doi:10.1017/S1431927613010805
Microsc. Microanal. 19 (Suppl 2), 2013

Quantitative Microstructure Characterization by Application of Advanced SEM-Based Electron Diffraction Techniques.

S. Zaefferer, N. Elhami, P. Konijnenberg, T. Jäpel

Max-Planck-Institute for Iron Research, Dept. for Microstructure Physics, 40237 Düsseldorf, Germany

Two electron diffraction techniques are available for microstructure quantification in an SEM, namely electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI). These two techniques are in many regards complementary to each other and allow, together, a comprehensive characterization of many aspects of microstructures on bulk samples. The different electron scattering processes that lead to ECCI and EBSD lead to different interaction volumes, i.e. spatial resolutions for both techniques, different requirements for signal detection and to different features that can be observed with them. Table 1 compiles a few numbers and information for materials of average atomic number (Z = 20 … 40) and average microscope conditions (E_0 = 15 … 25 kV).

A microstructure of crystalline matter, as we understand it here, comprises all defects of the perfect crystal structure, i.e. point defects like vacancies, interstitial or substitutional atoms, line defects like dislocations, planar defects like stacking faults and all kinds of interfaces and volume defects like voids, crystallites and precipitations, and stress and strain fields. With possible exception of the point defects, all defect types can be characterized in quite some detail by the mentioned SEM-based diffraction techniques. We will present some examples in the following.

Individual line defects, i.e. dislocations, can be observed using the ECCI technique (e.g. [1],[2]). To this end the crystal is positioned such that the primary beam excites one set of lattice planes at Bragg angle illumination, exactly as in the case of dark field TEM observations. As shown in figure 1, dislocations then appear as bright lines on a dark background. The technique can be used conveniently to perform in-situ observations of deformation processes on bulk samples.

Grain and phase boundaries are important planar defects. Their properties depend on the crystallography of the grain boundary which has 5 degrees of freedom, 3 parameters describing the misorientation between the two grains and 2 parameters for the position of the boundary normal. The complete 5 parameter description of a boundary can be measured by serial sectioning using EBSD where serial sectioning may be performed by focused ion beam (FIB) sputtering (e.g.[3],[4]). Figure 2 shows the reconstructed grain boundary measured with an EBSD-FIB system.

Volume defects can be observed by both, EBSD and ECCI. An important 3-dimensional defect is a residual stress field. In ECCI this may be qualitatively visualized by tilting the grain close to two-beam conditions and observing the spatial distribution of intensities. EBSD, however, allows quantifying the lattice strain and stress components, for example using a pattern cross-correlation technique [5]. In this way strains down to a 10^{-3} and, depending on the elastic modulus, stresses down to 10 MPa can be determined. Figure 3 shows and example of a quantitative measure of elastic strains in a steel sample.

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	Interaction volume [nm³]	Angular resolution [°]	Detector geometry	Observable features
ECCI	1 … 2 x 10^3	0.1 … 0.01	Integral detector in back scattering position	Individual defects (e.g. dislocations), no crystal orientations, comparable to dark field TEM
EBSD	1 … 2 x 10^4	1 … 0.01	Positional sensitive detector in forward scattering position	Orientations, grain structure, defect fields (e.g. geometrically necessary dislocations), comparable to TEM diffraction

Table 1: Comparison of ECCI and EBSD

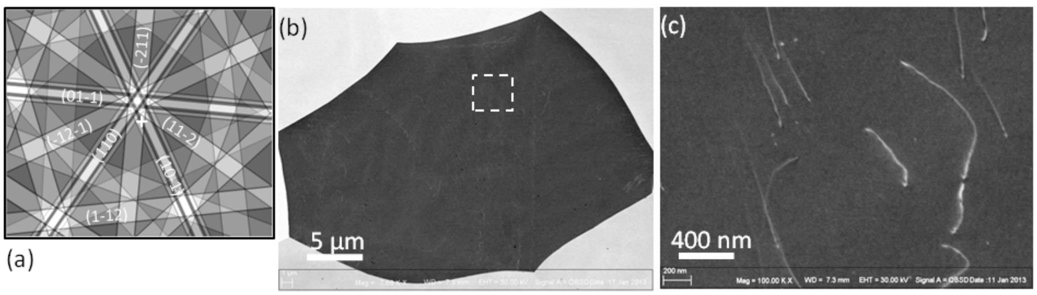

Figure 1. Dislocation observation by cECCI in an FeSi steel sample: (a) diffraction conditions, indicated in a simulated electron channelling pattern (b) Low-mag image of a grain. (c) Dislocation lines observed in the area indicated in (b).

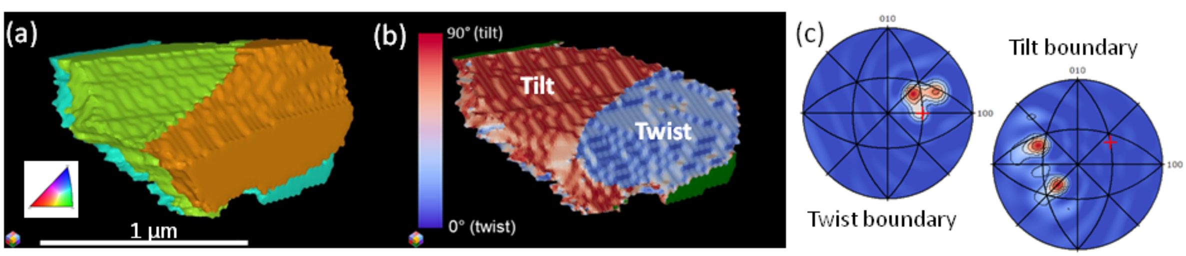

Figure 2. Grain boundary characterization in a FeNi sample using 3D EBSD:. (a) orientation map of 3 grains. (b) Tilt-twist colouring of the grain boundaries between the different grains in (a). (c) Stereographic projections of the grain boundary normals and misorientation rotation axis for the boundaries displayed in (b).

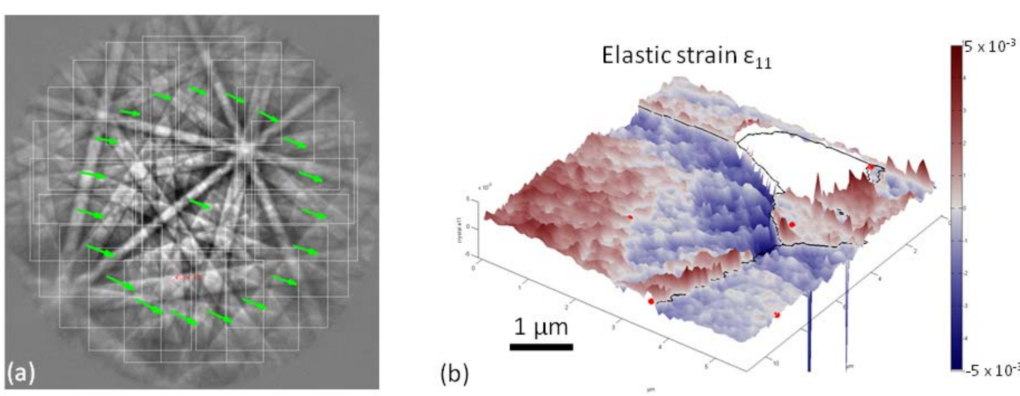

Figure 3. Characterization of elastic strains on a TWIP steel using cross-correlation EBSD. (a) EBSD pattern with arrows indicating the pole movement due to elastic strains. (b) Stress mapping of a small grain arrangement.

https://doi.org/10.1017/S1431927613010805 Published online by Cambridge University Press