EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

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.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal 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:** Slip transfer through interfaces due to local stress concentration	**Experiment number**:
Beamline: BM32	**Date of experiment**: from: 28.04.2015 to: 04.05.2015	**Date of report**:
Shifts: 18	**Local contact(s)**: Jean-Sebastien Micha	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr. Christoph Kirchlechner*

Nataliya Malyar*

Dr. Jaya Balila

Prof. Dr. Gerhard Dehm

Report:

Aim of the underlying proposal was to measure the local stress concentration at the grain-boundary by in situ µLaue diffraction in order to understand slip transfer through grain-boundaries.

The beamtime was succesful in terms of

(i) number of performed experiments
(ii) quality of performed experiments
(iii) beam stability

One problem we had during the beamtime was the limited read-out time of the CCD detector system at BM32. A faster readout would thereby allow for strainrate sensitivity measurements, which we would like to vary in the 10^{-2} – 10^{-5} s^{-1} regime.

Ad 1) we planned to deform 7 bi-crystalline micropillars and 10 single crystalline reference samples. The aforementioned amount of samples, and additional 3/5 “back-up” samples had been prepared by FIB techniques in our home laboratory and succesfully deformed.

Ad 2) Both, the initial Laue patterns (in terms of circular peak shape and residual strains) as well as the mechanical experiments (in terms of alignment and control of displacment) worked extraordinary well. After analyzing most of the data in early 2016 we are sure that we

aligned all samples better 0.2°. Also, the aimed stress state was always reached precisely (+/- 2%).

Ad 3) Compared to formed experiments the beam stability at BM32 (it´s position and size) considerable improved.

Data analysis and interpretation is still ongoing and simultaneously performed by one PhD candidate (Nataliya Malyar), one postdoctoral researcher (Dr. Anton Davydok) and one principal investigator (Dr. Christoph Kirchlechner). Even though the number of dislocations and the stresses could not simultaneously be analyzed yet, the experiments provide localized lattice **strain measurement with an resolution better 10^{-4}**. Other insights into the dislocation grain-boundary interaction were additionally obtained. For instance, we were able to **show the change of the grain-boundary angle during loading** as the simplest measure for the grain-boundary character during *in situ* loading (see Figure 1). This change strongly depends on the grain-boundary character (transmission factor) – with some grain-boundaries beeing a strong sink for dislocations.

Based on the Laue experiments it could also been shown, that the pillar geometry used world-wide suffers from considerable constraints also in the bi-crystalline case. While they are weaker in the bi-crystal (compared to the single crystals), it is clear, that the upper and lower third of the compression pillar is dominated by lateral constraints. **Only the center of the sample shows little constraints and thus, reveals the "intrinsic" material behaviour**. To our knowledge this was only be prooven by such kinds of Laue experiments yet.

We expect major results during the next month beeing published in an leading peer reviewed journal of the field (we aim for Acta Materialia).

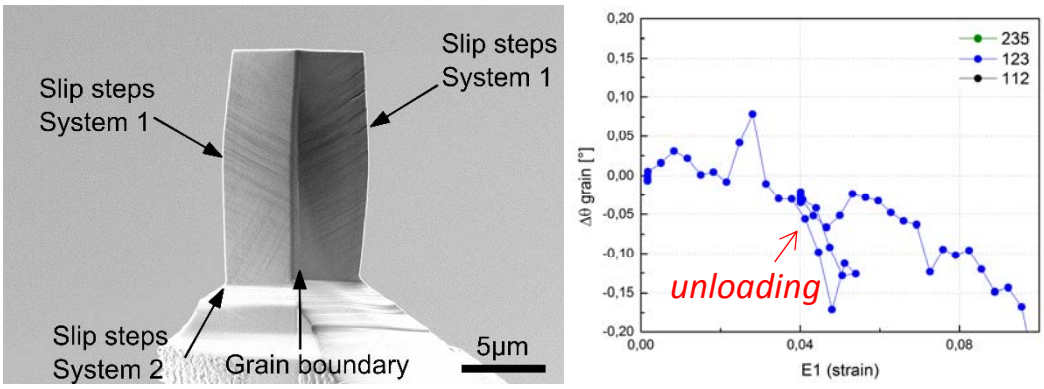

Fig. 1 (a) Post mortem SEM analysis of a general HAGB showing one family of slip steps in grain A and two kinds of slip steps in grain B. (b) change in GB misorientation angle as the simplest measure for the GB character during loading.

In the future we want to extend our work by analyzing the grain-boundary character prior to deformation on the atomic scale and try to understand differences in the individual deformation behavior.