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Report:

The interaction of oxygen with diamond is important both during diamond growth and etching. The addition of oxygen can play a positive role in the gas phase as well as in the surface chemistry during growth of diamond using chemical vapour deposition (CVD). Despite the importance of the surface chemistry of oxygen on diamond, detailed information on the structure is still lacking.

Our interest in the structure of oxygen on {001} diamond stems from our work on the etching mechanism of diamond. Etching in oxygen at high temperatures induces the formation of well-defined, square etch pits, which suggests that the crystals are etched by a layer mechanism. If the etching temperature is decreased to about 750°C, a rotation of the sides of the etch pits is observed. Since steps in the different directions have a different configuration of free bonds, this observation suggests a change in the O adsorption geometry at a temperature of about 750°C. The same can be observed on the {111} faces [1]. Here a rotation of the etch pit sides occurs at about 900°C. This again implies a temperature dependent change in the formation of carbon-oxygen complexes.

In the experiment we have used four single crystal diamond surfaces, two {100} surfaces and two {111} surfaces. The {100} surfaces were polished. AFM measurements showed polishing grooves in the [001] direction which were about 6 Å deep and 150 Å wide. To obtain a (100) surface with sufficiently large {100} terraces and to get the different carbon-oxygen complexes, the specimens were etched in our home institute, at 700°C and 900°C respectively. The two {111} specimens were different in that one specimen was naturally grown and the other was synthetically grown and cleaved. AFM measurements show that the {111} faces have large, flat terraces (width ~ 1 μm). To obtain the different carbon-oxygen complexes on the {111} surfaces, these were oxygen treated *in situ*.

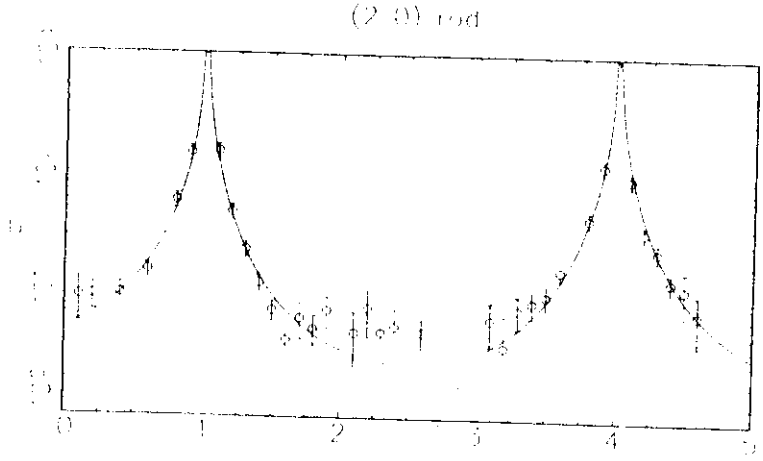

Figure 1. Structure factors along the (10) CTR for diamond {111}. The solid line gives the best fit.

The aim of the experiment was to measure the intensity along so-called crystal truncation rods (CTR's) on the different diamond surfaces. These CTR's are tails of diffuse intensity connecting the bulk Bragg peaks in the direction perpendicular to the surface and show the interference between bulk and surface structure. On the {100} samples this intensity could only be measured near the Bragg peaks, because of a fast broadening of the transverse momentum scans. This was due to large length-scale roughness induced by the polishing grooves. Unfortunately, the *ex-situ* etching of these two samples did not result in the formation of sufficiently large terraces between those grooves. Further annealing and *in-situ* oxygen etching of the {100} surfaces did not improve the surface flatness. The etching behaviour appears to be very different between high pressures (as used in the furnace in our lab) and the low pressures that we used at ID3.

The naturally grown {111} surface looked very promising with AFM. However, also on this surface the intensity could only be measured near the Bragg peaks. Although the FWHM of the transverse momentum peaks remained constant, the intensity dropped dramatically, caused by atomic scale roughness (with small lateral length scale).

Fortunately, on the cleaved {111} sample it was very well possible to measure a complete CTR, as is shown in figure 1. This could be done on the as-inserted sample (which was not etched) and also after *in situ* oxygen etching. These CTR's were measured at an angle of incidence equal to the critical angle (=0.1°) and at a wavelength of 0.72Å. Compared to previous experiments performed at ID3 [2] on a polished surface, it was much easier to obtain a good data set on the {111} cleaved diamond surface. Unfortunately there was no time left to measure enough different CTR's with their symmetry equivalents. Therefore a structure analysis is not yet possible. These preliminary results are a good starting point to investigate the structure of cleaved, oxygen terminated diamond {111} surfaces in the future.

[1] T. Evans and D.H. Sauter, Phil. Mag. 6 (1961) 429

[2] W.J. Huisman, J.F. Peters, S.A. de Vries, E. Vlieg, W.S. Wang, T.E. Derry and J. F. van der Veen, Surf. Sci. 387 (1997) 342.