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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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:** Microstructural Changes in Thermo-Mechanically Strained Copper Thin Films and Interconnects	**Experiment number**: MA-940
Beamline: BM32	**Date of experiment**: from: 25.11.2009 to: 01.12.2009	**Date of report**: 01.03.2010
Shifts: 18	**Local contact(s)**: Dr. Jean-Sebastien Micha	*Received at ESRF:*

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Report:

The aim of this experiment was to characterize micro structural features such as activated slip systems, dislocation densities, deviatoric strains and local orientations in micron sized Cu tensile samples in order to get a better inside in the deformation behaviour of materials at the micron scale. The analyzed samples consisted of two groups: Group ***A*** was deformed inside the scanning electron microscope (SEM) at our home laboratory. The second group ***B*** was focused ion beam (FIB) milled at our home facility and strained using our new in situ straining device designed for BM32.

Samples of group ***A:***

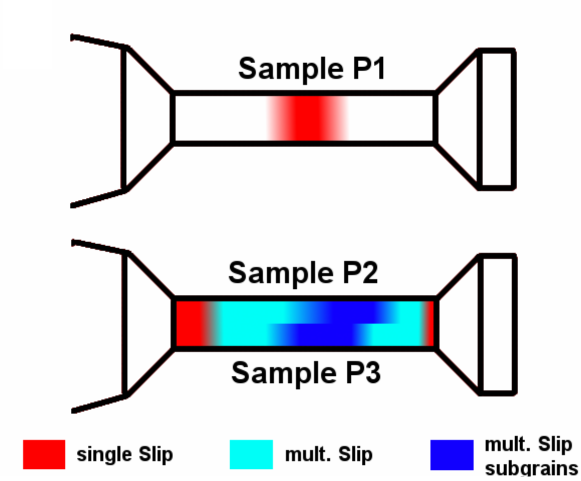

Fig. 1 Slip mechanism maps indicating if the peak streaking direction indicates a single or multiple slip with or without forming dislocation sub-structures.

Deformed Cu tensile samples, strained inside the SEM to 4% (Sample P1) respectively 25% (Sample P2 and P3) were raster-scanned with a step size of 1µm using a one micron sized white X-Ray beam.

Based on these measurements the local orientation and storage of geometrically necessary dislocations (GNDs) was analyzed leading to a schematic slip mechanism map. This map indicates if the stored dislocations originate from one or more slip systems. Furthermore, the formation of sub grains is indicated in the map. Note that the sample head and base were not deformed.

Results of the experiments with Group ***A*** are already submitted to Phil. Mag. Letters.

<u>Samples of group ***B:***</u>

Mainly tensile tests on samples ranging from 3 to 6µm crosssection diameter were performed. Fig. 2a presents a typical stress strain curve of a tensile sample. Positions where the images were read out are marked as red spots. Furthermore, some typical diffraction patterns of the (024) Laue spot are presented with the corresponding position showed in the stress strain curve. Laue peak streaking directions for different slip systems (rank in Schmid factor is displayed near the streak, highest Schmid factor = 1) are projected on the measured peak shape. Based on this images it's fact, that in the initial state of deformation an unpredicted slip system was activated. Nevertheless, by reaching the plateau of the stress strain curve, dislocations of this system were able to escape to the sample surface and the diffraction peak width was reduced considerable till to the maximum applied strain of 25%.

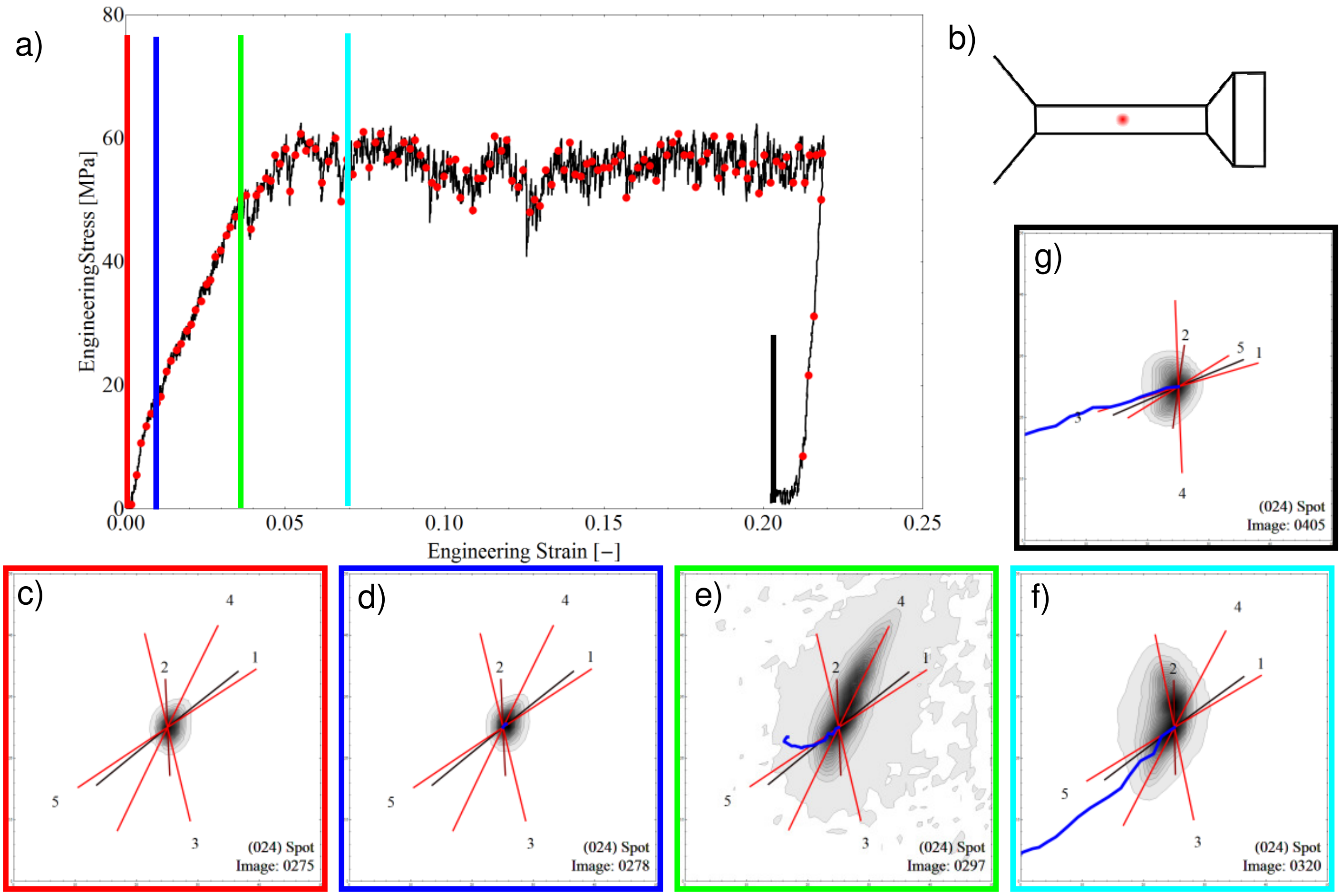

Fig. 2: (a) Typical stress strain curve of a single slip oriented tensile specimen. The red points indicate where images were read out from the CCD. b) indicates the position of the primary beam during the measurement. c-g) are showing the the (024) diffraction spot at different positions of the stress strain curve, indicated by the color code. The projected streaking axes are predictions for the storage of geometrically necessary dislocations on a slip systems. The length of the streaking axes indicates a crystal rotation of 1deg each. In addition, the blue path shows up the movement of the diffraction spot which is in agreement with the classical theorie of constrained tensile experiments.

In summary, the experiments were a sucess and – for our knowledge – the first in situ µLaue tensile test on micron sized samples. The setup – namely the stage, the optical microscope and detector system of BM32 in combination with our new straining device – fulfilled our expectations completely, especially the negligible vibrations and the thermal stability of stage and hutch were impressive. Nevertheless, it's clear that there is a demand for more in situ micromechanical tests. For instance we were not able to monitor the deviatoric strains during loading. Results of the ex situ analyzed samples of group ***A*** are already submitted to Phil. Mag. Letters. Furthermore we expect that data from the in situ experiments (group ***B***) will be submitted to a refered journal in the next weeks.