ESRF	**Experiment title: Quasi *in situ* 3D Investigation of Glide Prism Formation in GaAs Wafers by Means of X-ray Diffraction Laminography**	**Experiment number**: MA-4772
Beamline: ID15A	**Date of experiment**: from: 22.09.2021 to: 27.09.2021	**Date of report**: 18.10.2022
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Names and and affiliations of applicants (* indicates experimentalists):

E. Hamann[1*], D. Hänschke[1*], M. Kabukcuoglu[1,2*], M. Hurst[1*], A. Danilewsky[2], and T. Baumbach[1,3]

[1] Institute for Photo Science and Synchrotron Radiation (IPS), Karlsruhe Institute of Technology (KIT), 76344 Eggenstein-Leopoldshafen, Germany

[2] Crystallography, University of Freiburg, 79104 Freiburg, Germany

[3] Laboratory for Applications of Synchrotron Radiation (LAS), Karlsruhe Institute of Technology (KIT), 76131 Karlsruhe, Germany

Report:

1) Preperations and Setup Implementation

We brought our mobile and flexible setup for X-ray diffraction imaging to ID15A because X-ray diffraction laminography (XDL) and multi-azimuth rocking curve imaging (MARCI) requires at the same time high angular and spatial precision, which in combonation are typically not provided by tomography set-ups. Our dedicated setup, which includes a precise tilt stage, an air-bearing rotation axis, and a parallel kinematics, was successfully mounted in experimental hutch 2 on the lateral translation axis provided by the beamline equipment for aligning our set up perpendicular to the beam direction with the help of the beamline stuff. In combination with our pco.edge camera , the high-energy detector of ID15A was mounted on a tilt axis to align the detector plane perpendicular to the diffracted X-ray beam; a lower-resolution large area flatpanel detector (also brought by us) was used to capture Laue patterns in order to determine and adjust the sample orientation see Fig. 1-(a). During the experiments, we used our inhouse-developed control system CONCERT to control our set-up via a dedicated workstation, while the controlling of the motors belonging to the beamline was still done via the ESRF beamline control system.

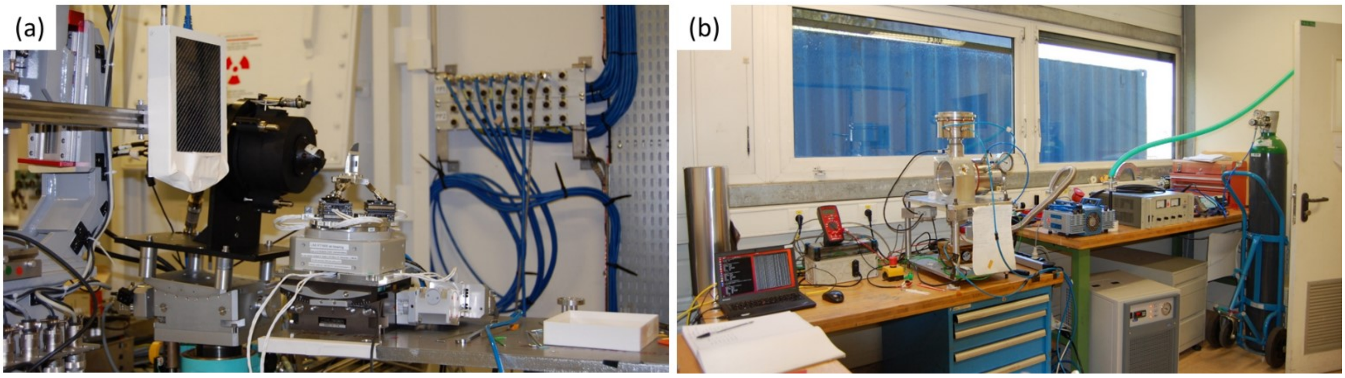

Figure 1: (a) Mobile X-ray diffraction imaging set-up consisting of a tilt axis, rotation axis, and parallel kinematics and tilted detector system mounted at ID15A experimental hutch, (b) Mirror oven heating system including oven, controller, cooing system, vacuum system, and argon tube (provided by ESRF) placed at ID15A preperation lab.

Moreover, we brought a mirror furnace system for sample treatments with a stand-alone control system that enabled us to well-controlled and safe heating cycles of gallium arsenide (GaAs) samples by monitoring the temperature via thermocouples during the thermal heatings in the argon (Ar) environment. The Ar supply was provided by ESRF. The complete heating system was placed in the ID15A preperation lab as shown in Fig. 1-(b).

2) Measurements

We started our 3D XDL scans to investigate static dislocations in two pre-characterized GaAs samples with 20x20x0.65 mm^3 volume. In order to compensate for locally varying image contrast, we combined XDL scans with MARCI by acquiring projections at the same azimuth rotation angle at different rocking angles, see the achieved image contrast in Fig. 2. This provides us with the 3D information of individual dislocations and also slip band formation, complementary to the dynamic investigation in 2D. In order to capture the dynamics of dislocations at different developed stages, two mechanically indented GaAs samples were scanned by performing quasi *in situ* XDL/MARCI-scans alternating with suitable step-wise annealings as proposed, see the evolution of dislocations in Fig. 3-(a). For using the beamtime effectively, one sample was thermally annealed for inducing dislocation mobility while the other sample was scanned to snap-shot the evolution of dislocations. Since the heating time and temperature were restricted due to safety aspects, the samples were heated several times (10 to 20 times) in between the 3D XDL scans to generate a reasonable development of dislocations. During our measurements, the beam dumped four times costing us about 2 shifts. Therefore our local contact extended our beamtime by one day and we used that time for completing our measurements, packing, and cleaning.

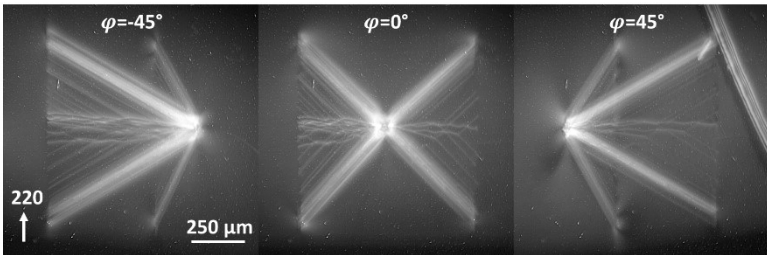

Figure 2: Snap-shots of XDL/RCI projections capturing static dislocation arrangement from different azimuth rotation angles in an indented and and gradually annealed GaAs wafer.

3) Data Quality and Results

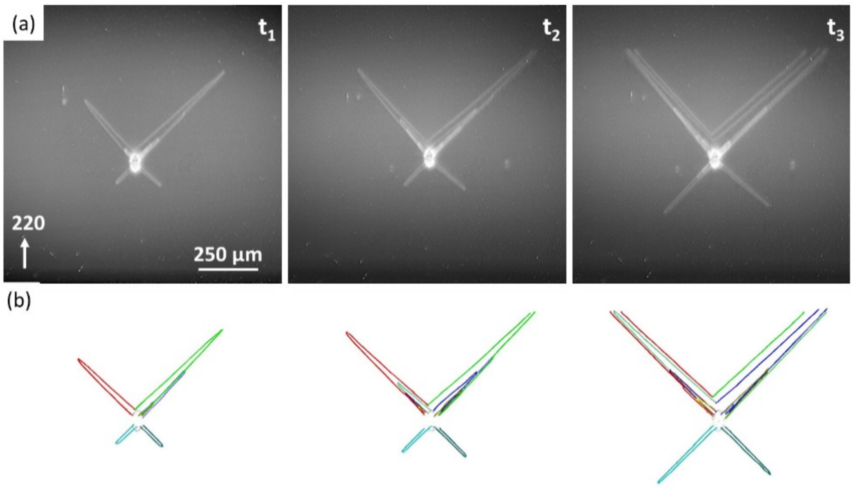

Figure 3: Three development states of dislocation arrangement and slip band formation in a GaAs wafer, characterized by quasi in situ XDL/RCI (a) snap-shots of 2D XDL projections after thermal tratments enabling 3D reconstruction. (b) Visualization of segmented 3D arrangment of dislocation arrangements. Color coding shows individual dislocations enabling to follow quantitative evolution of each single dislocation, individually.

The high quality of the acquired data fulfilled our expectations. As it is exemplarily shown in Fig. 3-(a), 2D projection data obtained from XDL scans show the development of a dislocation arrangement around the initial mechanical damage during gradual thermal treatments. We clearly see the individual dislocation lines in each evolving stage of several heating treatments between the scans ($t_1<t_2<t_3$). The image quality allows us to perform the 3D reconstruction of individual dislocation lines within the glide prisms. As a preliminary result, Fig. 3-(b) shows the reconstructed and segmented 3D volume of dislocations at the three evolving stages corresponding to the projections above. Each individual dislocation is traced in the volume and eidentified during its different evolving stages. Here, each dislocation is marked with the same color in order to follow their development.

Within this study, we conclude that the 3D quasi *in situ* XDL/MARCI experiment was successful, providing new insight into dislocation dynamics in GaAs on the large scale up to mm^3-sized sample volumes. The acquired data will give detailed information on the movement mechanisms of dislocation arrangements as well as the behavior of individual dislocations, e.g. interaction and multiplication mechanisms, and their relation to the mechano-thermally induced forces. The results could provide information for prediction, controlling, and even avoiding dislocation nucleation and propagation during industrial wafer processing, also for other III-V and II-IV zinc blende structured materials, such as InP and CdTe. The results will be published in peer-reviewed journals in the near future. Support and contributions of ESRF ID15A staff will be acknowledged accordingly.