ESRF	**Experiment title: 4D Imaging of GaAs Wafers by X-ray Diffraction Laminography: Dislocation Cell Structures and Their Role During Slip Band Formation**	**Experiment number**: MA/3930
Beamline: ID15A	**Date of experiment**: from: 06/06/2018 to: 10/06/2018	**Date of report**: 03.02.2020
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Report:

1) Preparations and Setup:

For X-ray Diffraction Laminography (XDL) especially at high energies (> 60 keV) a very accurate sample manipulation system is needed: the angular error must not exceed 10^{-4} degree and the spatial accuracy should be in order of a few µm. Thus, we brought our own sample manipulation stack along which fulfills all the specifications (shown in Fig. 1). It consists of a precise tilt axis with special gear box, an air bearing rotation axis and a piezo driven Tripod (SpaceFab). With the kind help of beamline staff, this stack was successfully mounted in the Experimental Hutch 2 at ID15A on the available Huber base structure consisting of a lateral and vertical translation axis and a rotation motor for aligning the tilt axis of our system perpendicular to the beam-direction. Together with the high-energy imaging detector from ID15A and an Andor Neo CMOS camera mounted on an inclinable Huber stage, provided by the beamline staff, we successfully measured several samples of different types and materials with XDL. Thanks to the support of the ESRF staff, we were able to integrate all our motors into the beamline system enabling a smooth experiment workflow from a central control system session.

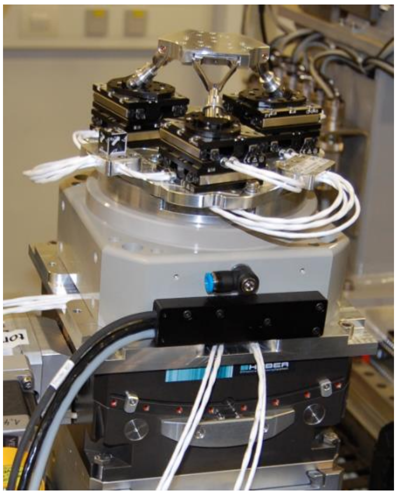

Figure 1: Mobile diffraction imaging instrumentation consisting of a tilt axis, a rotation axis and a piezo driven Tripod (SpaceFab).

At the time of the experiment, the available annealing equipment did not allow a safe annealing for quasi in-situ experiments with GaAs, due to potential As evaporation. Therefore, we focused on static XDL measurements on several industrially relevant semiconducting samples like CdTe, Ge and GaAs of which the majority was pre-annealed in our laboratories at the Karlsruhe Institute of Technology (KIT).

The source–sample distance of about 57 m at ID15A (EH2) is comparatively short for a full field diffraction imaging technique like XDL, which, together with the horizontally diffracting monochromator, leads to locally varying image contrast. Therefore we adapted our measurement principle for some scans by taking several images for the same azimuthal rotation angle at different rocking angles (Multi Azimuth Rocking Curve

Imaging, MARCI) and introduced so-called “virtual weak beam” data analysis in order to obtain the same image quality as we observed at long (>100 m) imaging beamlines like ID19. In order to narrow the bandwidth of the Si(111) monochromator as much as possible we unbent its crystals completely.

2) Measurements

XDL was developed for 3D imaging of dislocations in crystalline materials and successfully applied to low absorbing materials like silicon and diamond, but until now has never been used to characterize dislocations in strongly absorbing materials at high energy beamlines like ID15A. We started with a beam energy of 68.5 keV and scanned two regions (approx. 2x2 mm²) on an indented and pre-annealed 400 µm thick liquid encapsulated Czochralski (LEC) grown GaAs sample. Fig. 2 i) exemplarily shows three XDL projections from different view angles in which the cellular structured dislocation patterns, typical of LEC grown GaAs, as well as a dislocation rosette around the indent position are visible. The XDL-projections are taken at a deviation from the Bragg peak of -0.002° (weak-beam contrast). With the same X-ray energy a 500 µm thick former Medipix sensor was scanned subsequently. Whereas short-range strain fields at the pixel bonds are visible in the weak-beam projection (Figure 2 ii) left), the integrated intensity projection (generated from MARCI data) clearly captures two distinct small angle grain boundaries (Figure 2 ii) right). Furthermore, we scanned one single dislocation in the bulk of a 700 µm thick Ge sample, before we increased the energy to 120 keV for investigation of an 800 µm thick CdTe sample. Figure 2 iii) shows an integrated intensity XDL-projection of the CdTe-scan. Again, several small angle grain boundaries are very well visible.

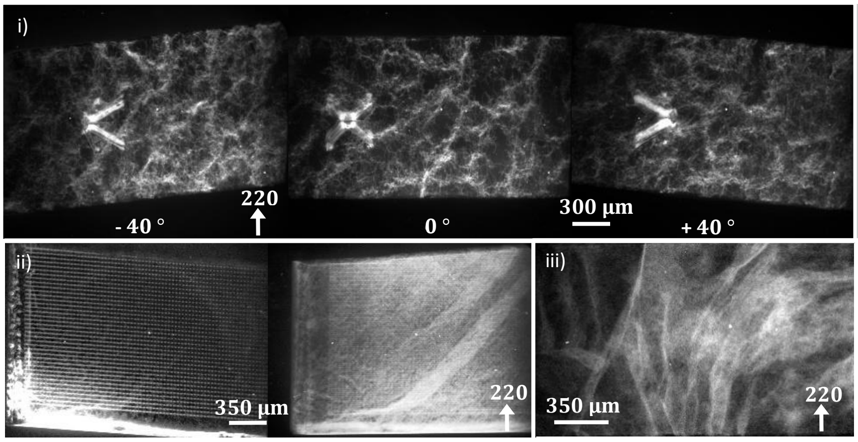

Figure 2: Selected XDL-projections exemplarily taken from full XDL scans of
i): an indented and annealed 400 µm thick LEC grown GaAs wafer from 3 different view angles captured in weak-beam contrast conditions,
ii): a former 500 µm thick GaAs sensor of a Medipix detector with weak-beam (left) and integrated intensity contrast (right) and
iii): a 800 µm thick CdTe sample captured with integrated intensity contrast.

3) Data Quality and Results

As described in section 2), experiment MA/3930 was the first time XDL was applied to high-Z materials (GaAs, Ge and CdTe) with energies > 60 keV. Theoretically, we expected to reach image resolution of about 10 µm. In fact, the image quality turned out to be excellent and we reached resolutions considerably better than 10 µm. The digitally zoomed area in Figure 3, right, (corresponding to the central region of Fig. 2 i) at 0° rotation) exemplarily shows the high image quality, allowing to resolve even single dislocations within the growth related cellular dislocation.

By combining the capabilities of MARCI and XDL, regarding different but complementary contrast mechanisms (weak-beam and integrated intensity contrast) we succeeded to measure single defect features, such as dislocations, within several crystal grains at the same time.

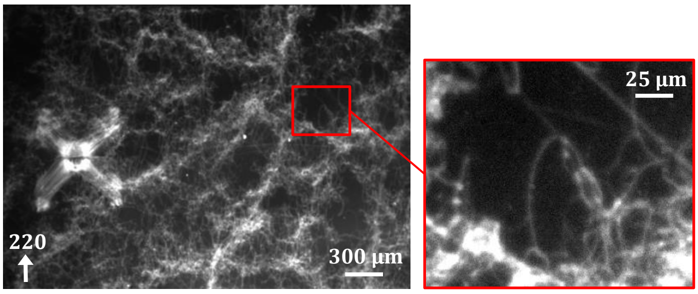

Figure 3: Same XDL projection as in the center of Figure 2 i). The digitally zoomed area shows the high image quality, even single dislocations could be distinguished with a spatial resolution of a few µm.

We conclude that the experiment was very successful, providing unprecedented insights into technologically important crystalline materials like GaAs, Ge and CdTe on the length scale between micrometers and millimeters. The acquired data provides useful information and results that will be published in peer-reviewed journals in the near future. Support and contributions of ESRF staff will be acknowledged accordingly.