ESRF	**Experiment title:** Characterization of the sub-surfaces of diamond substrates and CVD grown doped epitaxial diamond layers using reciprocal space mapping at grazing incidence.	**Experiment number**: HC2280
Beamline: ID01	**Date of experiment**: from: 26/04/2016 to: 28/04/2016	**Date of report**: 10/09/2016
Shifts: 9	**Local contact(s)**: Tao ZHOU	*Received at ESRF:*

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Report:

The ID01 beam was prealigned during the weekend before the experiment, courtesy of the beamline staff, which allowed us to start the measurements directly on the first day. With the 9 shifts that were allocated, we then managed to measure 7 samples.

This was a first experiment intended to explore whether microbeam grazing incidence diffraction could yield useful information on sample surfaces prepared within the Horizon 2020 Green Diamond project to produce ' all diamond' power transistors [1]. The results obtained exceeded our expectations as regards sensitivity of the method. So far, data from 3 of the 7 samples measured have been thoroughly analyzed, with the analysis of the remaining four now underway based on the data reduction methods developed for the first three samples. For this report, we only present reults from one of the samples, identified as GD01-1D. A unique feature of this sample amongst the seven measured is that separate surface areas underwent different plasma etching processes delimited by masking (Fig. 1 left). This sample enables us to directly compare the effect of the type and depths of the surface treatments on the same substrate. This is important as we are aware of variability in the bulk lattice quality of the different sample substrates. As a result, the longest time measuring was devoted to sample GD01-1D.

Two types of measurements were performed:

- standard grazing incidence diffraction, varying the beam incidence angle,
- zero incidence diffraction at the Bragg peak angle, with the beam ‘edge on’ to the sample, and scanning down the sample edge, i.e. at different depths to the sample surface.

Our goal was to analyze the extent of lattice damage depth caused by the abrasive polishing techniques used to planarize-polish the sample surfaces, and determibne how effective each plasma etching process was at subsequently removing this damage and the etch-depth dependence. This was not just for the different processes applied to sample GD01-1D but for different processes applied to the other six samples by various Green Diamond consortium members. The standard grazing incidence diffraction method provides a straightforward solution to the above problem, by enabling the penetration depth of the X-ray beam to vary depending on the incidence angle: the penetration depth was lowest (2.6 nm) at incidence angle 0.05°, and 68µm at incidence angle 6° (for the 8 keV beam).

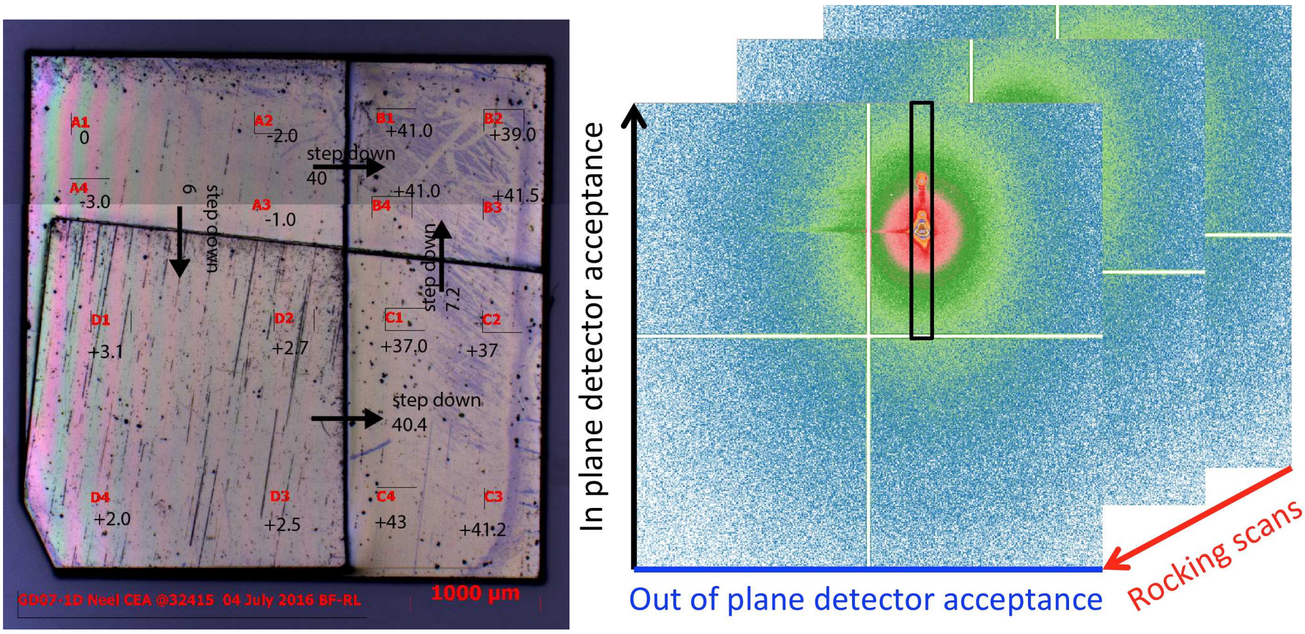


Fig. 1 : (left) optical microscopy image showing the four masked zones that underwent different etching processes and etch depths at CEA Saclay. (right) Stacks of 2D images acquired during a rocking scan that are used to reconstruct the in-plane intensity map, exploiting the large acceptance of the 2D pixel detector at ID01.

Rocking scans (Fig. 1 right) were performed for each beam incidence angle and the in-plane intensity maps reconstructed using data from the large acceptance of the 2D Maxipix detector. These maps are then used to generate line profiles of pseudo-radial and rocking scans to analyze strain and mosaicity respectively via Gaussian fits. Grains with slightly different orientations were often observed, so line profiles were generated separately for each grain before the fitting procedure. The whole process was executed with a Python program and using the Pygtk GUI (Fig. 2).

For the zone designated A on Fig. 1 for instance, the FWHMs for rocking and radial scans, as a function of the incidence angle and grain numbers (1, 2, 3) are shown in Table 1. This data is also plotted in Fig. 3 independently for each grain.

A number of conclusions can be drawn.

(1) Near the surface (i.e. for a 1/e depth < 10nm), the FWHM in the radial direction is larger than that in the rocking direction. This tells us that the most important impact of the polishing process at the surface is the increase of strain rather than the increase in mosaicity. This is true for all three grains measured.

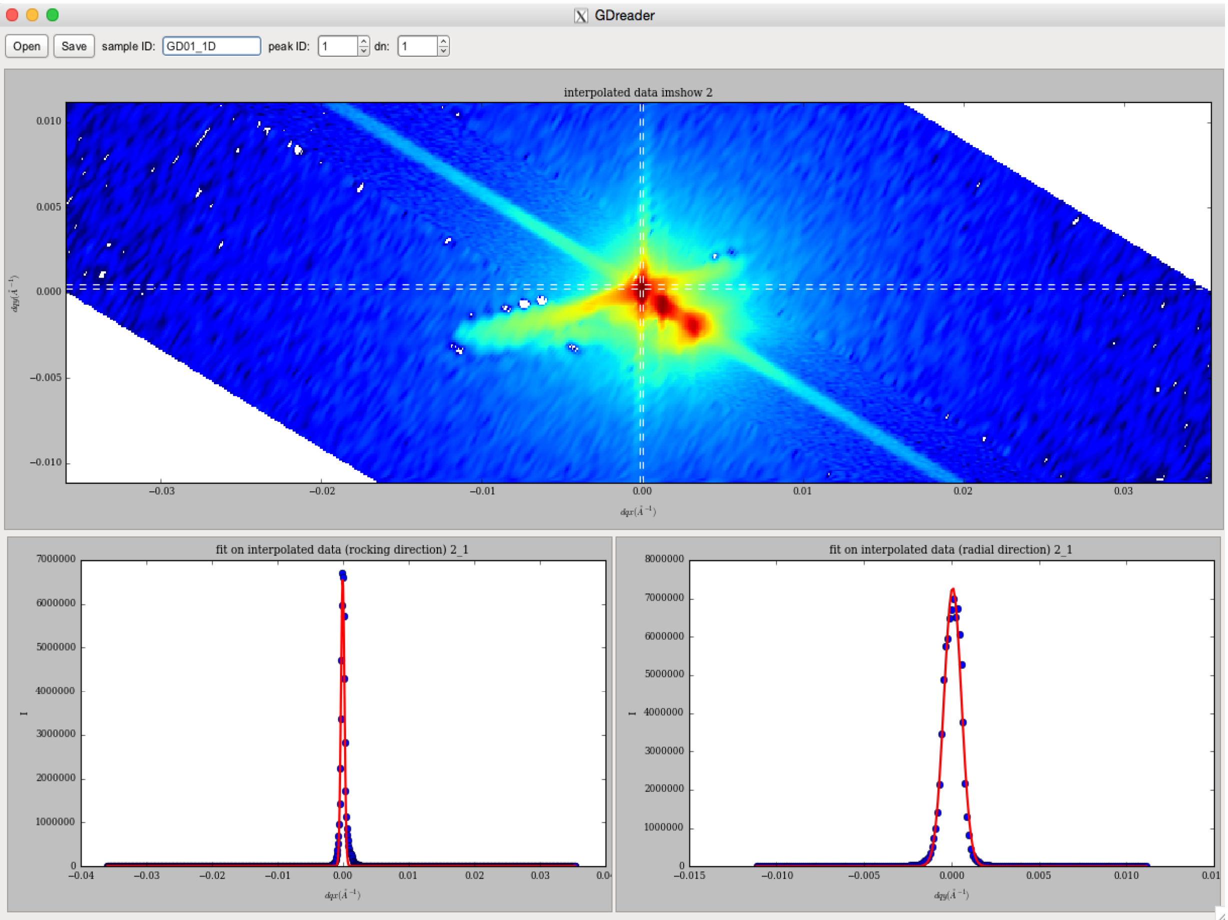


Fig. 2 : The program used to reconstruct the in-plane intensity map and to fit the extracted radial of rocking curve.

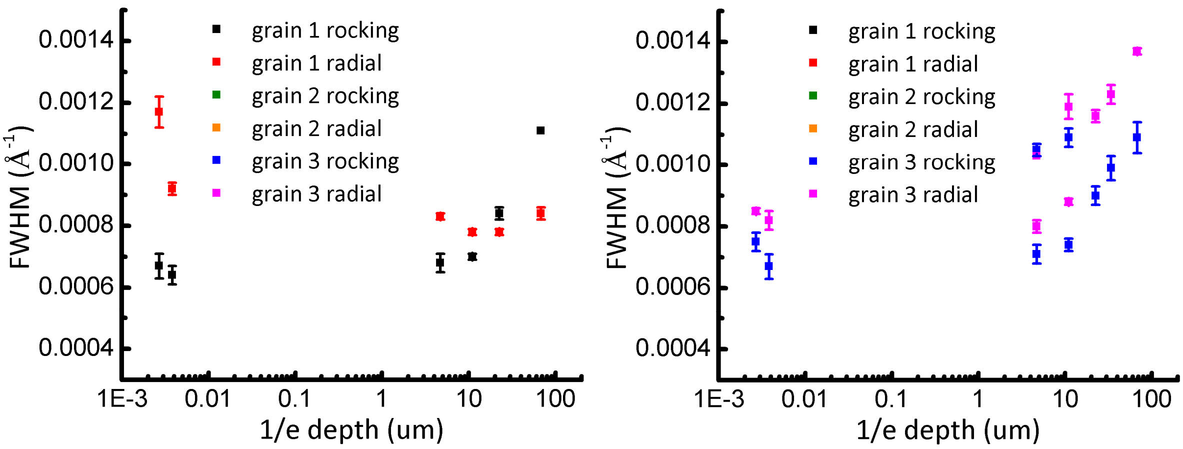
(2) For small penetration depth, the strain and mosaicity decrease with increasing depth. This is not surprising, as we anticipate that damage caused by the abrasive polishing is more severe as we approach the surface. This observation is also true for all three grains.

(3) The strain and mosaicity increase with the 1/e depth for large penetration depth. This we interpret as simply because the bulk is not a perfect crystal (we have X-ray topography images of these samples from previous measurements made at ESRF-BM05). As the penetration depth gets larger, we see a larger proportion of the bulk and hence more inhomogeneity. Unfortunately due to sample cost, these etch trials were made on low quality type 1b diamond substrates.

(4) The strain or mosaicity is almost the same for all grains between the 2nd point (1/e depth 0.0038µm) and the 3rd point of measurement (1/e depth 4.7µm). Thus, “a few microns" can be considered as the rough estimation of the depth to which the surface defects from polishing extend.

mu	0.1*	0.2	0.5	1.0	2.0*	3.0*	4.0*
1 Rocking (A^-1)	0.00067 +/- 0.00004	0.00064 +/- 0.00003	0.00068 +/- 0.00003 (R)	0.00070 +/- 0.00001 (R)	0.00084 +/- 0.00002		0.00111 +/- 0.00000
1 Radial (A^-1)	0.00117 +/- 0.00005	0.00092 +/- 0.00002	0.00083 +/- 0.00001 (R)	0.00078 +/- 0.00001 (R)	0.00078 +/- 0.00001		0.00084 +/- 0.00002
2 Rocking (A^-1)	0.00076 +/- 0.00003	0.00052 +/- 0.00002	0.00044 +/- 0.00005 (L) 0.00057 +/- 0.00001 (R)	0.00050 +/- 0.00001 (L) 0.00080 +/- 0.00002 (R)	0.00086 +/- 0.00009	~~0.00134 +/- 0.00075~~	0.00112 +/- 0.00017
2 Radial (A^-1)	0.00092 +/- 0.00002	0.00078 +/- 0.00001	0.00074 +/- 0.00001 (L) 0.00073 +/- 0.00000 (R)	0.00072 +/- 0.00002 (L) 0.00075 +/- 0.00001 (R)	0.00079 +/- 0.00001	~~0.00091 +/- 0.00002~~	0.00086 +/- 0.00001
3 Rocking (A^-1)	0.00075 +/- 0.00003	0.00067 +/- 0.00004	0.00105 +/- 0.00002 (L) 0.00071 +/- 0.00003 (R)	0.00109 +/- 0.00003 (L) 0.00074 +/- 0.00002 (R)	0.00090 +/- 0.00003	0.00099 +/- 0.00004	0.00109 +/- 0.00005
3 Radial (A^-1)	0.00085 +/- 0.00001	0.00082 +/- 0.00003	0.00103 +/- 0.00000 (L) 0.00080 +/- 0.00002 (R)	0.00119 +/- 0.00004 (L) 0.00088 +/- 0.00001 (R)	0.00116 +/- 0.00002	0.00123 +/- 0.00003	0.00137 +/- 0.00001

Table 1: the FWHM values obtained for the extracted pseudo radial and rocking scans.



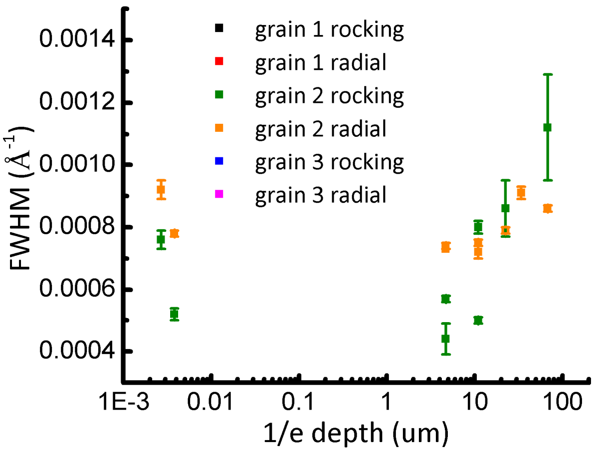


Fig. 3 : FWHMs from the extracted radial and rocking curves for grains 1, 2 and 3 as a function of the beam penetration depth (incidence angle).

(5) Grain 1 is mostly present at the surface, hence its mosaicity and strain should be constant for large 1/e depth. At the same time, its mosaicity or strain change dramatically for small 1/e depth.

(6) Grain 3 is mostly present in the bulk, hence its mosaicity or strain should be constant for small 1/e depth. Meanwhile, its mosaicity and strain increase for larger 1/e depth for the reason (non-perfect bulk) explained above.

(7) Grain 2 is present both at the surface and in the bulk.

(8) We can also deduce the da/a (strain), dΘ (mosaicity) from the values in Table 1.

The above are just the results from zone A of GD01-1D. We have also analyzed the data from zones B, C and D of this sample and were able to conclude to what extent each process performed is effective at removing the damged layer. We were able to correlate the results measured from different samples to compare the etching techiques used by different consortium members, although exact conclusions are still under discussion. Interpretation is to some extent complicated by the unwanted diffraction from defects in the bulk previously referred to (to avoid this in future measurements we will use much higher quality type IIa diamond substrates).

In summary, this first ‘exploration’ attempt has turned out to be very fruitful in demonstrating the sensitivity of the grazing incidence diffraction method and providing quantitative results that concur largely with our prior knowledge concerning the uni-directional abrasive polishing methods used on these samples. For the future, we intend to use grazing incidence diffraction as a standard tool to characterize the damage caused by the polishing process and the effectiveness of our different surface treatment process. This will be necessary both for qualifying the substrate preparation and defects that may arise in the thick (~30µm) CVD overgrown doped diamond layers that are now being produced within the Green Diamond project.