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Report:

Samples and experimental methods used

Synthetic single-crystalline HPHT-diamond is in principle the best-suited material for most X-ray optical elements to be used in 3rd and especially in 4th generation (e.g. FEL) X-ray sources. For applications such as coherent imaging, extreme focussing, and photon correlation spectroscopy a high-quality diamond material is needed that combines extremely high perfection of the crystal bulk and with an excellent surface finish. A substantial effort was undertaken (and need to be continued) in the fields of crystal growth, crystal processing and crystal characterisation to develop such material. The focus point of this proposal was part of those activities and we plan to give a kind of summary where we arrived and which are the major problems still to solve.

As already reported in previous experiement, the progress in the quality of the bulk of diamond has been remarkable in the last few years. At the moment the better solution is **diamond type IIa** material (low nitrogen impurity concentration), where regions of the order of 4x4 mm^2 may be defects free (no dislocations, no stacking faults, no inclusions, no local impurity concentration variations that result in local relative lattice parameter variations above few 10^{-8}).

The surface quality appears to be the most critical point at the moment. For very demanding applications the crystal plates should have rms-roughness in the order of 1 or 2 Å. This is very challenging

for diamond samples. Moreover, for coherent preserving optics, both the geometrical surface and the cristalline quality of the sub-surface layer are important. In order to achieve these requirements, a new polishing technique has been developed in collaboration between iThemba Labs and the University of Johannesburg. This method required an heated iron wheel whose temperature is on the order of 800° C. At this temperature, the carbon atoms are dissolved into the wheel itself, leaving a flat surface. Using the Hot Metal method is possible to polish also the 111 direction of diamond, what is impossible to do with normal scaif polihing techniques.

We concentrated on three different samples, each with a different orientation - 100, 110 and 111 (Figure 1). The 100 and the 110 samples have a face that is Hot Metal polished and the other face normal scaif polished. For what concerns the 111, only one face has been Hot Metal polished, while the othe face has been left “as- cleaved”.

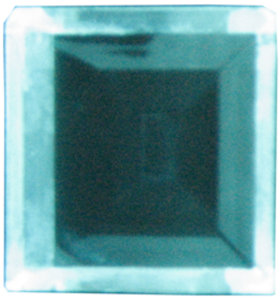
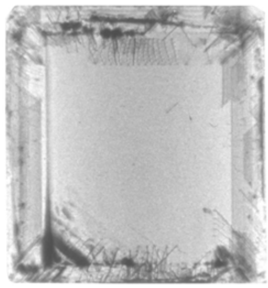
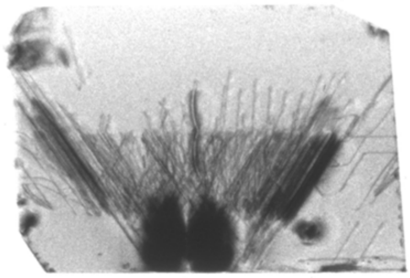
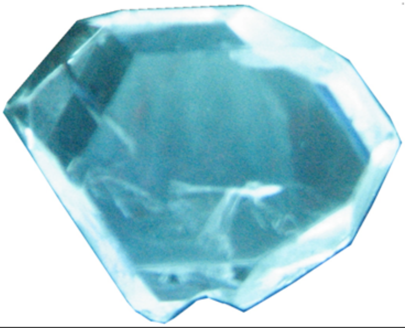
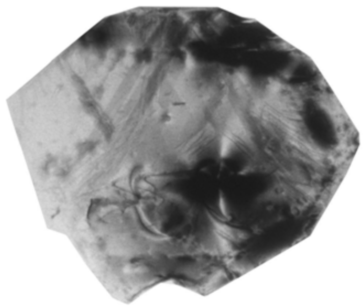
Diamond	UV Luminescence	White-Beam X-Ray Topography (14 keV, 220 reflection)
(100) 0.530 ct 2 mm		
(110) 0.290 ct 2 mm		
(111) 0.470 ct 2 mm		

Figure 1 UV luminescence images and white beam X-ray topograpsh of the three diamonds used. The reflections are 220 for the 100-oriented plate, 111 for the 110-oriented one, and 220 for the 111-oriented one.

In addition to optical methods and x-ray topography, the quality of the diamonds was characterized by the degree of the preservation of the transversal coherence using the Talbot effect [1]. The result depends on the bulk and surface quality. So the test method is rather close to the final application as an X-ray optical element at a beamline.

The geometrical surfaces have been characterized using a phase shift interferometer (Metrology Laboratory – Amparo Rommeveaux) and X-ray reflectometry (XRR) at ID01. Moreover at ID01 we performed Grazing Incidence Diffraction (GID) on the different sample surfaces. This method may be used in a depth controlled way and allows to obtain information on the crystalline quality of the sample at different depths. Those measurements were rarely done with such kind of diamonds. So we learned about

several prerequisite for succesful measurement which are not so evident when one works with other semiconductor materials. Already the fact that those HPHT-diamonds are rather small makes the life more complicated when doing XRR and GID. In addition to that, the low absorption makes it more important to correctly screen the “entrance side face” of the plate to avoit spurious signal contributions. We had to learn that it is a real problem to polish those small sample of that extremely hard material in a way that the processed surfaces are not bent (example in figure 2). In addition to this it appeared the it is not so easy, like for silicon samples, to process the samples such that the miscut angle (angle between crystal surface and lattice planes) is small, that is smaller than 0.2°.

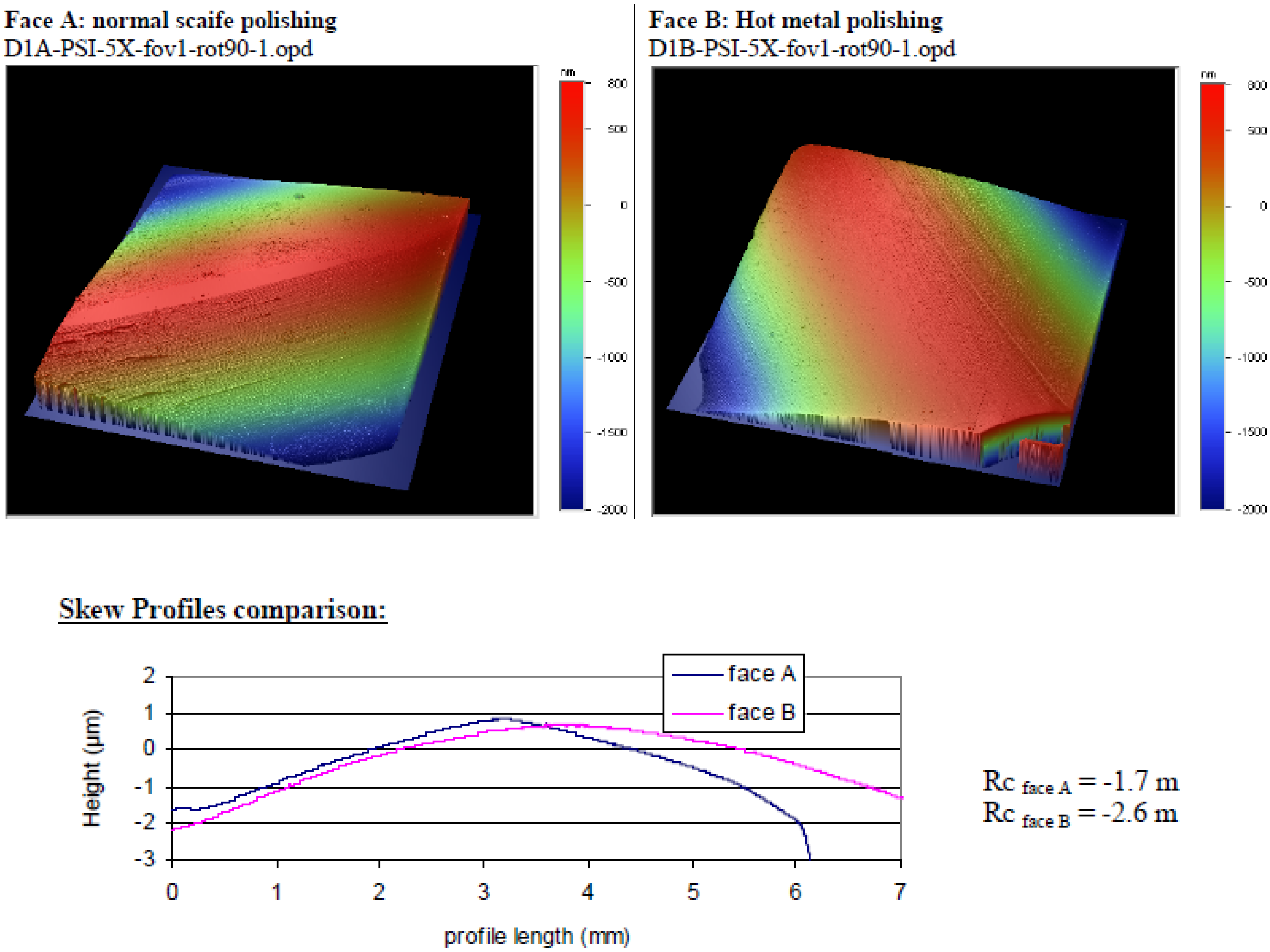


Figure 2: Surface profile of the 100-oriented plate

Results

Coherence measurements

The measured increase of the effective angular source size, here indicated as vertical and horizontal divergence, were carried out with the following experimental parameters:
Undulator U32, gap = 17 mm, E = 20.5 keV, 6µm mesh, mesh-to-camera distances 300mm and 900mm.

For comparison we give the **theoretical values**:
vertical divergence **0.2 µrad** and
horizontal divergence **0.9 µrad**.

A goal for the optically measured (for a magnification 50x) micro-roughness is **0.1 to 0.2 nm**

This value has to be approached and partly reached for coherence preserving silicon monochromators and surely has to be reached as well for diamond Bragg diffracting X-ray optical elements.

As experimental reference the values measured **without any sample**, but with windows, filters, absorbers and monochromator crystals on the way may be used:

vertical divergence **0.34 μrad**
horizontal divergence **1.1 μrad**.

It appears that they are close to the mentioned theoretical limit. Thus, windows, absorbers and the monochromator crystals decrease the coherence only on a limited but not negligible level.

Example of results obtained with a diamond 100 oriented, Bragg case on the 2 different surfaces (Hot Metal polished and scaif polished):

	vertical divergence	horizontal divergence
Hot Metal	**1.6 μrad**	**2.4 μrad**
Scaif	**2.2 μrad**	**3.0 μrad**

Table 1

From these results, it is visible that the coherence less disturbed by surfaces processed by hot metal polishing.

Reflectivity results

The results obtained with the Talbot effect are confirmed by optical measurements made in the ESRF metrology laboratory, where a smaller roughness has been measured on the samples that have been Hot Metal polished compared to the scaif polished ones (e.g. for the 100 oriented diamond, an average roughness of 6.6 nm for the hot metal polished compare to 15.8 nm for the scaif polished ones).

The reflectivity data taken at ID01 are still under analysis, a first example of fits of the specular reflectivity is shown in figure 3 and table 2. From these results, a deformed layer with a thickness of around 60-70 Å may be deduced, that is of the same order of magnitude like the mesured roughness.

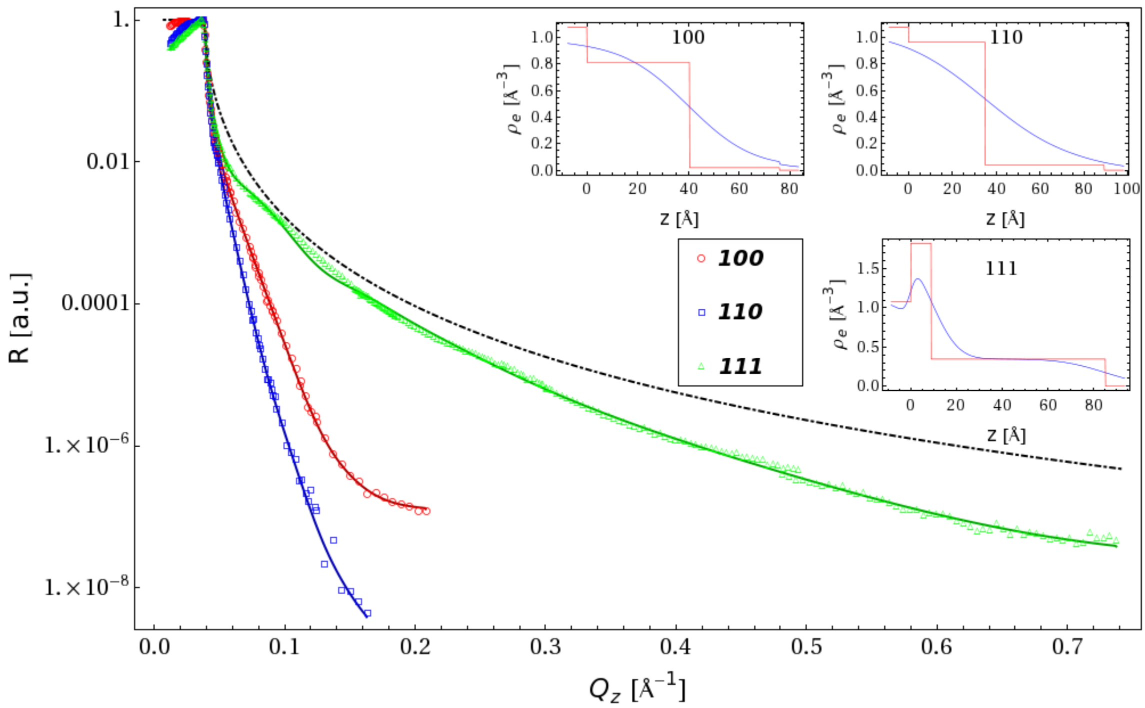


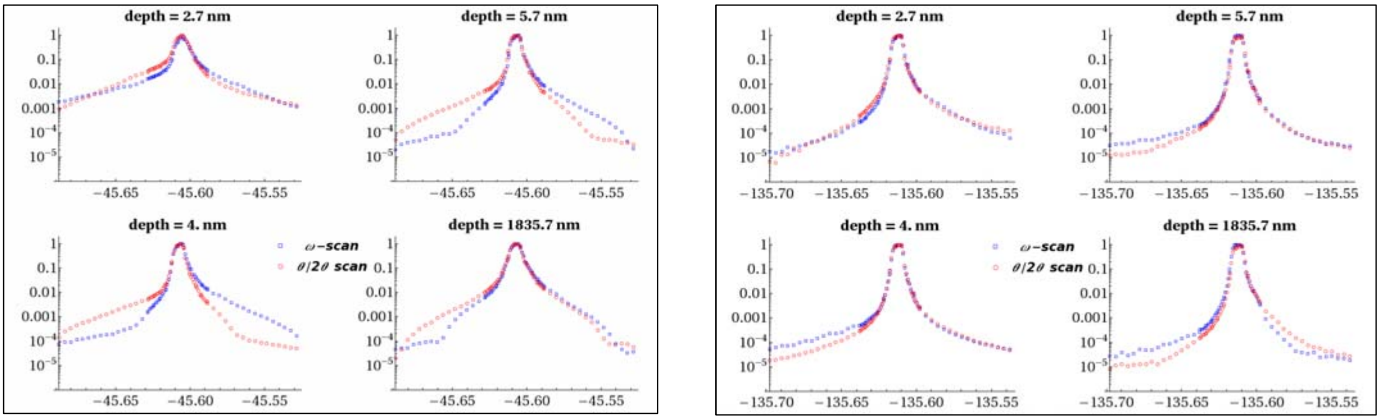
Figure 3: Fit of the pure specular data obtained for the three hot metal polished surfaces. In the insest, the fitted density profiles are presented that were extrapolated from the fits. The black curve is the theoretical reflectivity of a perfectly smooth and flat diamond.

fitting parameters		**100**	**110**	**111**
1st layer	*ρ_e [Å^{-3}]*	0.02	0.04	0.35
	thickness [Å]	36	54	76
	roughness [Å]	0.1	13	15
2nd layer	*ρ_e [Å^{-3}]*	0.81	0.96	1.83
	thickness [Å]	40	35	9
	roughness [Å]	3	29	9
substrate	*ρ_e [Å^{-3}]*	1.08	1.08	1.08
	roughness [Å]	56	73	23

Table 2: preliminary fit results for the three plates

<u>Grazing incidence diffraction results</u>

The analysis of the GID data is still in progress. The available man-power had to be concentrated on the upper problems. In the moment we only may show experimental rocking curves together with the theoretical penetration depths (figure 3).



a) *b)*

Figure 3: Some experimental GID results for the 100-oriented plate, scaife polished surface, a) 220-reflection and b) -220-reflection.

Reference

[1] R. Klünder, F. Masiello, P. van Vaerenbergh, J Härtwig, *Measurement of the spatial coherence of synchrotron beams using the Talbot effect*, Phys. Status Solidi A **206**, 1842–1845 (2009)