

Application Note # EBSD-02

Orientation contrast imaging and EBSD analysis on materials deformed by in-situ compression and tensile testing

The evolution of texture and microstructure during plastic deformation controls the bulk mechanical properties of virtually all materials used to manufacture engineering components and equipment. SEM based in-situ tensile/compression testing allows investigating the onset of plastic deformation as well as all intermediate deformation stages. Microstructure and texture evolution can be quantified when combining this technique with EBSD. The quantitative results can then be used for refining the plastic deformation theory as well as for confirming the existing simulation models.

In spite of its great potential for delivering extremely useful experimental information, the combination of the two techniques is still not extensively used due to technical challenges posed by drift, hardware design limitations and acquisition time. While the acquisition time issue has been solved with the introduction of high speed EBSD systems, other challenges still remain. This application note aims at discussing these remaining issues while presenting experimental results to prove the advantages of the recent hardware/software development advances.

Experimental procedures and results

The experiments were made using a ZEISS SUPRA 55VP FE-SEM and a 10 kN Kammrath & Weiss stage (see Fig. 1) designed for in-situ tensile/compression tests at 70° tilt for optimal EBSD signal.

The evolution of texture and microstructure was characterized by EBSD and orientation contrast imaging using the *eFlash^{HR+}* high resolution EBSD detector and its integrated ARGUSTM FSE/BSE imaging system, which features the unique capability of delivering color coded orientation contrast images. For details on the color coded orientation contrast imaging technique, please consult the paper by A.P. Day et al [1]. Samples with dimensions defined in the drawing shown in Fig. 1 were cut from annealed ARMCO steel with an average grain size of approx. 130 μm using spark erosion. Due to the relatively large size of the powerful in-situ tensile stage (maximum load of 10 kN) and to the special geometry required when performing EBSD analysis, the minimum possible working distance (WD) for scanning the sample was 27 mm. A very high WD would normally

Instrumentation

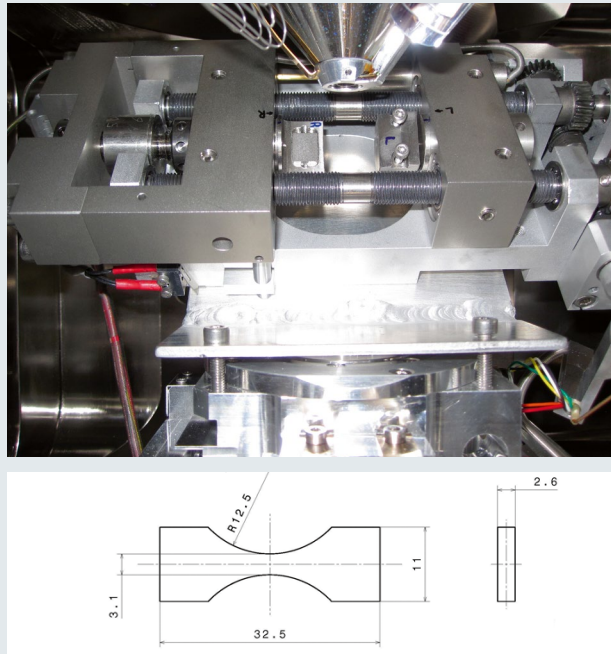


Fig. 1: Left – ARGUS™ FSE/BSE detectors and phosphor screen; right top – tensile stage inside the SEM chamber as seen from the EBSD port; right bottom – drawing of sample dimensions (values are given in mm).

make any EBSD analysis very difficult, as the point source would be much lower than the phosphor screen and signal would be of rather poor quality. The tilting capability of the *eFlash* detector was used for compensating for this difference in height, i.e. the phosphor screen was moved downwards to follow the electron beam focus point (see Fig. 2a and 2b). This allowed us to acquire Kikuchi patterns in an optimum sample-detector geometry resulting in a perfect signal distribution for both orientation mapping and orientation contrast imaging.

Equally important was to get the phosphor screen close enough to the sample for acquiring patterns with sufficient visible zone axes and good signal yield. This was achieved by safely inserting the phosphor screen in between the grip plates holding the sample (Fig. 2c). The safe phosphor screen insertion was executed through the software interface and it was possible by correlating the sample size with the detector head dimensions.

The most challenging issue when doing in-situ deformation tests is the sample/stage mechanical drift induced either by the assembly weight or by the stage once the deformation has started. The weight related drift was removed by “lan-

ding” the whole setup (in-situ deformation and SEM stage) onto a bulk metallic piece placed at the bottom of the SEM chamber. The drift induced by the deformation stage was corrected manually using the highly detailed orientation contrast images, also known as Fore Scattered Electron (FSE) images (Fig. 3).

Fig. 3 shows four FSE images acquired in the same area at different stages of the tensile deformation process. The images depict the initial and final microstructure (210 MPa), as well as two intermediate states, i.e. at 120 MPa and 180 MPa. The FSE images are extremely sensitive to small changes in the diffraction signal, thus allowing the user to observe the very beginning of the deformation process.

Fig. 4 presents the Grain Average Misorientation maps of four datasets acquired at different stages of the tensile test experiment. The four images indicate that, as expected, the misorientation inside the grains increases with strain (red areas). Please notice the high values of misorientations in the vicinity of grain boundaries (shown in black) confirming the migration of dislocations towards the grain boundaries, resulting in high dislocation density areas.

Detector tilt feature

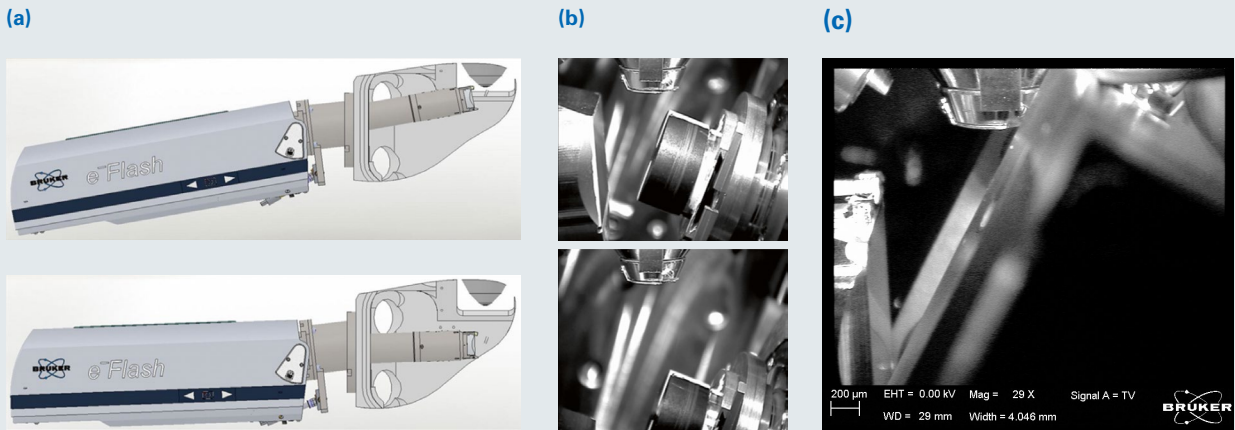


Fig. 2: (a) Schematic view of the unique detector tilt feature and (b) the corresponding infrared (IR) chamber scope images showing the phosphor screen “following” the sample over a large domain of WDs. (c) IR chamber scope image showing the tensile stage / EBSD detector setup used during the experiments.

Orientation contrast images

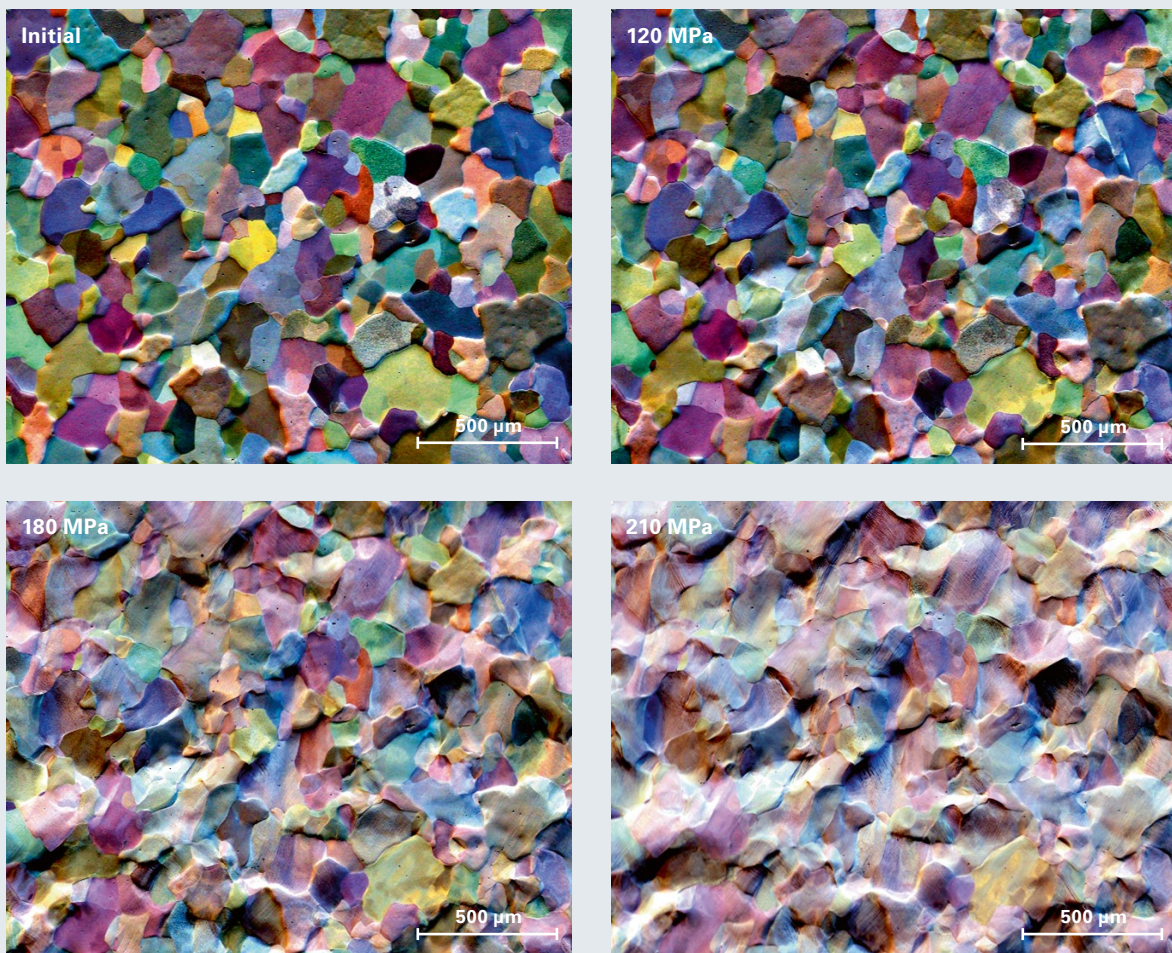


Fig. 3: Orientation contrast images showing microstructural evolution during in-situ tensile testing of ARMCO steel; initial microstructure (upper left) and after applying 120 MPa (upper right), 180 MPa (bottom left) and 210 MPa (bottom right).

Grain Average Misorientation maps

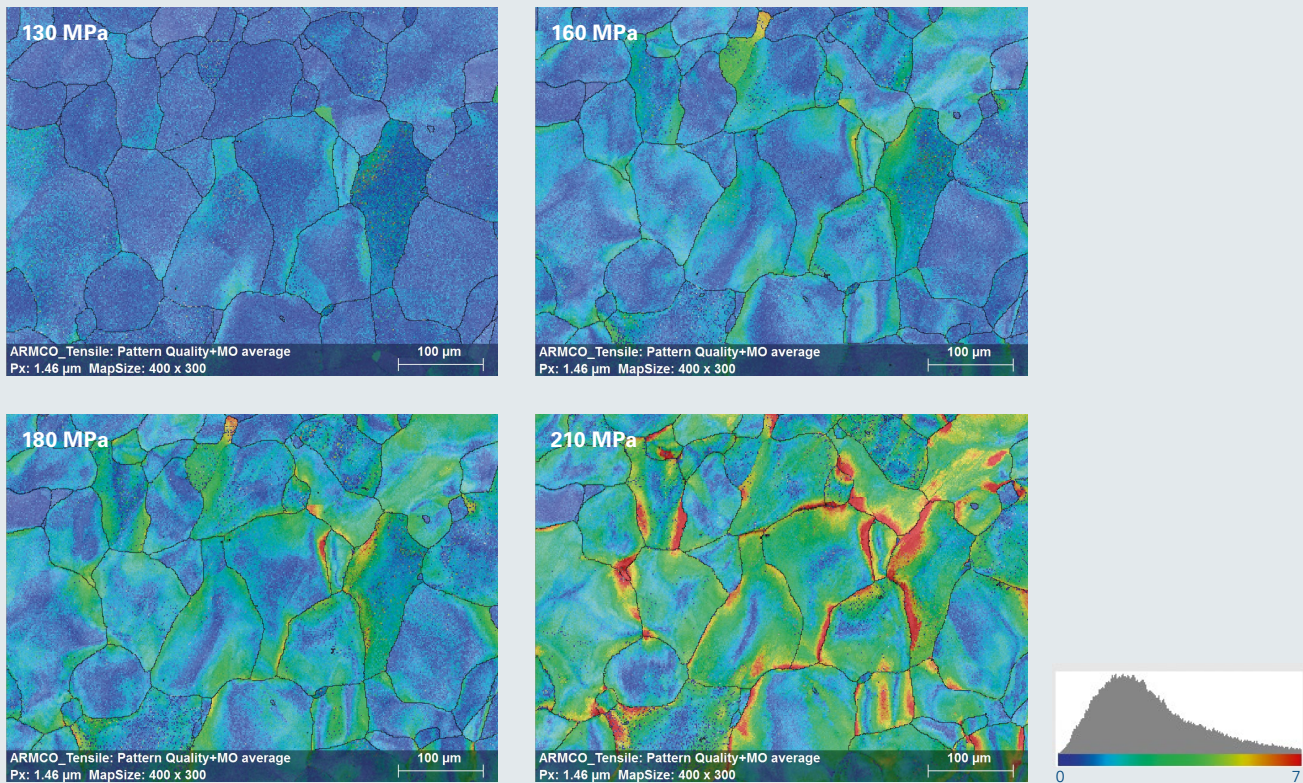


Fig. 4: Grain Average Misorientation maps showing the “accumulation of deformation” inside the ARMCO steel grains at different stages of the in-situ tensile testing experiment; colors show orientation changes from 0 to 7 degrees.

Conclusion

Recent hardware and software advancements have enabled in-situ tensile and compression testing experiments in combination with FSE imaging and EBSD analysis. The very beginning of plastic deformation was easily identified using FSE imaging, while orientation maps were acquired to allow a proper quantification of the evolution of texture and microstructure during the different stages of the deformation process. The results have shown that the combination of in-situ tensile testing with EBSD and orientation contrast imaging can be successfully used to deepen our understanding of the complex phenomena that take place during all stages of plastic deformation in different types of materials.

References

[1] A.P. Day et al, Journal of Microscopy, Vol. 195, Pt 3, September 1999, pp. 186-196.

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