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Experimental report of the HC/1449 session at BM32

Objective & expected results :

A precise knowledge of parameter values governing intragranular constitutive laws is crucial when trying to simulate stress concentration phenomena in metals. A promising way to identify precisely these parameters is to consider both elastic and plastic strain fields developing locally in mechanically deformed specimens.

Following our last experiment (HC/913), we showed that combining digital image correlation techniques and Laue microdiffraction enables the joint evaluation of plastic and elastic strain fields at the sample surface. In the case of homogeneous single crystals deformed in-situ in a four point bending device, we obtained measurements with a strain accuracy and spatial resolution suitable for a comparison with finite element models.

In this work, we extend the methodology to crystals containing grain boundaries (bi- and multi-crystals) in order to observe the influence of microstructural heterogeneities on the material behavior. These new data will establish an experimental basis for parameters identification procedures and comparison with finite element simulations.

Progress & conclusions of the study :

During the experiment two types of specimens (30*5*0.5mm) were scanned:

- Two 316L bi-crystals with a grain boundary at the center of the specimen. The misorientation between the two grains is close to 64°. The bi-crystals are deformed in a four point bending device. Laue and DIC measurements are carried out on the surface where stress varies from tension to compression (cf. fig.1b). Both samples were prepared with a flat and stress-free surface on which a speckle pattern of Mo particles was deposited for DIC analysis.
- One multi-crystal cut from a heat-treated industrial 316L with a grain size up to 1mm. A region inside one large grain with few twins is investigated. The sample was prepared with a flat and stress-free surface on which a speckle pattern of Mo particles was deposited for DIC analysis.

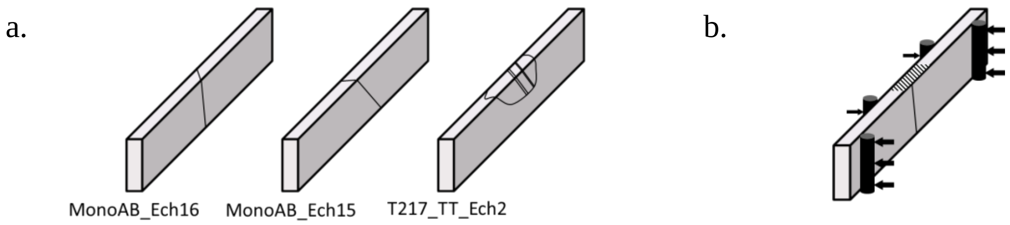

Fig. 1: (a) Schematic view of the two bi-crystals and the multi-crystal. (b) Configuration of the bending experiment. The observed area is hatched.

Thanks to the improved microdiffraction setup, a stable beam of size 1100 x 400 nm² (HxV) was achieved. The MARCCD detector was positioned ~6 cm away from the sample. An optical microscope with low lenses distortions was installed, in the experiment hutch, next to the diffraction setup. We used our dedicated four points bending device to strain the samples. The machine is equipped with an easy-removal system, which allows its transfer between the optical microscope and the microdiffraction setup.

The experiment runs as follows:

- The sample is inserted in the bending device, unstrained. The setup geometry is calibrated on two unstrained Ge single crystals and the beam size is checked at the sharp edge of a copper micro-block. A map of diffraction patterns is then acquired with characteristics depending on the sample:
  - For MonoAB_Ech16: the map size is 500 x 600 µm and the step size 25µm.
  - For MonoAB_Ech15 and T217_TT_Ech2: the map size is 600 x 700 µm and the step size 12µm.
- The bending device is brought under the microscope. Images are recorded before, during the loading, and after. We take care to acquire images focused and unsaturated.
- While the displacement is kept constant, the machine is transferred to the diffraction setup and the map is acquired at the same location as before.
- Several loading steps are carried out :
  - For the MonoAB_Ech16 and T217_TT_Ech2 : 4 steps are investigated (initial state, end of the elastic regime, after some plasticity, residual state after unloading)
  - For the MonoAB_Ech15: 10 loading steps are investigated including 3 steps in the elastic regime, 6 in the plastic regime and one in the relaxed state.

The diffraction patterns recorded are of good quality, with almost circular Laue spots. Data is currently being processed with the open software LaueTools, developed at BM32, and will be analyzed further with the new Laue-DIC method. Optical images have a contrast suitable for the digital correlation technique. We plan to use the software “VIC” to perform the correlation.

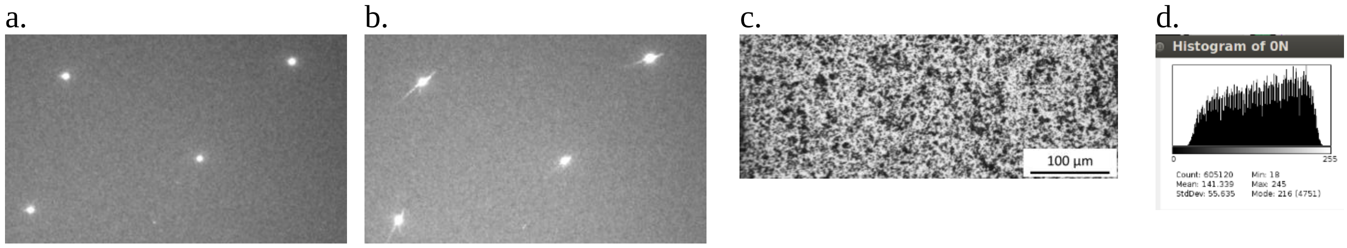

Fig. 2: Shape of the diffraction peaks from MonoAB_Ech16 (a) before straining the sample and (b) after some plastic deformation. (c) Speckle pattern used for digital image correlation and (d) its histogram of grey levels.

This experiment ran as expected. Thanks to the data collected, we should be able to study the evolution of elastic and plastic strain fields around two single grain boundaries and two twin boundaries.

The BM32 beam-line is now equipped with an optical microscope dedicated to digital image correlation. A very convenient improvement will be achieved when this microscope will be installed, as planned, on the diffraction setup, so that the experiment can be run in-situ without having to transfer the specimen from the microdiffraction setup to the optical microscope.

<u>Comments about the use of beam time :</u>

The first day of beam time was used for beam alignment, installation and calibration of the optical microscope and four points bending setup. Data has been acquired continuously since the second day. Some beam time (~2x6 hours) was lost due to a bug coming from the MARCCD acquisition software. In general, the experiment ran nicely.

<u>Publication(s) :</u>

We are currently working on the data acquired during the experiment. This work is a key part in the PhD of Emeric Plancher. A publication will be submitted when final conclusions and comparisons with a finite element model will be achieved.