ESRF	**Experiment title: Investigation of defect creation/ annihilation in Si semiconductor crystals subjected to uniform stress at high temperatures (part II)**	**Experiment number:** **HC 225**
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Report:

The problem of material response to internal/external stress at higher temperatures is important for silicon device technology and application (sensors) in microelectronics. Annealing Oxygen-containing Czochralski grown silicon single crystals (Cz-Si) under **enhanced hydrostatic pressure (HP) can** influence the process of Oxygen precipitation. Whereas defect creation and transformation during annealing at normal pressure has been carefully investigated (especially in the case of Cz-Si), not much is known about the same processes under high pressure (HP). The aim of the common investigation with the Institute of Electron Technology and with the Institute of physics of the Polish Academy of Sciences in Warsaw was to study the following HP **induced effects:**

a) Oxygen precipitation in **as-grown** Cz-Si under HP up to 10^9 Pa.

b) Oxygen precipitation in Cz-Si samples with **nucleation centers** for oxygen precipitation (created at 720 K to 1000 K) during annealing at 1230 K to 1400 K at up to 10^9 Pa.

In this work this phenomenon was investigated by a wide set of experimental methods, like Fourier Transform Infrared Spectroscopy (FTIR), selective chemical etching, electrical methods, in connection with synchrotrons radiation X-ray topography and diffractometry (reciprocal space mapping). The basic intention of this part of experiments was to check the potential of **reciprocal space mapping with a synchrotrons radiation source in** comparison with that using a classical one, and of course with X-ray topography and the other methods. This method may supply information (size, concentration, geometry) in a nondestructive way about smaller (micrometer and sub-micrometer range) defects in larger sample volumina and especially about defects which are smaller than that detected by X-ray topography. Thus, it gives complementary information to TEM, to chemical methods, and to X-ray topography.

The measurements were planned as a test to see, if the results of reciprocal space mapping, done at the ESRF using higher energies in the transmission (Laue) case and investigating (and averaging over) the sample in its whole depth, are different to those using lower energies in the Bragg case and investigating only a shallow surface layer like in the case of using conventional equipment. It appeared that it was possible to improve the sensitivity and resolution for detecting small crystalline defects. As demonstrated in Figure 1, the ESRF reciprocal space maps contain more details. They show an asymmetry which may provide information about the local deformation near the defects. In some cases, rather strong additional diffuse scattered intensity (like the two "lines" with an angle of 40° with respect to the horizontal axis) appear, which give hints about the character of the defects (e.g. stacking faults in 111 planes). This shows that it is possible to gain information about local deformations and the defect geometry in a non-destructive way.

The obtained results were related to those of other methods to study the above mentioned HP effects. Some examples for conclusions about high pressure induced effects are the following:

In the above mentioned case a) for the Cz-Si sample with an initial concentration of oxygen in interstitial positions of 1.1. 10^{18}cm^{-3}, this concentration diminished to 8.21017 cm^{-3} after annealing for 5 hours at 1230 K at atmospheric pressure (10^5 Pa), whereas after the same annealing but with HP (10^9 Pa) the concentration was much lower (3.4 10^{17}cm^{-3}, more precipitates).

In the case b) for example, the sample preannealed for 5 hours at 1000 K and 105 Pa to create nucleation centers for oxygen precipitation and later annealed for 5 hours at 1400 K and 105 Pa to cause oxygen precipitation, showed an oxygen concentration equal to 5.61017 cm^{-3}, whereas after the same preannealing but at 10^8 Pa and the same final annealing, the oxygen concentration still remaining in the form of interstitial was below 41017 cm^{-3}.

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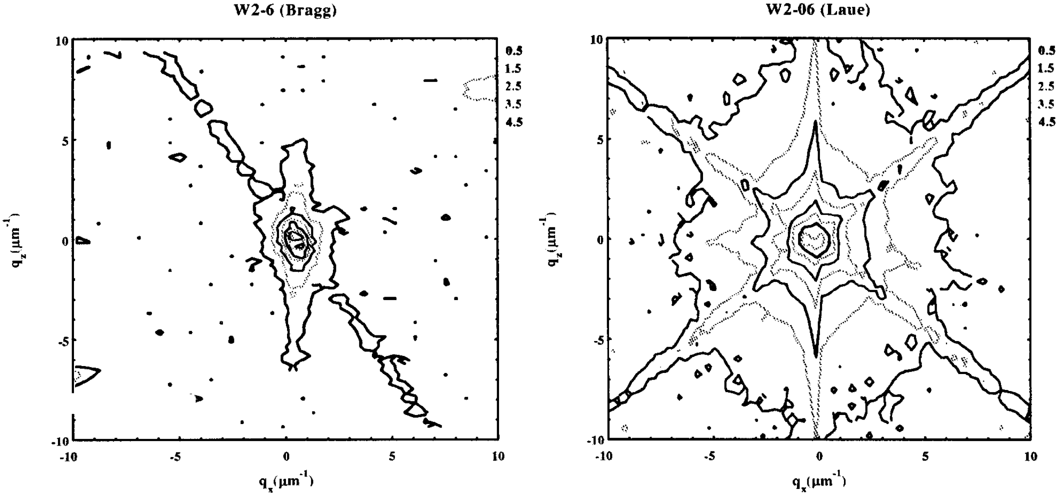

Figure 1 : Reciprocal space maps of a Cz-Si sample, initially defect free, with an initial Oxygen concentration of 1018 cm^{-3} , annealed 5 hours at 1400 K and at a pressure of 109 Pa.
Left: measured in the symmetrical Bragg case (004 reflection) on a Philips MRD at E = 8.048 keV (with a visible "analyser streak", inclined with about 34° with respect to the vertical axis);
right: measured in the symmetrical Laue case (220 reflection) on ESRF beamline BM5 at 15 keV (an "analyser streak", inclined with about 12.4° with respect to the horizontal axis, is nearly invisible; "monochromator streaks" are absent in both cases).