ESRF	**Experiment title:** Stress tensor determination and crystal orientation evolution during uniaxial loading of a copper bicrystal	**Experiment number**: MA - 555
Beamline: BM32	**Date of experiment**: from: July 9th 2008 to: July 13th 2008	**Date of report**: 31 Aug. 2009
Shifts: 12	**Local contact(s)**: Odile Robach	*Received at ESRF:*

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A copper bi-crystal was mounted in the compression-loading machine shown in Figure 1, with an additional stress-sensor (not shown). The grain boundary (GB) is perpendicular to the axis of loading. The machine was installed using a specially made plate into the BM32 microdiffraction setup. The working plane was 3 mm downstream of the ideal KB-focussing plane along the beam, in order to avoid collisions between the machine and the KB enclosure.

The bi-crystal deformation was investigated, using both white beam (for orientation and deviatoric strain mapping) and monochromatic beam (for lattice expansion mapping). Four states of strain were studied in a centred area including the GB: (1) before loading, (2) at yield, (3) in the plastic domain at ~2% strain, and (4) after unloading.

For each deformation step, the compression machine was removed from the microdiffraction setup to mount the electric motor and monitor the local deformation qualitatively (e.g. appearance of slip lines) and global deformation quantitatively (macroscopic strain) with a large-field optical microscope. The latter measure is calculated with the help of accurate photographs of two widely spaced lines of nano-indentation prints made on the top surface of the sample (see figure 2).

The experimental geometry was calibrated in the white beam mode using a Ge(111) single crystal. Three sets of measurements were collected at each loading step (with the grain boundary perpendicular to the x axis): using white beam (1) a large map of 500 x 500 microns in x and y, with a step of 5 microns in x and 10 microns in y and (2) three x-lines centred on the grain boundary, of 100 microns along x with a 1 micron step. Also, using monochromatic beam (3) one energy scan at 8 points along a x-line around the centre of the large map, at (-250, -125, -10, -5, +5, +10, +125, +250) microns in x from the grain boundary.

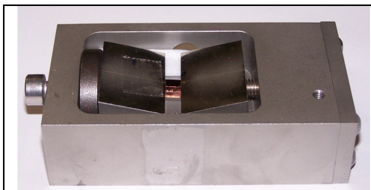


Figure 1: copper bi-crystal in the compression machine. The loading electric motor on the left side is unmounted here.

Data analysis shows correlation between the glide system, visible on the microscopic view, and the microdiffraction results, particularly in term of local lattice rotation. Figure 2 shows the sample surface before and during compression. The area studied with x-rays is marked with a red square. The orientation maps (i.e. local rotation of the lattice) are reproduced for the right grain. These maps were obtained from Laue patterns analysis with the XMAS program. The optical microscope view (fig 2-A) reveals slip traces after compression, in particular on the bottom of the grains, which are well correlated with the areas of high rotation plotted in fig 2-B. The effect of the GB on the local lattice rotation and on the slip patterns is clearly shown in this figure. Work is in progress to calculate the stress maps corresponding to the figure 2.

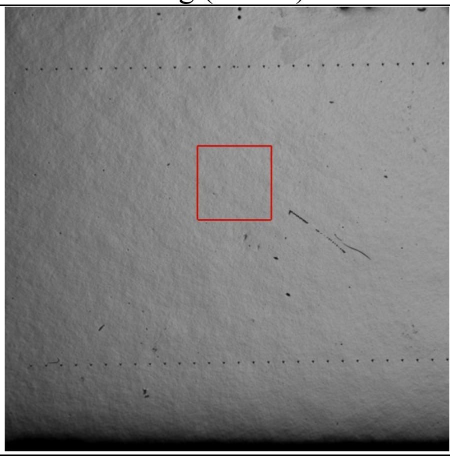
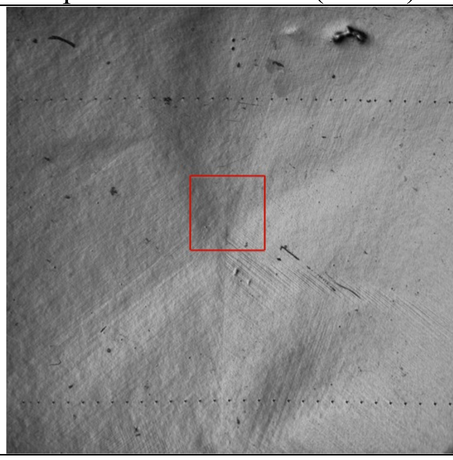
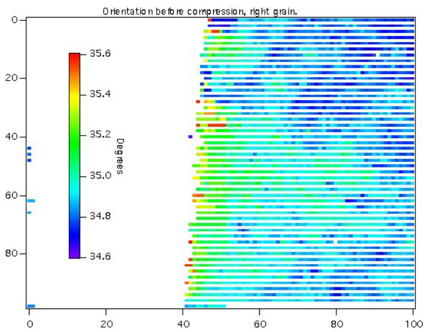
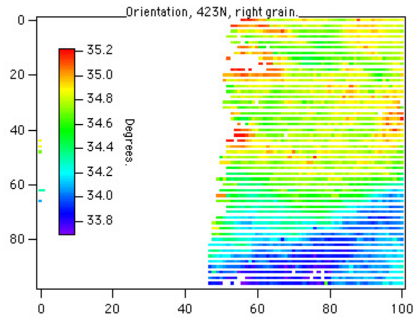
<table>
<tr><td></td><td>Before loading (state 1)</td><td>Sample loaded at 423N (state 3)</td></tr>
<tr><td>A) Optical microscope view (spacing of indentation points = 100 microns</td><td></td><td></td></tr>
<tr><td>B) Local orientation maps 500 x 500 microns^2</td><td>
</td><td>
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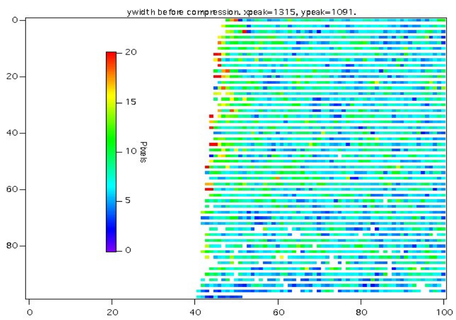
Figure 2: view of the compressed area and cartography of the local rotation in the grain on the right-hand side.

As a preliminary step to the micro plastic strain calculation, which affects the shape and width of the Laue spots, maps of the spot-width (as fitted with a Lorentzian shape function) are reproduced in figure 3. Additional data analysis and theoretical work is needed to calibrate the Laue peak shapes evolution in terms of orientation / strain distribution and dislocation density.



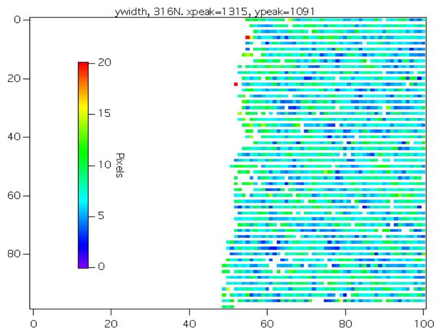


Figure 3: Map of the width (along the y axis) of a characteristic Laue peak, before compression and at F=316N

These results will be used to develop a physically justified crystal plasticity constitutive law accounting for the influence of grain boundaries during deformations. Results of this study were selected as an oral presentation in the TMS symposium "Neutron and X-ray studies of advanced materials" (February 2010, Seattle).