EUROPEAN SYNCHROTRON RADIATION FACILITY

ESRF User Office

BP 220, F-38043 GRENOBLE CEDEX, France

Delivery address: 6 rue Jules Horowitz, 38043 GRENOBLE, France

Tel: +33 (0)4 7688 2552; fax: +33 (0)4 7688 2020; email: useroff@esrf.fr; web: http://www.esrf.fr

Experimental report of the HC/913 session at BM32

Objective & expected results :

Strain heterogeneity develops at the micron scale in polycrystalline metals during mechanical deformation, often leading to detrimental back stresses. In order to reproduce this phenomenon through numerical simulation, one should pay attention at the constitutive laws used to describe intragranular elasto-plastic behavior.

Our current research, funded by the French ANR project "Microstress", focuses on collecting trustworthy data required for parameter identification of some 316L steel constitutive laws. Especially, we try to assess elastic strains (or stresses) and total (or plastic) strains fields at the micron scale. Total strains are usually obtained by the well-established digital image correlation technique (DIC). Hence efforts concentrate on asserting the quality of elastic strain measurements, in our case obtained by two promising techniques: synchrotron-based Laue microdiffraction and SEM-based High Angular Resolution EBSD (HAR-EBSD).

One of our previous concerns was to determine the surface characteristics needed for acquiring reliable HAR-EBSD data. In order to compare the in-depth strain information of Laue microdiffraction (>10µm) with the near-surface information of HAR-EBSD (<50nm), we suggested to manufacture standards from Si, 316L steel (Fe-Cr-Ni) and W single crystals, with controlled surface preparation. A preliminary experiment, performed at BM32 in Febrary 2013, was carried out on a 316L single crystal with optimal preparation (flat surface, roughness <1µm, no back stress).

It turns out that even in this optimal case, comparison of HAR-EBSD and Laue microdiffraction datasets remains a challenge. In particular, this preliminary work highlights insufficient accuracy of the alignment procedure in the SEM when performing HAR-EBSD. As we are currently addressing this issue, working with only few 316L standard samples, with optimal preparation, seemed relevant.

We thus redirected part of our work on acquiring both Laue microdiffraction (elastic strain evaluation) and proper optical images for image correlation analysis (total strain measurements), on two 316L single crystals deformed in a four points bending machine. The machine was specifically developed in our lab for the purpose of this experiment. Coupling the two fields must lead to more efficient ways for identification of constitutive law parameters.

Progress & conclusions of the study :

During the experiment two types of specimens were scanned:

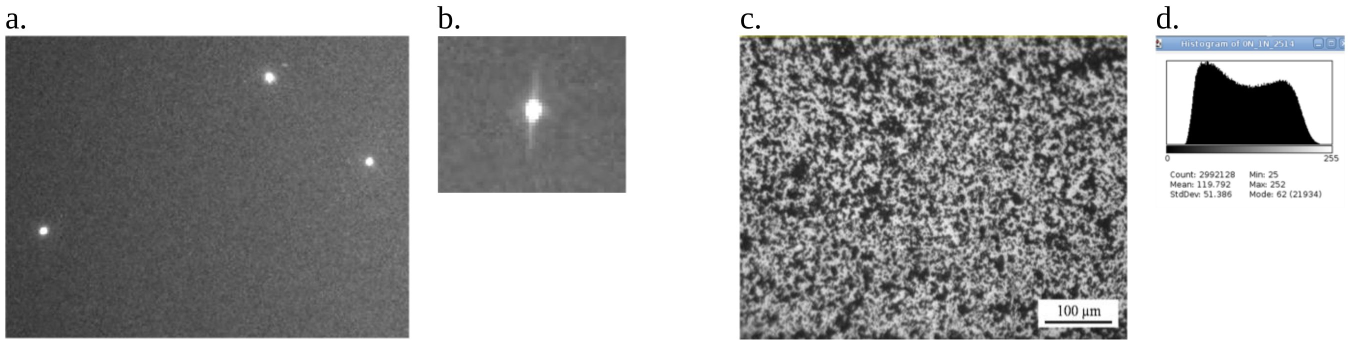
- Two 316L single crystals, used as reference samples for comparison between HAR-EBSD and Laue microdiffraction, in different states: (i) stress free and (ii) plastically deformed and unloaded. Theses sample were carefully embedded in mounting resin, prepared to obtain a flat surface, no roughness (<1µm) and no back stress from polishing.
- Two 316L single crystals in the shape of four points bending samples (30*5*0.5mm). Measurements are carried out on the surface perpendicular to the loading direction where stress vary from tension to compression. Both samples have a [001] direction normal to this surface. The direction along the length is [100] in one sample and [110] in the other. Both samples were prepared with a flat and stress-free surface on which a speckle pattern of Mo particles was deposited for DIC analysis.

Thanks to the improved microdiffraction setup, a stable beam of size 500 x 280 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 a 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 on bent samples runs as follows:

- The sample is inserted in the bending device, unstrained. The setup geometry is calibrated as usual on an unstrained Ge single crystal and the beam size is checked at the sharp edge of a gold micro-block. Three line scans of 300 Laue patterns are acquired.
- The bending device is brought under the microscope. Images are recorded before, during the loading, and after. We take care to ensure images are focused and unsaturated.
- While the displacement is kept constant, the machine is transfered to the diffraction setup and three scans are acquired at the same location as before.
- Respectively 8 and 10 loading steps are carried out, including 3 to 4 steps in the elastic regime and one in the relaxed state.

The diffraction patterns recorded are of good quality, with almost circular Laue spots. We observed nevertheless strange shapes at the foot of some peaks in Ge patterns. Few images also come with one-pixel-thick bright lines. All the data is currently being processed with the open software LaueTools, developed at BM32, and will probably be analyzed further with the new Laue-DIC method. Optical images have a contrast suitable for the digital correlation technique. We plan to use EDF's in-house code to process the data.



(a) Three diffraction peaks from the dataset acquired on a 316L single crystal. (b) Diffraction peaks with an elongated foot from Ge calibration data. (c) Speckle pattern used for digital image correlation and (d) its histogram of gray levels.

This experiment ran as expected. Thanks to the data collected, we should be able, for the first time, to evaluate stress and plastic strain fields on two single crystals during deformation. The coupling of both fields seems to be a promising way to improve parameter identification procedures of constitutive laws. We are planning to extend this experimental procedure to materials exhibiting a microstructure closer to industrial ones, such as bi- and poly-crystals, in order to evaluate the relationship between the microstructure and field heterogeneities. The grain boundary effect on the deformation will then be measured.

The BM32 beam-line is currently acquiring 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 and a half day of beam time was used for beam alignment, installation and calibration of the optical microscope and four points bending setup. No machine day was available before our experiment.

Data has been acquired continuously since then and until the end of the run. Some beam time (~8 hours) was lost due to a crash of the main computer. In general, the experiment ran nicely.

Publication(s) :

We are currently working on the data acquired during the experiment. This work is a key part in the PhD of Emeric Plancher. In the coming months, we will submit a short paper on some of our results. More extensive publications will be submitted when final conclusions and comparisons with a finite element model will be reached.