Microstructural investigations of materials after severe plastic deformation by means of orientations mapping in TEM and SEM

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Abstract. Qualitative and quantitative microstructure descriptions of crystalline materials are crucial for characterization and better understanding of the mechanisms of deformation and recrystallization processes. In this study orientation mapping in SEM and TEM were applied to investigations of materials after plastic deformations by means of cold rolling and hydrostatic extrusion (HE) methods. Statistical analysis of local orientation, texture and grain boundary characterization were done for chosen materials of different symmetry like cubic aluminium and hexagonal titanium with high resolution capability down to a few square nanometres. It was proved that TEM/ Orientation Mapping (OM) investigations can be successfully applied for complementary analysis to conventional SEM/EBSD.

1. Introduction

Properties of the crystalline materials depend on their microstructure. Materials after plastic deformation are characterised by reduction of grain size and high density of dislocations. Microstructure descriptions need to be performed in a unified manner with resolution adjusted to grain size dimensions. Introducing the SEM/EBSD technique to microstructural analysis, made the process more versatile and comparable. Thanks to that many characteristics of the materials can be obtained and the influence of processing on the properties can be better described. However, the Orientation Mapping (OM) technique in TEM has also been developed lately, in addition to EBSD studies in SEM. While TEM mapping is not so meaningful from a statistical point of view (such as the measurement of texture), it is well suited for local detailed analysis critical to particular problems.

In this paper, two metals of different crystal structure fcc (aluminium 6013) and hcp (titanium grade 2) were investigated after plastic deformation by cold-rolling and hydrostatic extrusion (HE), respectively. Both materials were characterised by large strain applied and strong enhancement of mechanical properties.

2. Material and methods

Materials after severe deformation are, because of their very good properties, especially interesting for researchers. The classical EBSD methods are generally insufficient for the observation and understanding of mechanisms of deformation and recrystallization in highly
deformed materials. Investigations performed by means of transmission electron microscopy (TEM) in cases when the high resolution facilitated this method is necessary are useful supplements to SEM/ESBD. However, in order to obtain statistical information, a combination with EBSD analysis is required.

For several years, many attempts were made to create TEM systems (using different kinds of diffraction patterns or dark field images), including those that were commercialised [1-5]. Also for the SEM, the new technique of Transmission Kikuchi Diffraction (TKD) was developed [6-7]. Yet none of the considered systems are as common as conventional EBSD. In this paper, the results from investigations performed by means of conventional EBSD together with OM in TEM are presented.

Samples for investigations were chosen from the two groups of different symmetries and different deformation method. Aluminium alloy 6013, after cold-rolling up to 90 %, and pure Titanium grade 2 after HE were investigated. In both cases the material was prepared for observation in TEM and SEM by standard polishing and electropolishing methods.

![Schematic of the Orientation Mapping system in the TEM](image)

**Fig. 1.** Schematic of the Orientation Mapping system in the TEM

For SEM/EBSD investigations, the FEI Quanta 3D FEG SEM was used, equipped with the EDAX TSL OIM system for data collection and analysis.

For orientation mapping in the TEM, the system established in OML Skawina was used [8]. This system is based on a FEI TECNAI G20 transmission electron microscope with Lab6 cathode and equipped with a GATAN ORIUS SC200 camera. It operates on the same idea as the previously built systems presented in [3,8] with Digital Micrograph scripts to control the microscope activities. The new commercial software KikSpot was used to index sets of diffraction patterns [9]. Unlike in previous investigations [10-12] both spot and Kikuchi line type diffraction patterns (fig.1), obtained in different TEM modes, were indexed. In [8], results from the application of convergent beam Kikuchi type electron diffraction patterns for the precise analysis of changing orientations, with angular resolution about 0.1 ° are
presented. In this research, spot patterns in microdiffraction mode were used to obtain better spatial resolution (less than 10 nm) than in EBSD systems.

For statistical analysis OIM Analysis 7.2 software was used in both cases to obtain good compatibility of the statistics.

3. Results and discussion

After deformation aluminium alloy 6013 presents grains elongated in the rolling direction (RD) and a bimodal distribution of precipitation. In the laminar microstructure of the matrix, small (<=1 μm) and large (1-3 μm) precipitates of other phases are distributed. In the region surrounding large particles, deformation zones (DZ) formed, and these play important roles in the recrystallization process [11]. From this point of view, it is most interesting to analyse the orientations and grain size in these areas. Conventional methods of EBSD were applied with a step size below 100 nm (fig. 2a) to very high quality prepared surfaces of those samples. The presented map shows that good quality patterns were not possible to obtain from the DZ areas. Only matrix grains could be characterized using the SEM/EBSD method. From the same material, thin foils were prepared to perform TEM investigation. The microdiffraction spot patterns were acquired from the particular area with a step size of 5 nm, and indexed using KIKSpot software. From the orientation map obtained in that way, particular features of the microstructure are visible. It is observed that RD elongated grains become curled around the second phase particles, and are narrower. Their dimensions in the directions perpendicular to the RD are less than 100 nm. The grain misorientations between neighbouring bands are characterized by High Angle Grain Boundaries (HAGB). Apart from that, very small equiaxed grains are visible in the closest vicinity of precipitations with diameter less than 50 nm (fig.2.b). These could be responsible for nucleation of new grains in that area which are presented in [11].

![Fig. 2. Aluminium alloy 6013. (a) Orientation map (IPF coloring) from SEM/EBSD with IQ (Image Quality) map. (b) TEM Bright Field image with orientation map, grain size distribution and misorientation changes across the marked lines 1 and 2, DZ – Deformation Zone, P-Particle](image)

The second investigated example was pure titanium grade 2, examined after HE. It possesses a strong fiber texture with grains elongated in the direction of extrusion (ED). Observation from SEM/EBSD maps reveals that average grain size is about 11 μm, and shows the existence of local areas with very high dislocation density, which cannot be resolved by
conventional SEM/EBSD (fig. 3c). These “black spaces” on EBSD maps correspond to the low Confidence Index of the pixels which do not form the grains. Observations of the thin foils cut from the bulk samples were performed using TEM. On the bright field images it could be observed that, among the elongated grains in the ED, other, smaller ones were also visible. It was not possible to index the acquired Kikuchi diffraction pattern in those areas of smaller grains, because of strong diffusion of the lines on diffraction (Fig. 3c). However, with the help of microdiffraction analysis, it was possible to obtain orientation maps (Fig. 3b). The average grain size diameter for this map was about 380 nm and HAGB fraction was on the level of 0.8. In Fig. 3. c) grains with diameters about 100 nm are coloured black. Additionally, the orientation of the grains in those areas deviates strongly from <10-10> direction. This means that local TEM characterisation of the microstructure brings completely new information about the investigated material which was not resolved by the SEM/EBSD mapping.

**Fig. 3.** Titanium grade 2. (a) Orientation map (IPF coloring) from SEM/EBSD enlarged part with low CI (black), (b) TEM orientation map, (c) TEM Bright Field image with diffractions patterns of two types (d), grain size distribution with marked grains with diameter about 100 nm

### 4. Conclusions

The orientation mapping with the TEM is an appropriate tool for qualitative and quantitative characterisation of microstructure at nanoscale for materials after severe plastic deformation. It closes the gap between EBSD/ SEM and BF/DF TEM with spatial and angular resolution on the nanolevel.

A combination of both diffraction techniques provides a complementary characterization of material microstructure, especially with high density of dislocations produced after plastic deformations.
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