Experiment title:

Characterization of optical components for ID14

Experiment number:

MI-61

Beamline: **Date of Experiment:**

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Shifts: **Local contact(s):**

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Report:

We are developing the beamline ID 14 “Quadriga” for macromolecular crystallography. The centrepiece of the optics are transparent diamond monochromator crystals in order to supply the different experimental stations with a monochromatic X-ray beam. Several synthetic diamonds from Sumitomo Inc., Japan, have been characterised in detail:

Two (11 1) oriented crystals, size - 5*5*0.4 mm, type IIa; two (100) oriented crystals, size - 6*6*0.1 mm, type IIa; one (11 O) oriented crystal, size - 10*3* 0.5 mm, type Ia.

The experiments have been carried out using a 13 µm * 100 µm (FWHM, her. *vert.) white beam illuminating a part of the crystal. The divergence of the beam is about 8*5 µrad. The diffracted beam (energy 13290 keV) has either been analysed by scanning another 13 µm slit trough the diffracted beam and measuring the signal of a scintillation counter. These scans have been done at different distances from the sample (Typically 200 mm, 670 mm, 1770 mm). Different parts of the diamond crystals have been analysed. These scans were used to obtain a section topography in the case of the (111) crystals which were analysed in Bragg mode (111 reflection). In the case of the (100) or (11 O) crystals, Laue mode (111 reflection) was used and we were interested in focusing effects and in the dispersion of the diffracted beam. In order to analyse this dispersion more in detail the energy distribution in the diffracted beam for a given position of the 2nd slit has been measured using the (555) reflection of a channel cut Si-crystal in vertical scattering geometry. The energy resolution of the analyser crystal was about 0.07 eV. These measurement showed the polychromatic dispersion of the crystals in Laue mode. In order to obtain full rocking curves, the energy bandwidth has been measured without the second slit.

Fig. 1 shows. section topographies for different positions on the diamond crystal in Bragg mode. The main diffraction comes from the surface, where the beam enters the crystal (on the right). Imperfections, especially domain boundaries lead to diffracted intensity arising from the volume of the crystal. These topographies showed the high quality of the type IIb diamond crystals. There are only a few, rather big domains.

Fig 2. shows a scan through the reflection from a crystal in asymmetric Laue mode (111 reflection, asymmetry angle 55°). These crystals show very markedly phenomena due to dynamical diffraction. As well, focusing with a focus at -2500 mm is observed when the maximum of the intensity is considered, whereas the rms width of the diffracted beam is still divergent.

These crystals shows only the presence of a few domains which behave like ideal crystals. By consequence the reflectivity of these crystals is low, which combined with the polychromatic dispersion of the beam due to the Laue orientation is a major drawback for the use of diamond monochromator crystals in Laue mode.

Intensity
(arbitr. units)

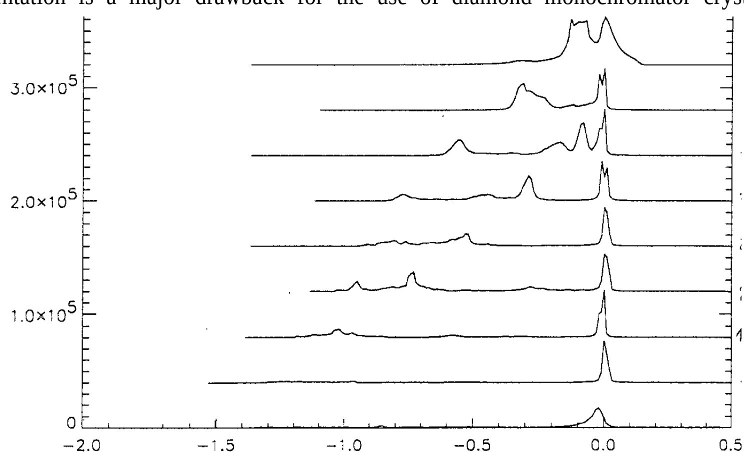
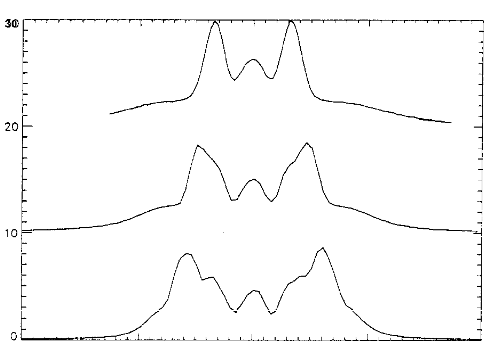


Fig. 1 Scans across the diffracted beam, distance from crystal d=200 mm
The different curves correspond to different positions on the crystal face, horizontal translation in mm.

Intensity
(arbitr. units)

d=1260 mm

d=670 mm



d=200 mm

Fig. 2 Scans across the diffracted beam at different distances in Laue mode.