EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:
https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal (“relevant report”)

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *“preliminary report”),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as “relevant report(s)” in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title: The 4D Evolution of Plastic Deformation in Si Wafers Studied by Correlated X-ray Diffraction Laminography and Rocking Curve Imaging**	**Experiment number**: MA-4057
Beamline: ID19	**Date of experiment**: from: June 20, 2018, 8:00 to: June 24, 2018, 8:00	**Date of report**:
Shifts: 12	**Local contact(s)**: Dr. Lukas HELFEN	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

1) Preparations and Setup:

We brought our own sample manipulator to ID19, since the combination of X-ray diffraction laminography (XDL) and rocking curve imaging (RCI) requires sample motion in specific degrees of freedom which are not provided by regular tomography setups. We successfully mounted our device onto the medium resolution tomography setup at ID19 which served as a base-structure to align our instrumentation with respect to the incoming X-ray beam. We were able to integrate all motors into the beamline control system and achieve the high level of precision in real and angular space needed for our measurements. With kind support of ID19 staff we realized a tilt of the detector (provided by ID19) in order to align the detector plane perpendicular to the diffracted X-ray beam, see Fig. 1. Furthermore, we brought a mirror furnace for the sample annealing with a stand-alone control system that enabled *in situ* monitoring of the temperature profile on the wafer during the heating treatment via termocouples and an infra-red camera.

2) Measurements:

Two silicon samples with suited damages (introduced by nano-indentations with loads of 350 to 400 mN) underwent the proposed measurement scheme: The wafers were scanned by RCI with varying azimuth angle in Bragg reflection geometry

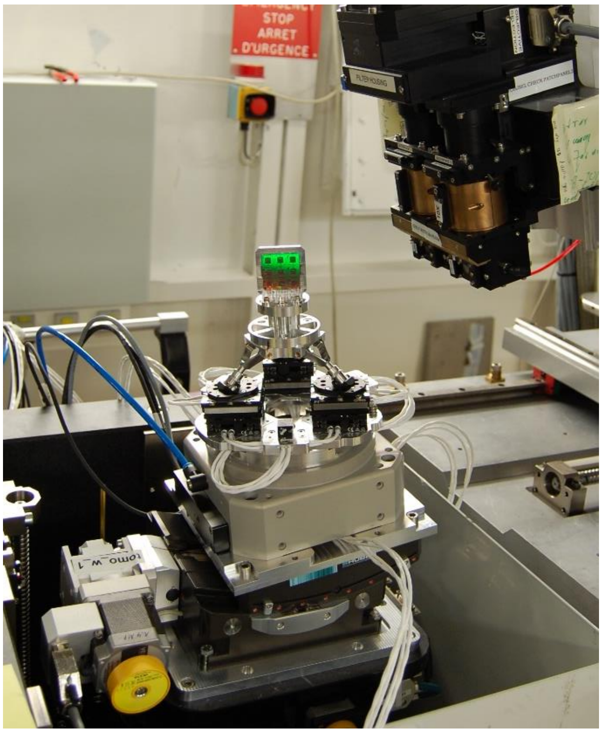

Fig. 1: Mobile X-ray diffraction imaging setup integrated in ID19 tomography setup and tilted detector system.

to capture the 3D information about the initial strain in the material around the damage sites, see Fig 2. During a gradual annealing intermediate XDL/RCI-scans were performed in Laue transmission geometry to provide snap-shots of the emerging dislocation arrangements in different development stages, see Fig 3. The pattern shown in Fig. 3 emerged around the indent marked with a red triangle in Fig 2. After completion of this first measurement campaign we continued with a complex dislocation pattern in a previously annealed silicon wafer. We also applied XDL to a silicon transistor array and a silicon carbide wafer (SiC). For the first time a so-called micro-tube in SiC was imaged with XDL.

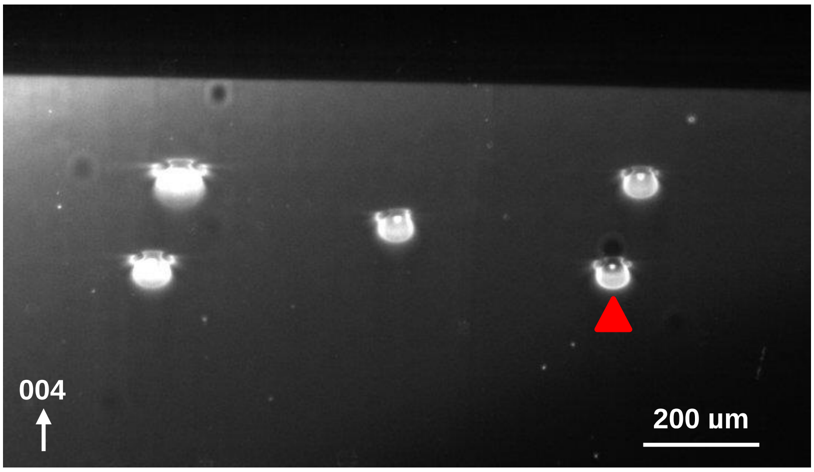

Fig. 2: Image from a rocking curve imaging scan of a silicon wafer in Bragg reflexion geometry. Around five indents in an X-like pattern initial strain in the material creates characteristic diffraction contrast. The indent marked with the red triangle is the origin of the emerging dislocation arrangement shown in Fig. 3.

3) Data Quality and Results

The high image quality of the acquired data – also owed to the outstanding beam properties at ID19 – fulfilled our expectations. Fig. 3.a) exemplarily shows projection data from XDL acquisitions of an emerging dislocation pattern during the gradual annealing. We clearly see the individual dislocation lines in each step of the successive heating treatment. The data quality also allows the 3D reconstruction of more complex dislocation arrangements. As a preliminary intermediate result Fig. 3.b) shows the reconstructed 3D crystal volume at the three development stages corresponding to the projection data above, compare Fig. 3.a). The evolution progess during one heating step could be controlled in a way that allows to identify individual dislocations in their different stages. Here, to trace the individual dislocation lines in their step-wise progression they were marked in different colors.

Screening and quality inspections of the data up to now lead to the conclusion that the quasi *in-situ* application of XDL was successful.

The acquired RCI data sets will enable the correlation of initial strain properties with the 3D structure of the dislocation arrangments and their dynamics during the heating treatment. The analysis on this topic is ongoing.

Furthermore, the extensive data sets are also suited to enable fundamental theoretical considerations, which is especially owed to the combination of XDL with RCI.

In a feasibility study we have demonstrated the applicapability of XDL to SiC. Futhermore, the obtained data may give new insight on the properties of micro-tubes in this material.

In summary, we conclude that the experiments were very sucessful and that the acquired data will provide 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.

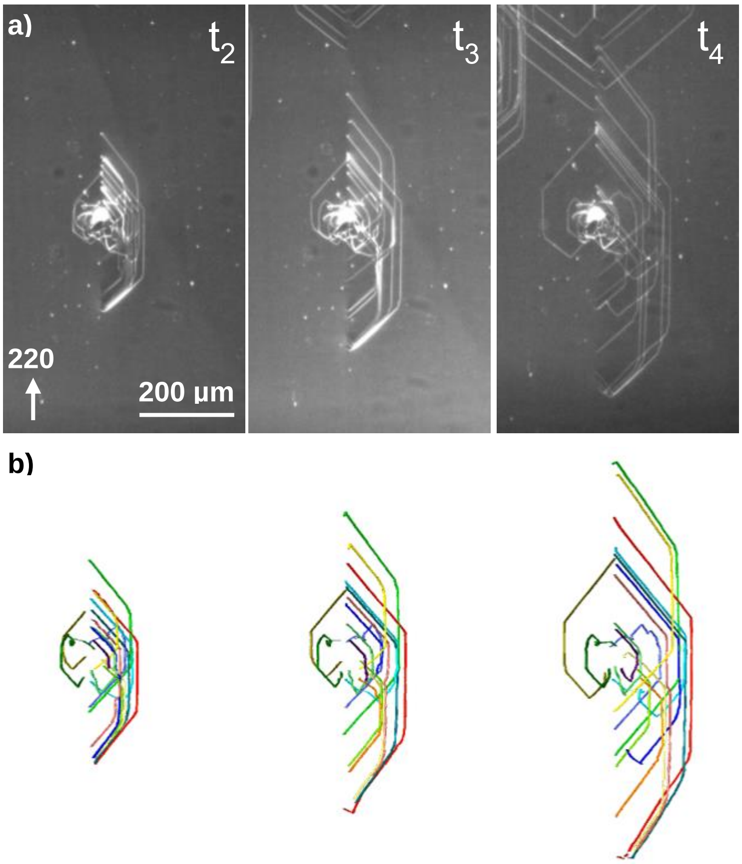

Fig. 3: a) Three exemplary projections acquired in Laue transmission geometry, each belonging to a complete snap-shot data-set of a dislocation structure originating from a single indent in the same sample as shown in Fig. 2. b) Images of the corresponding reconstructed 3D volumes, showing the individual dislocations color-coded in different states of the evolving arrangement.