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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 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-451
Beamline: BM32	**Date of experiment**: from: 14.02.2008 to: 18.02.2008	**Date of report**: 01.03.2009
Shifts: 9	**Local contact(s)**: Dr. Biquard Xavier	*Received at ESRF:*

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Report:

Originally, the aim of this experiment was to characterize structural changes in Cu thin films and interconnects using position resolved µLaue diffraction. The results should have provided detailed information on the structural changes caused by thermal fatigue of microelectronic devices based on Cu.
For the experiment, the users has prepared Cu thin films with different thickness in the range of 50-2000 nm as well as Cu interconnects with the width in the µm range. The structures deposited on Si(100) substrate were locally cyclically heated using pulsed laser radiation at home institution. The laser thermal cycling induced a fatigue with many hillocks, ripples and irregularities on the surface.
Unfortunately, the quality of the µLaue signal collected from laser cycled features was so bad that it was no possible to extract the information on the thermally induced strains in the films. The streaks in the Laue patters were extremely pronounced. Moreover, about one day of the beamtime was lost due to technical problems with the fast shutter.

Besides the Cu films and interconnects the user made an attempt to characterize also free standing Cu pillars and small tensile samples. The pillars were produced using focused ion-beam and tensely strained at home institution.
It is known that mechanical properties of single crystals become size-dependent in the micrometer and sub-micrometer regime. This phenomenon, which can not be explained by the classical continuum mechanics of plasticity, is recently studied worldwide and from substantial interest for micro electronic devices.
Hereafter, result from a tensile strained samples are produced. A SEM image of a deformed sample with clearly visible glide steps is presented in Fig.1. The upper inset in Fig. 1a shows representatively $(\bar{1}\bar{1}1)$ µLaue spots with the corresponding recording positions marked as red dots. In the undeformed regions, namely the gripper head on the left and the massive copper single crystal on the right side, one can see an almost circular shaped peak. In the deformed regions not only the peak position but also the peak-shape is changing (streaking).

By analyzing the peak position it was possible to determine the local crystal orientation, which is in agreement with the EBSD data. Therefore one can conclude that deformation features observed on the sample surface (via EBSD) are representative for the change of the whole volume (analyzed via µLaue). The streaking of Laue spots are directly caused by the storage of excess dislocations in the deformed volume.
Concerning the shift of the diffraction peak one can calculate a misorientation angle with respect to the undeformed crystal. The point to origin misorientation angle was evaluated not only for the µXRD data, but also for the data measured by EBSD and is plotted in Fig. 1b. In the plastically deformed area an almost linear increase of the misorientation angle, reaching a maximum in the middle of the specimen's length axis, is observed. Furthermore, it is worth to notice that the misorientation angle depicts a symmetric behavior over the entire plastically deformed region. The EBSD orientation data is in agreement with the presented µXRD data. Note that due to high deformation or shadowing effects of a large glide step it was not possible to obtain information on the crystal orientation in the center of the sample by EBSD and µXRD.

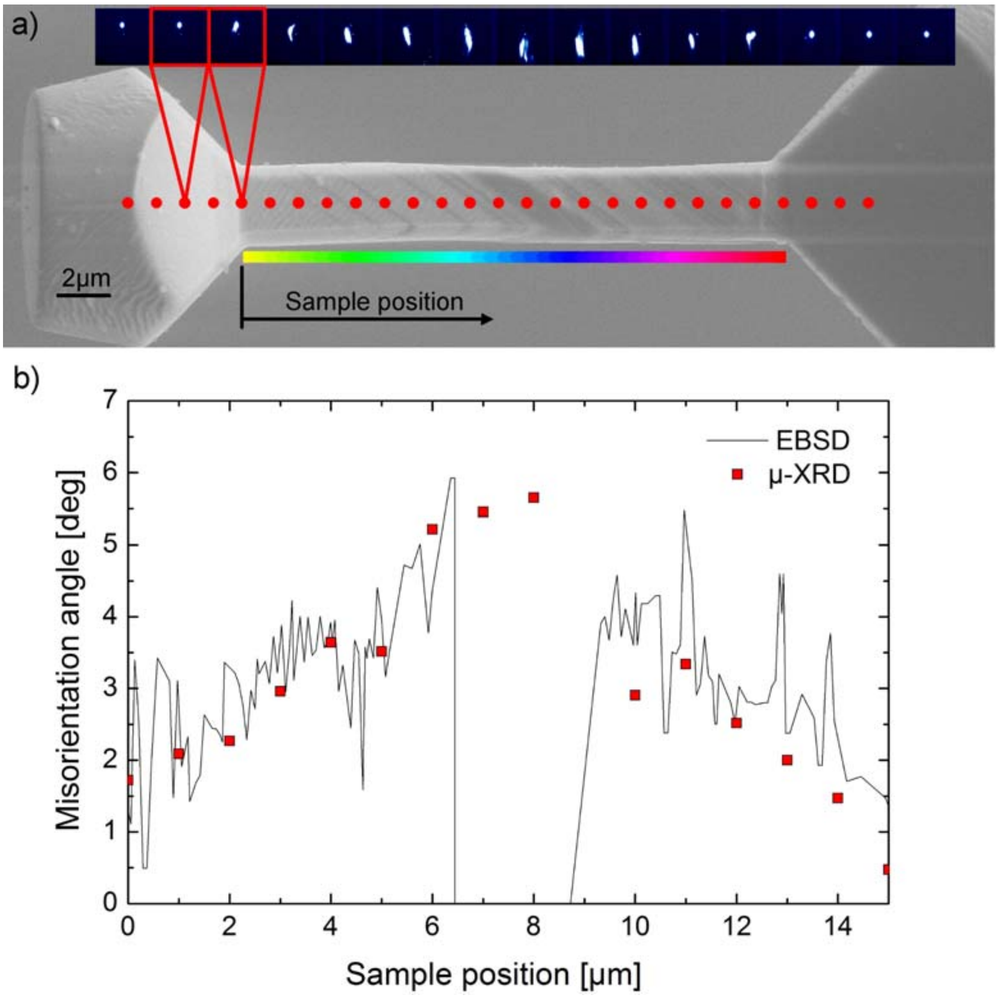

Fig. 1: (a) SEM image of the Cu specimen after the tensile test. Red dots indicate the positions where Laue patterns were collected. The top inset shows ($\bar{1}\,\bar{1}\,1$) micro Laue spots. For clarity, only every second spot is presented. Furthermore the color code for the position resolved trail in the ODF space in Fig. 2 is indicated in the bottom inset. (b) Misorientation angle evaluated from EBSD and µXRD data.

In summary, microdiffraction experiment was used to characterize structural properties of a single-crystalline Cu tensile specimen with the dimensions of 3x3x15 µm fabricated using a focused ion beam workstation. The specimen was strained in a scanning-electron microscope and exhibited a serrated flow which was correlated with the formation of pronounced glide steps on the sample surface. The position-resolved µLaue diffraction along the sample axis revealed a substantial peak streaking and strong changes in the crystalline orientation in the plastically deformed crystal. A comparison of the experimental data and simulated Laue patterns indicates that a single slip accompanied by the storage of dislocations is the dominating deformation mechanism in the tensile specimen.
The results from the µLaue experiment on the Cu samples will be submitted to a refered journal in next weeks.