ESRF	**Experiment title:** 3D Characterization of Dislocation Networks by Diffraction Laminography: Spatio-temporal Evolution and Extension to High-absorbing Materials	**Experiment number**: MA-3236
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Shifts: 15	**Local contact(s)**: Lukas Helfen	

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Report:

In the proposal for the experiment MA-3236 at ID19 we described two main objectives, both regarding a further development of X-ray diffraction laminography (XDL), enabling 3D imaging of dislocations and other defects in large crystalline samples. In particular we aimed on:

A) Studying the thermally driven nucleation and emergence of slip bands in almost perfect crystal silicon wafers by alternating annealing cycles and XDL scans in order to extend the before static method to the characterization of temporally evolving structures.
B) Extending the applicability of XDL to high-Z materials, i.e. to the cases in which the absorption of X-rays cannot be neglected compared to the diffracted signals.

In the following, we briefly summarize the experiment and our achieved progress during the first preliminary analysis of the acquired data:

A) We were able to optimize the measurement procedure of XDL towards sufficiently fast alignment and projection acquisition, in this way enabling us to follow the evolution of dislocation networks during a gradual, step-wise annealing of damaged Si wafers.
For the above mentioned step-wise annealing procedure we brought a mirror oven along (controlled by SPEC) which enabled, together with a thermocouple, a systematic and monitored thermal treatment of the Si samples. In order to make efficient use of beamtime, we always measured and heated two samples at the same time, alternatingly. To achieve a suitable evolution of dislocation networks (on the one hand enough development per step to cover in total a reasonable interval of the investigated process, but on the other hand sufficiently small steps to still allow a correlation of subsequent snapshots), the mechanically indented regions of the wafers were heated up to a temperature of about 850 °C for at least 5 and maximum15 s in each step. Exemplarily, Fig. 1 a)-d) show topographic projections from a series of XDL scans (view angle always $\varphi \approx 90°$, directly recorded after four subsequent annealing steps) following the formation of a complex dislocation network. Fig. 1 e)-h) show a subset of projections of the annealing state d), now covering different

view angles (φ ≈ 25°, 65°, 105°, 145°). Note the sharp and narrow contrast of the dislocation lines, which could be achieved by the careful adjustment of weak-beam contrast conditions, for which the topographic images are taken at a few thousandth of a degree deviation from Bragg condition of the (ideally) undistorted crystal matrix. During the forthcoming analysis of the data, the 3D arrangement at the different time steps will be reconstructed and the movement as well as the interaction of the individual dislocations inside the network will be analyzed in detail.

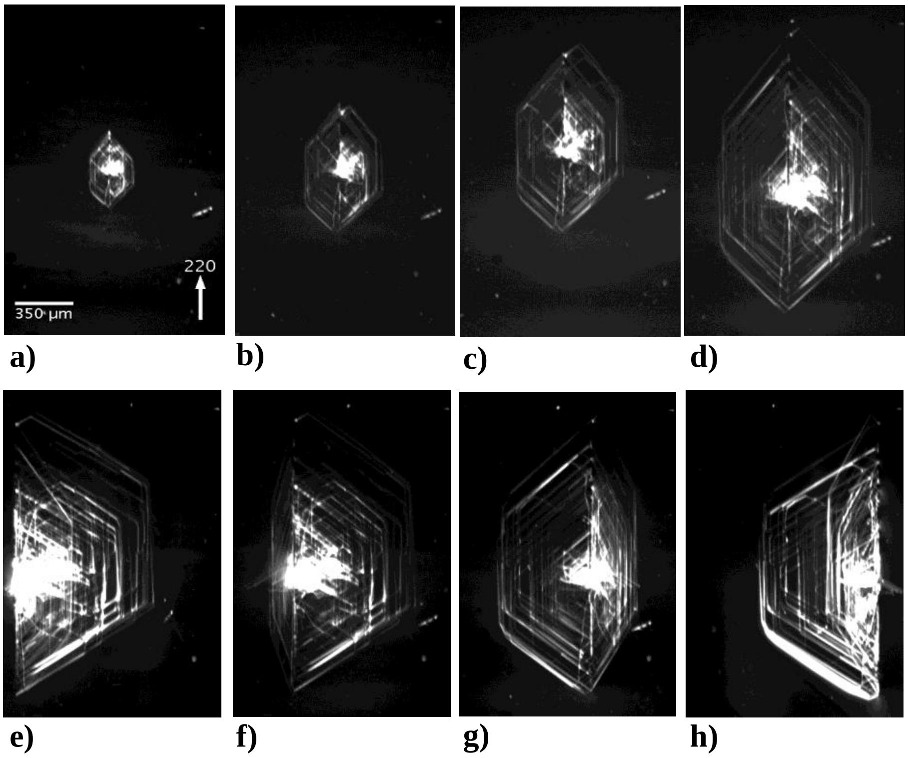

Fig. 1 a) – d): Four projections (φ ≈ 90°) of the same indent in a silicon wafer, acquired during XDL scans after subsequent annealing steps.
e) – h): Four projections of d), but from different view angles : φ ≈ 25°, 65°, 105°, 145°.

In total, we are highly satisfied with the quality of the obtained data for the performed systematic study of thermally treated Si wafers. We are confident that the data will be highly suited for our planned analysis.

B) Regarding the second objective of the proposal, we successfully applied the established XDL alignment and measurement procedure on a selected 2 x 2 mm² region of interest of a 610 µm thick GaAs wafer. This shows the general feasibility of 3D defect imaging by XDL for the characterization of dislocation networks within the bulk of large and at the same time highly absorbing crystals. The achievable quality of 3D reconstructions from such XDL data sets will be carefully evaluated during our forthcoming analysis.
The growth of nearly defect-free GaAs, for example, still remains a challenge and the typical cellular structure of very dense intrinsic dislocation networks (originating from processes during crystal growth from the melt) is clearly visible in the recorded XDL projections with a pixel size of about 1 µm (see Fig. 2).

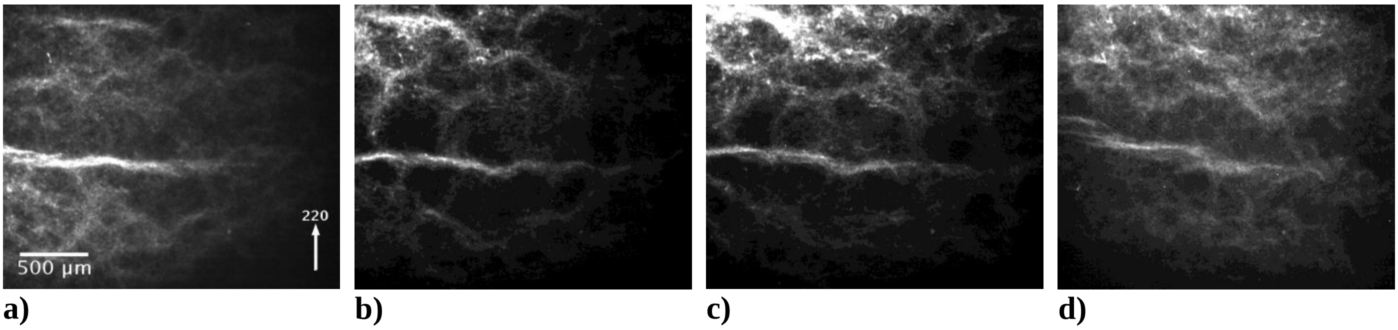

Fig. 2 a) – d): Four topographic XDL projections (view angles: φ ≈ 33°, 72°, 111°, 150°) of a selected region of interest on a LEC GaAs wafer with 38 mm diameter. The typical cellular dislocation structure of this material is clearly visible in all projections.

However, it has to be noted that even for the best compromise between photon flux and exposure time we could achieve at ID19 (60 keV and still acceptable 3 hours of overall scan duration), the SNR turned out not to be satisfying or is at least at the absolute lower limit for analysis by 3D reconstruction. For future XDL measurements for systematic studies of high Z-materials we thus suggest the use of X-rays with an energy of at least 80 keV and with at least the same flux, like available e.g. at ID15A.