ESRF	**Experiment title:** **Coherence properties and surface quality of Bragg-diffracting diamond plates**	**Experiment number**: MA-805
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Shifts: 12	**Local contact(s)**: Gerardina CARBONE (ID01) Tobias SCHÜLLI (ID01)	*Received at ESRF:*

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Report:

As a direct result of this experiment at ID01, a paper has been accepted for poster presentation at the X-TOP 2010 conference (20th-23rd September, Warwick, UK). The abstract is given below, and serves to give key information regarding the motivation for MA-805, and some of the results:

Abstract accepted for poster presentation at X-TOP 2010:

INVESTIGATION OF SURFACE AND SUB-SURFACE DAMAGE IN HIGH QUALITY SYNTHETIC DIAMONDS BY X-RAY REFLECTIVITY AND GRAZING INCIDENCE DIFFRACTION

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High quality synthetic diamonds are under development as X-ray optical elements for 3rd generation synchrotron and XFEL sources, due primarily to their superior heat-handling properties compared to silicon. For such applications, highly perfect crystals, both in terms of bulk quality and surface quality, are required [1] and much progress has been made in achieving this in recent years [2]. Grazing incidence X-ray reflectivity (XRR) and in-plane diffraction (GID) are surface- and sub-surface-sensitive techniques giving complementary information. We compare results from the cleaved surfaces and the hot-metal polished surfaces of 111-oriented samples. Data were collected at undulator beamline ID01 at the ESRF.

Polishing diamonds is a challenging task, given the hardness of diamond, and it cannot be achieved without considerable miscut on the 111 surface by conventional scaif polishing. The hot-metal polished surfaces showed a higher roughness than the cleaved surface, while the cleaved surfaces are terraced. X-ray specular

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reflectivity data show that the samples behave as if there are thin layers at the surface (a thinner, smoother “layer” for the cleaved surface) with bulk material below. Transverse diffuse reflectivity curves confirm differences between polished and cleaved surfaces.

GID reflections $02\bar{2}$ and $0\bar{2}2$ have been measured, the data exhibiting contributions from bulk, surface and background intensity. Data of the $02\bar{2}$ reflection indicate a damaged, near-surface region under compressive stress on the polished surface (Fig. 1).

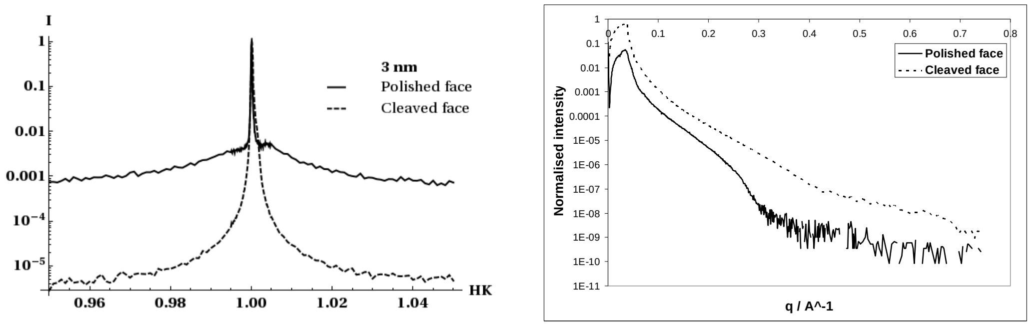

Figure 1. Left: GID comparison between polished and cleaved faces for a penetration depth of 3 nm. Right: Specular XRR for the polished and cleaved faces.

[1] R.C. Burns et al., J. Phys.: Condens. Matter **21**, 364224 (2009).
[2] R.C. Burns et al., Proc. of SPIE Vol. **6705**, 67050K1-6 (2007).

End of abstract

X-ray optical applications require that the highest intensity, quality and uniformity, and optimum coherence of the X-ray beam be delivered to the sample position. The study of production and preparation processes, and their effects on bulk and surface quality, are critical for development of suitable optics. New, reliable and reproducible techniques must be developed, which produce defect-free bulk, a smooth surface finish and preserve flatness.

Surface-sensitive XRR and depth-dependent GID experiments, including reciprocal space mapping, were carried out at ID01, exploiting the high brilliance, low divergence, small-size beam necessary for diamond samples only a few millimetres across, and benefitting from the expertise of the staff there. We focused on three (111)-oriented samples, studying one in particular in depth. Optical profilometry measurements were carried out at the ESRF’s Metrology Lab. We find that: a) cleaved surfaces have terraces which are individually quite smooth; b) polished surfaces are generally rough and/or curved with height differences across the sample of ~1-3 µm; c) specular XRR data generally require two near-surface “layers” of different density and roughness in order to fit the data, though the model structure depends on the direction across the sample of the X-ray beam; d) some XRR data cannot be fitted, probably either because the surface is too rough, or the nature of the “layers” changes as the footprint of the beam changes, e.g. across a terrace; indeed, some samples showed no specular XRR, only diffuse reflectivity arising from the roughness; e) the damaged surface layer appears to be under compressive strain; f) the damaged region makes a significant contribution to diffuse scatter in GID scans; and g) surface miscuts ranged from zero to ~5° - control of this parameter is important for the final application of diamonds as X-ray optical elements, but also as it limits the accessibility and affects interpretation of GID. Detailed analysis is ongoing in preparation for the conference poster and the manuscript for the published proceedings.

Other samples in the set remain to be measured, which would significantly add to our understanding of these materials and processes, including the effectiveness and reproducibility of the crystal growth, cutting and surface preparation techniques.

White beam and monochromatic beam topography and rocking curve imaging are complementary techniques which have been or will be applied to such samples, giving information on the bulk and surface quality by imaging defects such as dislocations, inclusions and scratches.