ESRF	First in situ µLaue Diffraction Experiment on Tensely Strained Cu Pillars @ BM32	**Experiment number**: MA 1058
Beamline: BM32	**Date of experiment**: from: 13th May 2010 to: 18th May 2010	**Date of report**: 15.11.2011
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 perform the first *in situ* µLaue tensile experiments on micron sized tensile samples at BM32 in order to understand size dependent placticity processes. Preliminary experiments dealing with the right instrumental setup and also running the first test have already been performed during MA940.

Beamtime Preparation in our home Facility:

In total, 25 single crystalline copper tensile samples were produced in our home facility using a focussed ion beam (FIB) workstation. The size of the samples was in the range of from $3x3x9\mu m^3$ to $7x7x35\mu m^3$. Also 3 counterbodies of different size were produced.

The Experiment itselves:

Our experiments were carried out as follows:

1) Alignment of sample and gripper by the (very good) optical microscope at the beamline
2) Alignment of the sample in the beam by X-ray fluorescence mesh scans
3) Mesh scan of the undeformed sample
4) *In situ* scan: A time scan with fixed X-ray beam placed in the sample center with continous, displacement controlled straining of the micron sized samples
5) Mesh scan of the deformed sample
6) In some cases a second and third loading sequence.

Due to the limited time during the experiment we were only able to test 10 of these samples *in situ*. Noteable, the vibrations at the beamline, the optical microscope and the provided software (for instance for X-ray fluorescence mesh scans) allowed very nice experiments. BM32 at ESRF in combination with our straining device [1] is the ideal tool for performing this kind of experiments.

Scientific Results:

The experiment shows “Expected and unexpected plastic behavior at the micron scale” which is summarized in our recent publication [2] accepted in Acta Materialia. In the expected case there is no storage of geometrically necessary dislocations (GNDs) in the sample center and a large share of multiplied dislocations is able to freely move to the sample surface. On the other side there is unexpected behavior, where dislocations of an unexpected slip system (low Schmid factor) multiply and keep beeing stored at low strains due to an native oxide or FIB-damage layer. This is shown in Fig. 1a (expected) and b (unexpected), where not only the stress strain curve but also the peak width in streaking direction (a measure for the density of GNDs, red) and in transverse direction (statistically stored dislocations, blue) is shown.

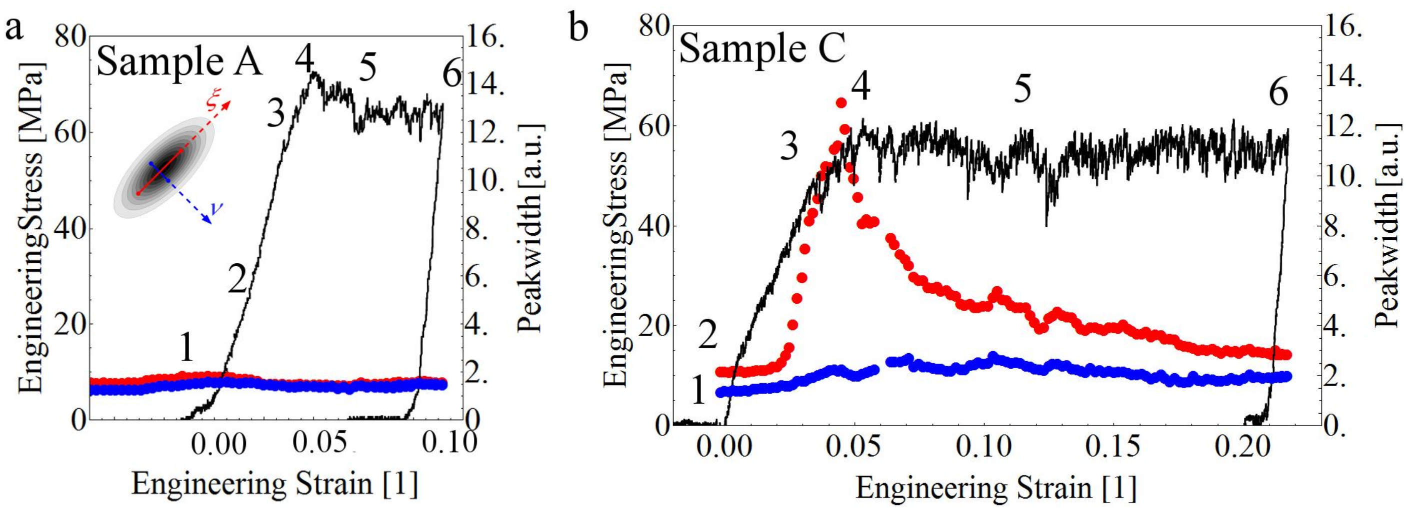

Fig. 1 Stress versus strain curve of samples A and C including the evolution of the diffraction peak width in ξ and ν direction. Sample A shows the macroscopically expected case. Sample C the unexpected case caused by the limited sample size and dislocation statistics. (See text for details).

Summary:

The experiments during MA1058 were succesfull from an experimental as well as from a scientifical point of view. The insights obtained by this method are unique and provide the experimental evidence for advanced small scale material models and laws. By this, these experiments signficantly contributed to the understanding of size dependent plasticity.

[1] Kirchlechner C, Keckes J, Micha JS, Dehm G. Advanced Engineering Materials 2011;13:837.

[2] 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.