

**Experiment title:**

Investigation of bonded diamond crystals for XFEL monochromators

Experiment**number:**

MI-989

Beamline:

BM05

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9

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Report:

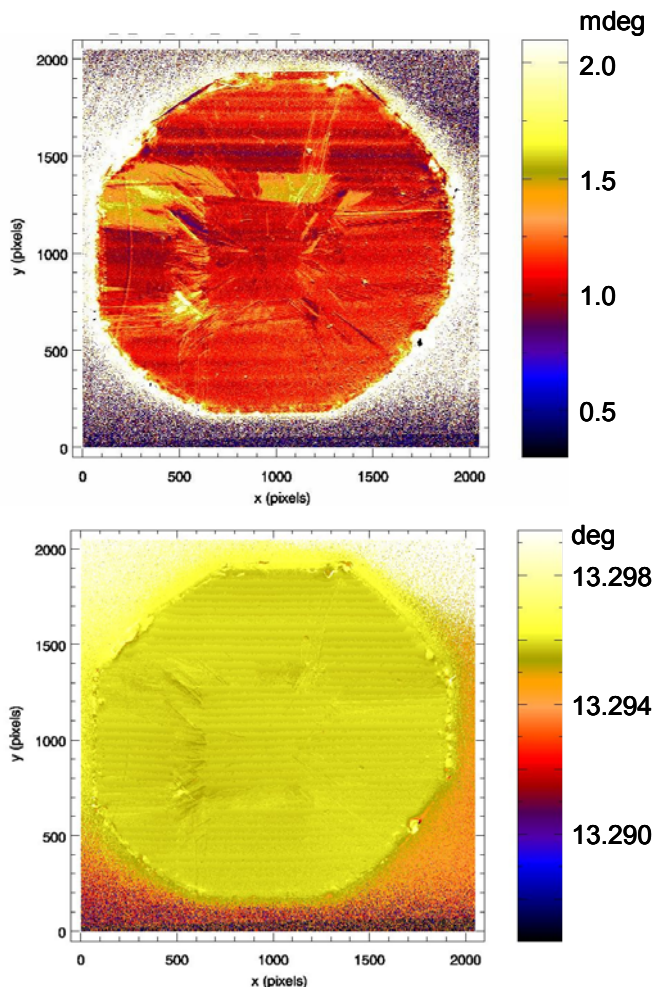
The entire project aim is to establish a new direct bonding technique for diamond single crystal monochromators that can provide an optimal heat transition but also a stress-free mount for high quality diamond single crystals. In this experiment we planned to characterize a series of HTHP IIa and CVD diamonds with (100) and (111) surface orientation using X-ray techniques suitable for characterization of thin diamond plates with strong masking of the working area by thick CVD heat sink material.

In the first part of the experiment the white beam topograms in Laue geometry were taken from all the samples to obtain information on defects in the crystal and strain field distributions along the entire surface as well as in depth. To quantitatively characterize the deformation state of the samples the rocking curve mapping, i.e. recording the local rocking curves over the entire surface in monochromatic setup were carried out. Experiments have been done at the BM05 beamline, double crystal Si(111) monochromator was used to diffract at diamond samples oriented for symmetric (220) Laue-case reflection. A reference wavelength was $\lambda=0.062$ nm (20keV). The maps were recorded with the FReLoN camera with effective pixel size 3 μm . VISROCK software [1] was used for processing the experimental data. In addition the Si prototype of the monochromator was studied using both white beam topography and rocking curve mapping at energy 30 keV.

Results:

The half-width of the reflection curve correspond to a near-perfect crystalline quality, the rocking curve widths for the 1x1 mm² areas free from defects are shown in the table 1. For a fixed position on the detector, the rocking curve width is defined by the spectral distribution of the beam impinging on the crystal, convoluted with the angular resolution of the recording device [2]. The angular effect of the spectral resolution $\Delta E/E = 1.3 \times 10^{-4}$ of the silicon-(111) reflection was 1.04 mdeg. The angular resolution of the detector was the pixel size divided by the distance between the diamond crystal and the camera, which was $3\mu\text{m}/0.4\text{m} = 0.42$ mdeg. The rocking curve width for a perfect crystal is $\Delta\omega=(1.04^2+0.21^2)^{1/2}=1.06$ mdeg. The white beam topograms and rocking curve maps show that most of the crystals have high quality and local

defect-free areas of the crystal up to $2 \times 2 \text{ mm}^2$ may be sufficient for monochromator applications in Laue geometry. The crystals showed no effects of bending associated with the manufacture process.



In the Si prototype rocking curve mapping, the local lattice plane inclination induced by bonding and the lattice strain FWHM in different points are seen. One can estimate that the radius of curvature due to the bonding about 20 m. Reconstructed surface distortions in 220-diffraction plane are shown at Fig.2.

Prospects:

In a follow-up experiment, after the samples under investigation are bonded to non-perfect CVD heat sinks, new maps will be generated to monitor exactly the strain fields introduced by bonding. It will allow to characterize and improve the diamond bonding technologies necessary for the high heat load diamond monochromator layout.

Table 1.

Crystals (surface orientation)	FWHM mdeg
#1 (100)	1.5 ± 0.2
#2 (100)	1.2 ± 0.1
#3 (111)	1.5 ± 0.2
#13/1 (100)	1.1 ± 0.1
#16/1 (100)	1.4 ± 0.2
CVD (100)	1.2 ± 0.1

Fig.1 (220) symmetrical Laue reflection rocking curve width FWHM (top) and peak position (bottom) maps of (100)-oriented HTHP IIa diamond crystal recorded with FReLon camera.

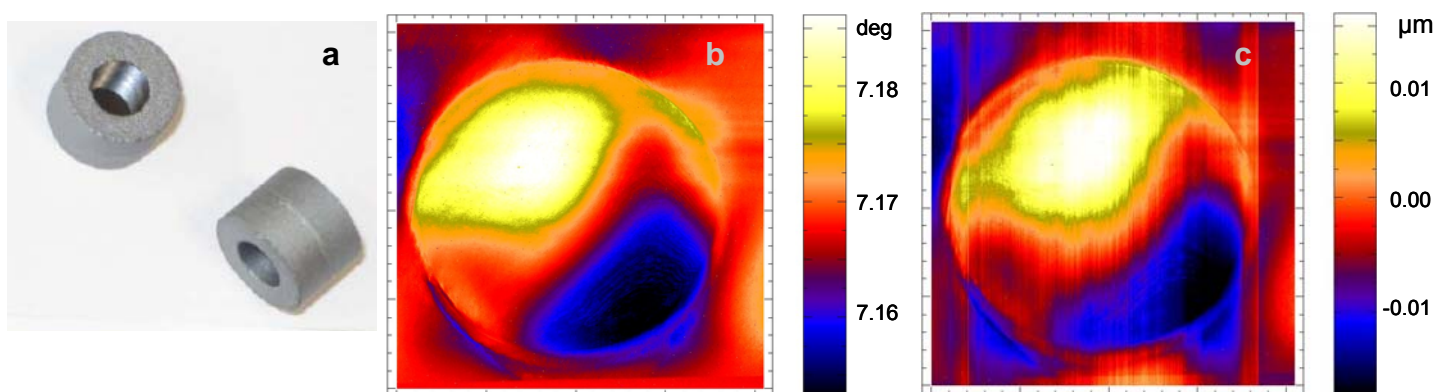


Fig.2. (a) The prototype consisting of a 100 micron thin (111)-oriented silicon crystal in the center and two thick rings bonded onto the thin disk on each side (outer diameter is 10 mm). Si (220) peak position (b) and corresponding height errors (c) maps of the 100- μm bonded silicon crystal.

References:

1. D. Lübbert and T. Baumbach, *J. Appl. Cryst.* (2007). **40**, 595-597
<http://www.esrf.eu/computing/scientific/VISROCK/MAIN.htm>
2. J Hoszowska et al, *J. Phys. D: Appl. Phys.* (2001) **34**, A47-A51