Cryogenically Cooled Diamond X-ray Monochromator Crystal

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Undulator radiation from the third generation storage rings, such as the ESRF, APS and SPring-8 rises a challenging heat load problem for the first optical component because the power and the power density exceed several hundreds of W and a few hundreds W/mm^2, respectively. They will be even higher for the fourth generation sources, such as TRISTAN MR (Tsukuba), and for SPring-8 long undulators to be installed in 30-m-long straight sections as well. It was shown experimentally that due to their excellent thermal characteristics and a low absorption coefficient of x-rays diamond single crystals perform much better than silicon, and are presently regarded as the most promising material for high heat load monochromators [1, 2]. Diamond crystals have been used on beamlines at the ESRF for several years and proposed to use them at SPring-8[3] . One can define a figure of merit for the performance of a monochromator material as $k/\mu\alpha$, where k, μ and α are the thermal conductivity, the absorption coefficient at 10 keV and the thermal expansion coefficient, respectively. It is well known that the thermal performance of silicon is much improved at low temperatures as the figure of merit indicates inFig.1.

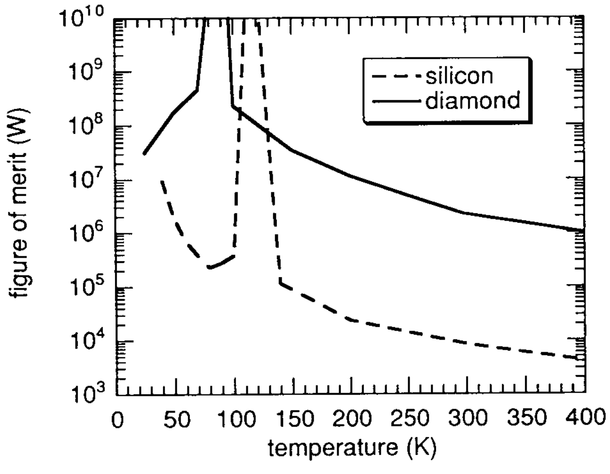

Fig. 1 Figure of merit, $k/\mu\alpha$ vs. temperature, where k, μ and α are the thermal conductivity (W/Km), the absorption coefficient at 10 keV (m^{-1}) and the thermal expansion coefficient (m^{-1}), respectively. The zero crossing points of the thermal expansion coefficient for Si and diamond are at about 125 K and 80-90 K, respectively.

The figure of merit of diamond crystals at room temperature is, however, already better than that of silicon at liquid nitrogen temperature, and the superiority of a diamond crystal is enhanced as the temperature approaches that of liquid nitrogen . In this report we briefly describe the first experience with a cryogenically edge-cooled diamond crystal exposed to a high power x-ray beam at the ESRF, confirming its excellent performance and revealing some technological problems[4].

A (100)-oriented synthetic type IIa diamond crystal of about 5x4 mm^2 in area and 0.2 mm thick manufactured by Sumitomo Ltd. was used. It was characterized at BL-15 of the Photon Factory (PF). The entire surface of the diamond was illuminated and the mosaic spread was 3.3 arcsec at the energy of 16.3 keV. The diamond crystal monochromator, cooled to cryogenic temperatures, was tested at the *wundulator* beamline, BL 3 (ID 9) at the ESRF. The cooled diamond crystal was exposed to the white beam after reflection by a tunable toroidal mirror coated with Pt set at a glancing angle of 2.8 mrad which was used to focus the beam. The vertical focus was 5.5 m upstream of the crystal, while the horizontal focus was 3.4 m downstream, in order to protect the Be windows before and after the crystal. The horizontal beam size was limited to 1.8 mm using a water-cooled tungsten-carbide slit in front of the diamond crystal. The vertical beam size was measured to be 0.2 mm (FWHM). The diamond (400) planes were positioned at a Bragg angle of about 17° (incidence energy of about 24 keV). The deformation caused by thermal and/or mounting strains was measured with a Si (440) analyzer crystal in almost dispersion-free double crystal (+, -) geometry. The calculated instrument resolution broadening due to the slightly dispersive condition was 2.4 arcsec for a vertical beam divergence (FWHM) of 28 arcsec obtained using the ray tracing program "shadow". The power loaded on the monochromator was measured with a copper calorimeter placed in front of the monochromator. The thin edge-cooled diamond was mounted on a Cu holder coated with Ni-Cr. The thermal contact between the crystal and the holder was established through three layers of In-Ga (75%/25%), In (0.3 mm thick), and In-Ga (75%/25%), respectively, providing some mechanical flexibility to release the strain generated during the cooling down to liquid nitrogen temperature. The crystal holder was connected to a standard ESRF cryogenic cooling system. The holder temperature was monitored at several points with thermocouples. During usual operation of the system, the flow rate and the pressure of liquid nitrogen before the crystal was 2.5 l/min (corresponding to a flow velocity of 0.5 m/s at the site of the crystal holder) and 3×10^5 Pa, respectively. Fig. 2 shows three rocking curves for the edge-cooled diamond crystal; one measured at room temperature with a "cold" beam and two at liquid nitrogen temperature with "hot" beams. All attenuators were removed in the hot beam case except a 0.26 mm thick graphite filter and three Be windows of 1.5 mm total thickness. The cold beam was obtained by inserting 4.6 mm thick Al attenuators before the crystal. The temperature indicated on the figure refers to the thermocouple in the crystal holder very close to the crystal. At room temperature the measured width of the cold beam rocking curve was 3.2 arcsec (FWHM), from which after deduction of the instrument broadening a FWHM of 2.2±0.2 arcsec is obtained. This result may be surprising when comparing it with the results of the characterization at the PF. The difference can be explained by the fact that the defects giving rise to the measured mosaic spread of 3.2 arcsec were grouped at the periphery of the crystal whereas the central part reflection the beam in this study was rather defect-free as found also in another experiment. On the other hand, mounting strain could as well have contributed to the measured width. Upon cooling from room temperature, the rocking curve width first became wider due to the strain introduced by the contracting crystal holder through the thin In-Ga and/or thick In layers interfaces. At a temperature of 90 K, the rocking curve gradually narrowed with time. After loading full beam power on the crystal, the observed width stabilized. Then the observed rocking curve width remained at 3.3 arcsec as shown in Fig. 2 with an incident beam of a total power of 120 W (13 W absorbed) and a power density of on the order of 96 W/mm^2 on the crystal surface (11 W/mm^2 absorbed, normal incidence power density of 330 W/mm^2) respectively. As explained above, the In-Ga/In/In-Ga stack absorbs the mechanical strain occurring between the crystal and the holder to some extent, that could be enhanced under full power irradiation. This process was time-dependent and probably more time will be needed for the crystal to completely relax. Also at 90 K, the incident power for the stable crystal was then reduced to 17 W (13 W/mm^2 on the crystal) by inserting 1.5 mm of Al. This resulted in a little larger width as shown in Fig. 2. The thermal contact probably became worse under the low power beam irradiation due to less power reaching the volume, and resulting in the larger rocking curve width observed.

In conclusion, no thermal strain could be observed on a cryogenically edge-cooled diamond crystal in the ranges of power and power density reported in this work. Edge cooling is very attractive because it permits beam multiplexing but the drawback is higher temperature increase with respect to face cooling. In the earlier work at room temperature, no thermal broadening of rocking curves was found at a total power of 280 W (8.7 W absorbed) and a normal incidence power density of 3.5 kW/mm^2 (109 W/mm^2 absorbed) when using edge-cooling of a 0.1 mm thick synthetic crystal. However a temperature rise of as much as 100°C was indicated. This can be drastically reduced by cryogenic cooling, but the thermal resistance across the In-Ga interfaces will limit the gain that also depends on the contact area of the interfaces. Presently the power absorbed by a 0.2 mm thick diamond crystal was estimated to be about 11 % of the incident power which at 90 K gave a temperature rise of the crystal itself less than only about 9 K for the absorbed power of 13 W. On the other hand, the In-Ga interface gave a rise to a temperature increase of about 55 K assuming a heat transfer coefficient of 0.04 W/mm^{2}K. Therefore, the size of the crystal must be as big as possible.

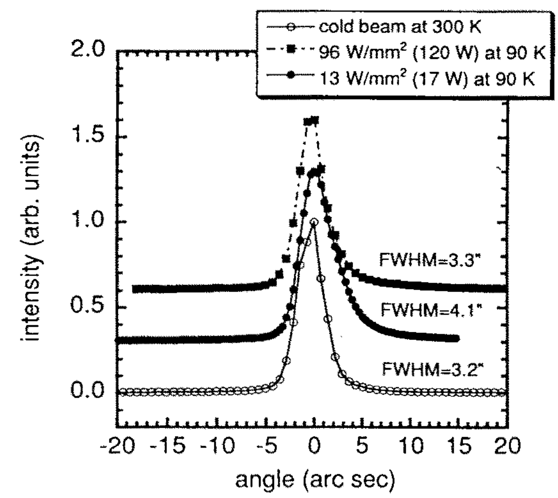

Fig. 2 Three rocking curves measured for the edge-cooled diamond crystal; one at room temperature with a cold beam and two at liquid nitrogen temperature of 90 K with hot beams. The power densities given are normal to the crystal surface.

References

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