ESRF	**Experiment title:** Complementary analyses of the 3D grain structure and elastic strains in a steel polycrystal submitted to elastoplastic deformation.	**Experiment number**: MA1922
Beamline: ID11	**Date of experiment**: From 23/01/2014 to 28/01/2014	**Date of report**: 25/06/2015
Shifts: 15	**Local contact(s)**: Wolfgang Ludwig	*Received at ESRF:*
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Samples

This experiment aimed at characterizing the 3D grain structures of polycrystalline samples and investigating their mechanical behaviours during in situ elasto-plastic deformation. Two different materials were used for the experiment: a high-purity aluminium alloy (Al-0.3%Mn) and a high-purity model alloy (Fe-30%Mn). The former has the advantage of developing intragrain rotation fields in the early stage of plastic deformation, while the latter (which also deforms by crystallographic slip) allows to reach higher stress (which is required for grain-scale stress analyses). Tensile specimens with a square section of ~1x1 mm² and a gage length of 1.5 mm (see Fig. 1a) were spark-machined out of cold rolled sheets and heat-treated in our laboratory to produce fully recrystallized microstructures with average grain sizes of about 100 µm (Al-0.3%Mn) and 50 µm (Fe-30%Mn). This sample preparation was essential for successful grain structure mapping by DCT.

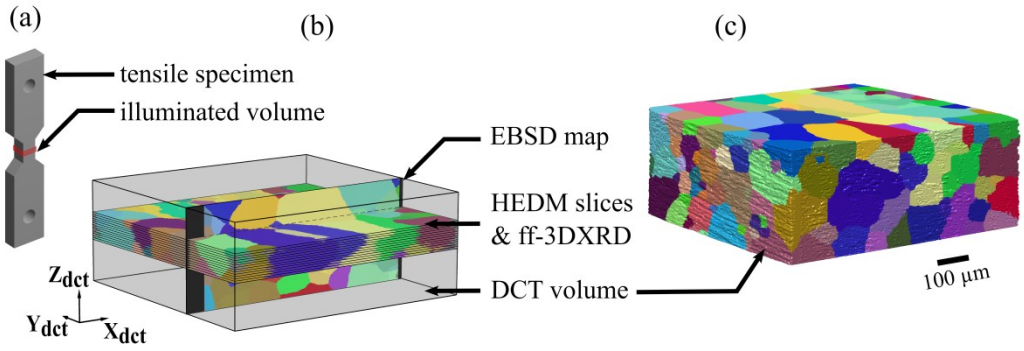

Fig.1 : Comparison between HEDM, DCT, EBSD and 3DXRD on the same region of an aluminium sample [1]. Colours related to orientations.

During beamtime

Fifteen shifts were allocated for the experiment, during which five distinct samples were measured. Details of the measurements are summarized in the following table.

	Material	Measurements
S1	Al-Mn	- 3 DCT scans (3 1x1x0.5 mm^3 volumes to map the whole gage length) - Far-field 3DXRD during tensile test (0% -> 4%, 7 states)
S2	Fe-Mn	- DCT (1x1x0.5 mm^3 volume) - Far-field 3DXRD during tensile test (0% -> 3%, 4 states)
S3	Fe-Mn	- Far-field 3DXRD during tensile test (0% -> 5%, 6 states)
S4	Al-Mn	- Far-field 3DXRD (0% -> 10%, 6 states)
S5	Al-Mn	- DCT scan (after 1% plastic deformation) - HEDM coupled with far-field 3DXRD (planar focused beam, 14 layers)

Samples 1-4 were deformed using the Nanox rig available at ID11, which was mounted on the diffractometer for in situ far-field 3DXRD measurements. Throughout the experiment, the energy of the beam (60 keV) could be monitored thanks to a device available at ID11 and based on a silicon single crystal wafer. In principle, this method provides a determination of the energy with an error as low as 1 eV. In practice, variations of the order of 100 eV during the experiment were later identified, which turned out to be detrimental to an accurate determination of grain elastic strains / stresses from the 3DXRD data [2].
Overall, 1 shift was dedicated to beam alignement and switching between samples or techniques, 3 shifts were used for DCT scans, 8.5 shifts were used for 3DXRD, while 2.5 shifts were necessary to set up the semi-transparent, 3D detector, obtain a focused planar beam with 2 µm in height and perform HEDM layer-by-layer measurements.

Data analyses

DCT measurements were later analysed at ESRF with the help of W. Ludwig. The 3 volumes of sample 1 were reconstructed separately, carefully aligned and finally merged. This provided an image of the full gage length of the sample (Fig. 2), as required by the intended polycrystal deformation analysis.
HEDM data were analysed using the forward-modeling software IceNine. It was the first time that the HEDM technique was applied to data acquired at ESRF. The DCT, HEDM and far-field 3DXRD data were then compared in detail (Fig. 1). A first publication was written and submitted to IUCrJ in May 2015 [1]. This work demonstrated that DCT is an efficient method even for an aluminium alloy deformed to 1% plastic strain and detects subgrain structures with disorientations as low as 1°.
Far-field 3DXRD measurements were analysed with ImageD11 and with in-house software packages. In particular, for sample 4, some grains were followed from the underfomed initial state up to 6.6% plastic strain. By analysing the shape and intensity distribution of their diffraction spots, it was possible to characterize and better understand the formation of dislocation walls and subgrain structures in aluminium due to plastic deformation (PhD of E.F. Filippelli [3]). A publication on the subject will be submitted by the end of summer 2015. Another major study is still in progress on sample 1. The grain lattice rotations are being analysed in detail and compared to the predictions of finite element crystal plasticity simulation (PhD of L. Renversade). The results will be compared to a recently-developed theory [4].
Data related to Fe-30Mn are still under processing.

Fig.2 : DCT volume made of 3 subvolumes (bottom, middle and top). Colours according to orientations. The volume has a section of 900 x 900 µm^2 and a height of 1.5 mm, and contains 1870 grains.

Publications related to the experiment ([1-3] result from the experiment)

1) Renversade, L., Quey, R., Ludwig, W., Menasche, D., Maddali, S., Suter, R.M. and Borbély, A. (2015) *Comparison between Diffraction Contrast Tomography and High Energy Diffraction Microscopy on a slightly deformed aluminum alloy*. Submitted.
2) Renversade, L., Kenesei, P., Wright, J. and Borbély, A. (2015). *On the Misalignements of a High-Energy X-Ray Diffractometer and the Accuracy of Cell Parameter Measurements*. TMS 2015, Orlando, Florida, USA.
3) Filippelli, E.F., Renversade, L., Quey, R. and Borbély, A. (2015). *In-situ characterization of dislocation wall formation in deformed tensile polycrystal by 3DXRD*. Micro-DICE 2015, Montpellier, France.
4) Quey R., Driver J.H., and Dawson P.R. *Intragrain orientation distributions in hot-deformed aluminium: Orientation dependence and relation to deformation mechanisms.* Submitted.