ESRF	**Experiment title:** **Rocking curve imaging with extremely high strain sensitivity**	**Experiment number**: MA-1017
Beamline: BM05	**Date of experiment**: from: 10th March 2010 to: 17th March 2010 (with interuptions) from: 07th July 2010 to: 08th July 2010 from: 25th Nov 2010 to: 09th Dec 2010 (with interuptions)	**Date of report**: 10th August 2013
Shifts: 12, only 3 on proposal time, further ones as IHR	**Local contact(s)**: Tamzin Lafford (BM05)	*Received at ESRF:*

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Report:

In measurements carried out in September 2006 at ID19 we were for the first time able to demonstrate (measurements done in the last few hours of the beamtime) on one sample the existence of smaller areas with residual strains close to few 10^{-8} [1, 2] . Next we improved the necessary strategy to measure residual strains with extremely high sensitivity and high spatial resolution. This was the focus point of this proposal. Its realisation was retarded by several facts - the transfer from ID19 to BM05 (together with all other X-ray topography instruments) of the necessary key instrument, its re-installation and the necessary long comissioning, and later by the decision to tak BM05 out ot the beamtime allocation program.

As a direct result of the experiments MA-1017 at BM05 oral presentations and posters have been accepted for several conferences, like the Fourth International Workshop on Diamonds for Modern Light Sources DMLS 2011 at APS in May 2011, the DeBeers Diamond Conference 2011 in July 2011 at the Warwick University, UK, as well as French and German (abstract below) national conferences on crystal growth. To give the motivation for MA-1017, and some of the results, one of the related abstracts is given below. However, the most important results of this work is the thesis of Fabio Masiello, submitted in 2011 to the Université Joseph Fourier in Grenoble, France and successfully defended at the ESRF in May 2011 [3].

Measurement of residual strains with quantitative X-ray topography

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Due to its excellent optical, thermal and mechanical properties synthetic single-crystalline diamond is a very well suited material for several X-ray optical elements to be used in 3rd generation X-ray sources [4-7]. In the case of 4th generation X-ray sources like the XFEL in Hamburg it is probably the only possible material for some applications. Diamond is used for Bragg diffracting elements like monochromators, beam splitters or phase plates/polarisers. However, the beam quality should not be spoiled by those elements. Thus, a high perfection in the crystal bulk and very good surface quality are crucial.

In recent years, considerable progress has been made in the field of the HPHT synthesis methods (high-pressure high-temperature). This has allowed the growth of diamond crystals with linear dimensions of around 10 mm and with low nitrogen content (below 40 ppb instead of hundreds of ppm). The result is a material with extended areas ($20 mm^2$ and more) that are free of macroscopic defects like dislocations, stacking faults and inclusions. The residual strains in these areas may be on an extremely low level (smaller than 10^{-7}), fulfilling the stringent requirements on crystal quality. The sources of residual strains are long range (over millimetres!) strain fields of still existing dislocations, or local variations of the impurity concentrations. Surface scratches, which may even not be visible under an optical microscope and other imperfections at the surface, also play a role. Such low strain levels are far away from the detection limit of standard methods of X-ray diffraction. The classical measurement of the FWHM of rocking curves is far too insensitive. Even such popular X-ray topography methods like Lang topography (laboratory) or synchrotron white beam topography are not sufficiently sensitive to the levels of strain which are required to be measured, even by some orders of magnitude.

Our goal was twofold. On the one hand we wanted to push the detection limit for residual strains as far (low) as possible, and on the other hand, we wanted to obtain quantitative results with spatial resolution, based on X-ray topographs. Thus, to vary the strain sensitivity and to measure extremely low strain values, we had to use sophisticated non-dispersive double crystal diffraction topography methods (“plane” wave topography). The idea was to use a non-dispersive (n,-m)-setup with a bendable silicon monochromator, combined with high-order reflections and to also use rather high X-ray energies. In this way narrow rocking curves with extremely steep flanks could be obtained. This results in extreme strain sensitivity when using the steepest part of the flank. **We were able to achieve detection limits even down to 10^{-9}** (see figure 1 below)**. Quantitative 2D-analysis of local strain was possible with two different experimental methods**. We shall demonstrate this based on results obtained from diamond plates purchased from Element Six.

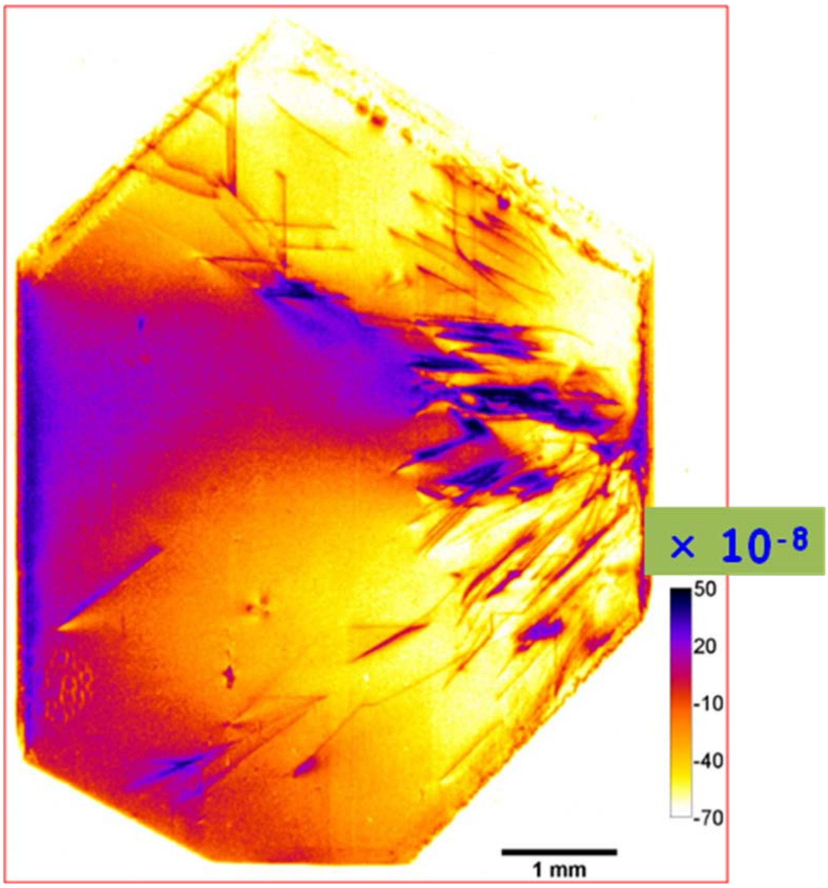

Figure 1

Example for highly sensitive quantitative X-ray topography.
This is an effective misorientation (strain) map of a 110-oriented diamond plate. Used energy 20keV, reflections Si [880] C* [660].
The used instrument is described in [8]. For the present configuration the detection limit (smallest detectable effective misorientation or change of the local Bragg angle) was $\delta\theta > 8 \cdot 10^{-9}$.
The effective misorientation is of the order of 4×10^{-8} for a region of interest of 0.5×0.5 mm^2 and 1×10^{-7} in a region of 1×1 mm^2. This is a result similar like in 100-plates which are cit in an optimum way out of the pyramidal 100 growth sector (perpendicular to the growth direction). The 110-plates are cut parallel to the growth direction. On purpose the regions with the inhomogeneous distributions of single dislocations were not cut of (needed for crystal holding). This results in its slight bending.
It is worth mentioning, that it is impossible to measure such weak local strains with the classical rocking curve analysis and FWHM-determination.

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