ESRF	**The behavior of grain boundaries during *in situ* µLaue tensile testing**	**Experiment number**: MA 1313
Beamline: BM32	**Date of experiment**: from: 29th April 2011 to: 03th May 2011	**Date of report**: 29.11.2011
Shifts: 15	**Local contact(s)**: Jean-Sebastien Micha	*Received at ESRF:*

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Aim of the Experiment:

Aim of this experiment was to anaylze the behavior of grain boundaries and their effect on the behavior of dislocation in micron sized compression and tensile samples.

Experimental Procedure

The experiment was performed as done in our previous beamtimes using the novel micro diffraction tool at BM32 with an beamsize below 1µm in full width at half maximum (FWHM) and our in situ straining device [1]. More information on the instrumental setup is published in the experimental reports (MA940, MA1058) or in references [1-4].

Results:

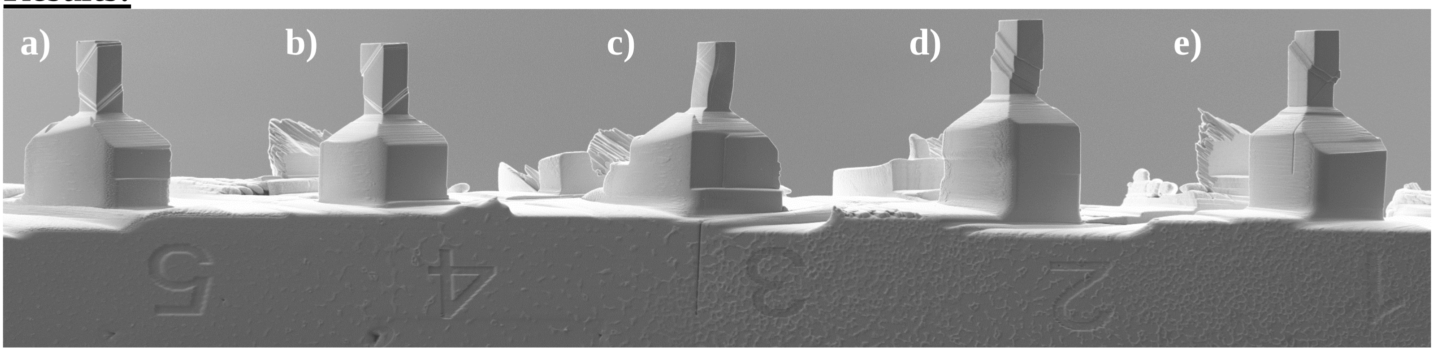

Fig. 1 SEM micrograph of *in situ* deformed µPillars made of (a,b) grain A, (c) a bi-crystal consisting of grain A and B and (d,e) grain C.

Fig. 1 represent scanning electron micrographs of five pillars deformed during the *in situ* µLaue experiment. Pillars A and B (Fig. 1a,b) are single crystalline and oriented for multiple slip, pillars D and E (Fig. 1d,e) are also single crystalline but oriented for single slip and pillar C (Fig. 1c) is a bi-crystalline pillar. Note that the single crystalline pillars exhibit huge slip steps which are caused by the free movement and escape of dislocations, whereas the bi-crystalline pillar possesses a border for dislocation glide and therefore, the slip

steps of the pillar are much finer. It is worthy to note that the SEM micrographs are only able to image slip steps, which are a evidence for escaped dislocations to the sample surface.
The µLaue experiment show a complementary image: Stored dislocations lead to a broader and asymmetric diffraction peak. The first qualitative µLaue results are shown in Fig. 2. In case of the single crystalline pillar (Fig. 2a) the diffraction peaks are only slightly streaked after 20% strain. Only in the sample center geometrically necessary dislocations (GNDs) are stored. However, in the bi-crystalline sample huge peak streaking and the formation of split Laue spots evidences the storage of GNDs and – in parts – the formation of a dislocation cell structure. Also the observed stress strain behavior with tremendous hardening for the bi-crystalline sample and minor to no hardening for the single crystalline samples fully agree with this observation.

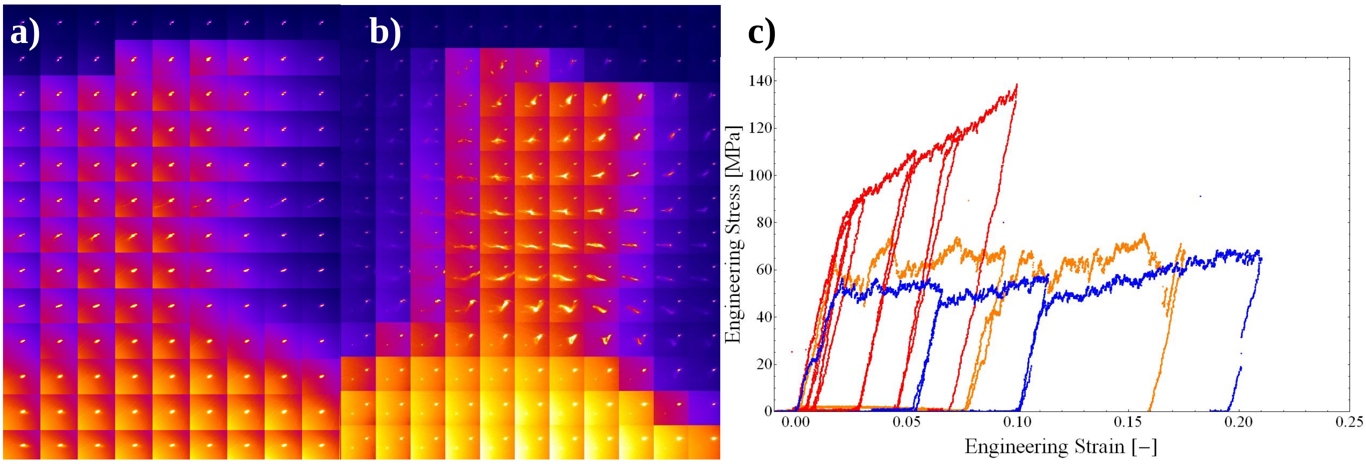

Fig. 2: (a,b) Composite Laue image showing one Laue spot of (a) a single crystalline compression sample after 20% strain and (b) a bi-crystal compression sample after 7% strain. Note that the streaking is much more pronounced for the bi-crystalline sample compared to the single crystalline one shown in (a). In addition, the engineering stress versus strain curve is shown (red=bi-crystal, blue = grain A, orange = grain B).

It is necessary to quantify this qualitative results and to derive mechanism based models on the interaction of dislocations with general grain boundaries. This is goal of our current work and will be published in the future.

Summary:

The implementation of *in situ* test methods for micromechanics at BM32 performed during MA940 and MA1058 now enables to answer more sophisticated questions in material science, as shown by this experiment. The data evaluation of data obtained during this beamtime is still ongoing, however, the first results qualitatively show the power of µLaue at BM32 to study the behavior of grain and twin boundaries at the micron scale. The quantitatively analyzed data will be used for models describing the dislocation-grain boundary-interaction.

[1] Kirchlechner C, Keckes J, Micha JS, Dehm G. Advanced Engineering Materials 2011;13:837.
[2] Kirchlechner C, Keckes J, Motz C, Grosinger W, Kapp MW, Micha JS, Ulrich O, Dehm G. Acta Materialia 2011;59:5618.
[3] Kirchlechner C, Imrich PJ, Grosinger W, Kapp MW, Keckes J, Micha JS, Ulrich O, Thomas O, Labat L, Motz C, Dehm G. Acta Materialia 2011;accepted manuscript.
[4] Kirchlechner C. Plasticity at the Micron Scale: A µLaue Study. Leoben: Austrian Academy of Sciences and University of Leoben, 2011.