ESRF	**Experiment title:** **Investigation of defect creation in the system Si-SiO_2 with missfitting particles in the matrix under the influence of quasi-hydrostatic pressures up to 10 GPa**	**Experiment number:** **HS-68**
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Report: The following Czochralski grown silicon (Cz-Si) samples, some with preliminarily created oxygen-related defects, were investigated:

A. Cz-Si samples with an initial oxygen concentration c_o above $1 \cdot 10^{18}$ cm", in the form of thin foils of about 50 μm thickness and of similar other dimensions, subjected to preanueling:

a) samples (Sl) preannealed at 725°C for 20 h , containing small defects (mostly oxygen clusters) with a total density of $1.3 \cdot 10^6\ cm^{-2}$, and

b) samples (S2) preannealed at 725°C for 20 h and at 1050°C for 20 h, what leads to the creation of larger oxygen-related defects (oxygen precipitates and stacking faults) with a total density of about $3 \cdot 10^6\ cm^{-2}$.

B. As-grown Cz-Si samples with c, equal to $8 \cdot 10^{17}\ cm^{-3}$.

C. Cz-Si samples with different initial c_o, subjected to different preannealiug procedures (at 600 C - 1127°C) to create oxygen-related defects of different kind and orientation. The dimensions of the samples B and C were about 5 x 5 mm^2 and 10 x 10 mm^2, respectively, with a thickness of about 600 pm.

The samples A were pressurized in a Diamond Anvil Cell (DAC) in a H_20 - methanol mixture at 85 - 105 kbar at room temperature for 20 - 36 h (treatments I), samples B in a Bridgman press in a BN medium at 57 kbar at 800°C for 3 h (treatment II) and the samples C in a pressure furnace under Ar atmosphere at up to 10 kbar at up to 1127°C for up to 10 h (treatments III).

To investigate changes in the defect structure experiments were carried out at the Topography Beamline (ID19) of the ESRF. White beam topography was used for the samples A and B, and double crystal topography for the samples C.

Results: Samples A: The topographs of these samples showed elongated, very weak Laue spots. This is a sign of asterism which has its origin in the transformation of the single crystals to crystals with crystal grains. From this effect it is possible to estimate the mosaic spread of the single grains and the orientational distributions of the grains. It appears that these mosaic spreads are in the order of 2 to 8 arc minutes for the individual grams and of 12 to 30 arc minutes for the grain distribution (see table 1). For these samples the following kinds and densities of defects (in cm^{-2}) in the samples before the DAC-treatment were determined by selective etching in a Yang solution:

sample Sl: stacking faults, 4.10^2, small oxygen clusters, $1.3.10^6$
sample S2: stacking faults, 3.10^6, large oxygen precipitates, $2.1.10^4$
In the case of the samples Sl (with smaller oxygen clusters) a strong fragmentation after the DAC treatment at 105 kbar (the treatment at 85 kbar caused minor changes, see table 1) was observed. In the case of the sample with large oxygen-related defects (S2) such fragmentation was observed just after the treatment at 90 kbar. This can be considered as some kind of proof that at sufficiently high pressure larger oxygen-related defects can cause a massive creation of additional defects, and consequently, the fragmentation of the crystal. However, because of the small dimensions of the samples (in the order of 50 μm) and the limited resolution of the X-ray topographic method (in the order of some micrometers) images of individual dislocation were not resolved and the above mentioned supposition has to be considered as preliminary.
Samples B: In this case the single crystals were destroyed nearly completely and transformed to a polycrystal with small grains and grain orientation distributions of some tens of degrees (an evidence of complete fragmentation).
Samples C: The double crystal topographic results showed that these samples C remained to be of high crystalline quality after the high pressure - high temperature treatment. These samples were investigated to use properly the allocated time after ascertaining that the investigation of the samples A and B can not give conclusive results.

DAC treatment	individual grain	grain distribution	comments
85 kbar, 36 h	2	12	sample with small oxygen clusters (Sl)
105 kbar, 20 h	4	25	
105 kbar, 20 h	6-8	30	
90 kbar, 36 h	4-6	20-25	sample with large oxygen precipitates (S2)
90 kbar, 36 h	2-6	30	

Table 1: Estimation of the mosaic spread half widths within individual grains and those of grains within the whole crystal (in arc minutes)

Conclusions:
Samples A: A misfit dislocation network was not directly proven to be created in the samples A, because of the too small dimension of the samples (about 50 μm) in comparison with the resolution of the used X-ray method (in the order of a micrometer), and also because of problems with the mounting of those very small samples.
Samples B: The single-crystallinity was completely destroyed. A further elaboration / interpretation of the observed X-ray patterns is needed.
Samples C: The high pressure treatment influenced the oxygen precipitation and thus, the strain fields in the Cz-Si bulk, as it was seen on double crystal topographs.

The results obtained in the HS-68 ESRF experiment were included into the abstracts proposed to be presented at the following conferences:

1. A. Misiuk, J. Bak-Misiuk, J. Härtwig, J. Jung, B. Surma,
Reciprocal lattice mapping of pressure - annealed czochralski grown silicon,
XVII Conference on Applied Crystallography, Wisla, Poland, 31.08 - 04.09.1997

2. A. Misiuk, J. Härtwig, J. Bak-Misiuk, M. Tkacz,
Investigation of defect creation in the Si - SiO_2 system at pressures up to 11 Gpa,
4-th Polish Symposium of Users of Synchrotron Radiation, Cracow. Poland. 18.06. - 19.06.1997