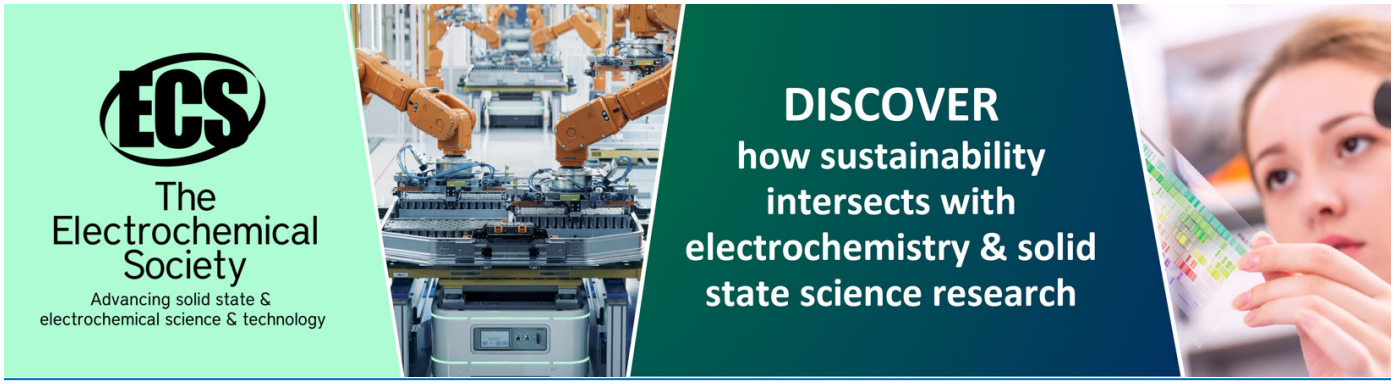
EBSD analysis of the microtexture of Ba-hexaferrite samples



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EBSD analysis of the microtexture of Ba-hexaferrite samples

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Abstract. The microtexture of differently prepared Ba-hexaferrite samples is investigated by means of electron backscatter diffraction (EBSD). Kikuchi patterns are obtained with a high image quality, enabling a spatial resolution of the EBSD maps of about 20 nm. The spatially highly resolved EBSD mappings provide additional information (individual grain orientation, misorientation angles, grain size distribution) as compared to the standard analysis techniques, which can contribute to an optimization of the growth process. Furthermore, as the crystallographic orientation of each grain is known, an exact analysis of the grain aspect ratio becomes possible which provides further insight to the microstructural dependence of the magnetic properties of ferrites.

1. Introduction

The analysis of the achieved texture is of great importance for ferrite materials, either bulk or thin films. The magnetic properties were found to be strongly dependent on the orientation, size and shape of the crystallites, and sample homogeneity plays an important role as well [1,2]. Furthermore, in the case of hexaferrites, a strong texture of the *c*-axis is important especially in applications of next generation microwave devices [3,4]. The recently developed electron backscatter diffraction (EBSD) technique, which works within a scanning electron microscope, enables a spatially resolved study (a resolution of about 20 nm is possible on perfectly prepared surfaces of oxidic materials [5,6]) of the crystallographic orientations by means of recording of Kikuchi patterns. A good surface polishing/cleaning is essential for this analysis, as the method requires an undisturbed surface area for a high image quality. This information is recorded to each measured Kikuchi pattern, together with a parameter describing the quality of indexation. In the present contribution, we will analyse two differently prepared samples by means of EBSD.

2. Experimental procedure

The EBSD system employed here consists of a FEI dual beam workstation (Strata DB 235) equipped with a TSL (TexSEM Labs, UT) analysis unit. The Kikuchi patterns are generated at an

acceleration voltage of 20 kV, and are recorded by means of a DigiView camera system. To produce a crystallographic orientation map, the electron beam is scanned over a selected surface area and the resulting Kikuchi patterns are indexed and analysed automatically. A detailed description of the measurement procedure can be found in Ref. [7]. Automated EBSD scans were performed with a step size down to 10 nm; the working distance was set to 10 mm.

The sample surfaces were treated by mechanical polishing down to 0.25 µm diamond paste, followed by polishing with colloidal silica solution (Struers, OP-S). The final surface finishing was made by Ar-ion polishing; the details of this method will be discussed in detail elsewhere [8].

The sample N2 was prepared by conventional ceramic processing [9], whereas sample N3 was prepared by a one-step topotactic reaction technique that will be published elsewhere [10].

3. Results and discussion

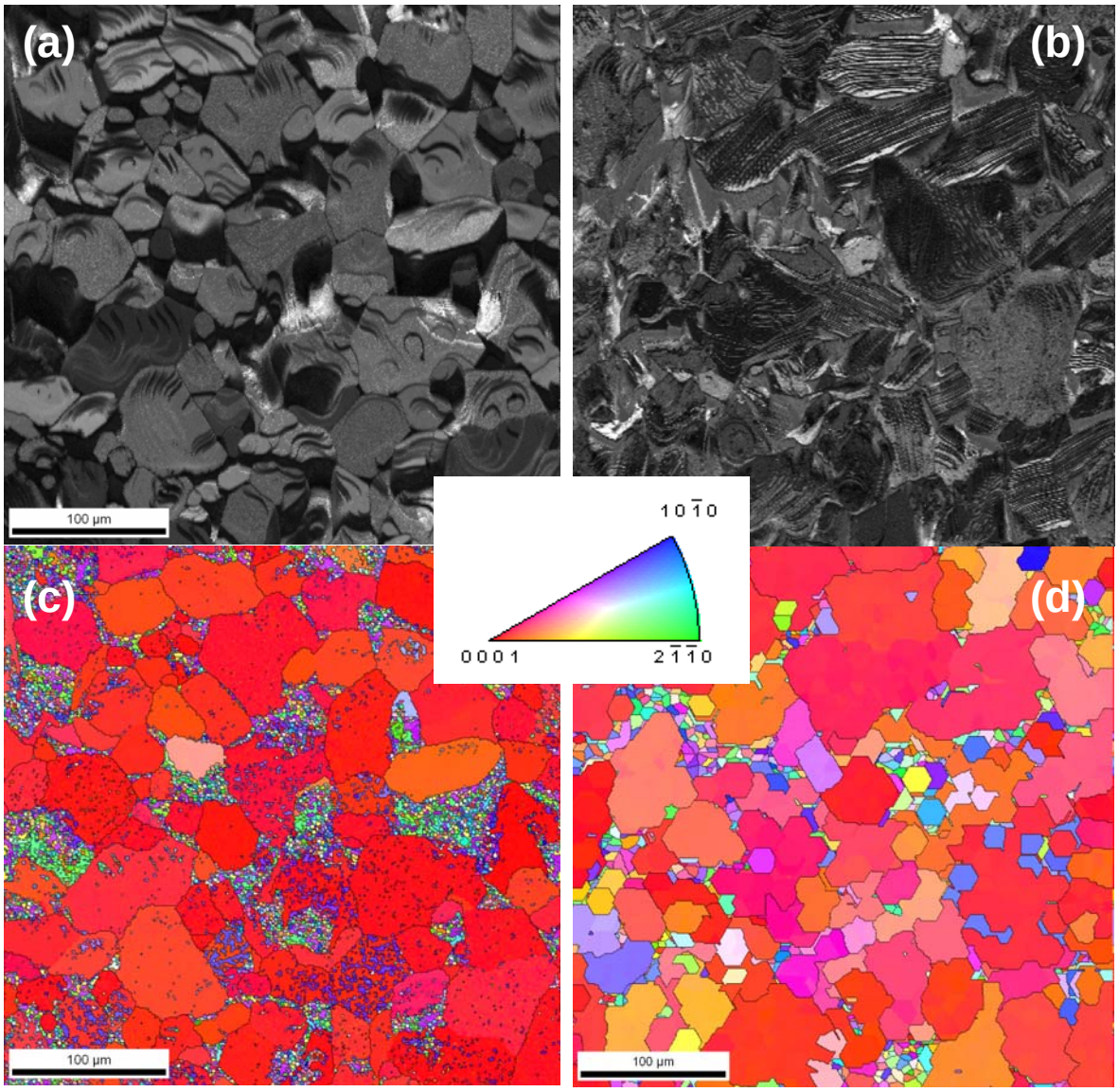

Figure 1. IQ-maps of sample N21 (a) and N3 (b) and the IPF maps of the same area in (0001)-direction. The colour code for the crystallographic orientation is given in the stereographic triangle.

Figure 1 presents image quality (IQ) maps of both samples, which resemble backscatter electron images (under EBSD conditions, i.e., 70° tilt) and inverse pole figure (IPF) maps which give the crystallographic orientations. The large grains in (c) show a certain texture, but there is a large amount of misoriented small grains acting as a filler material.

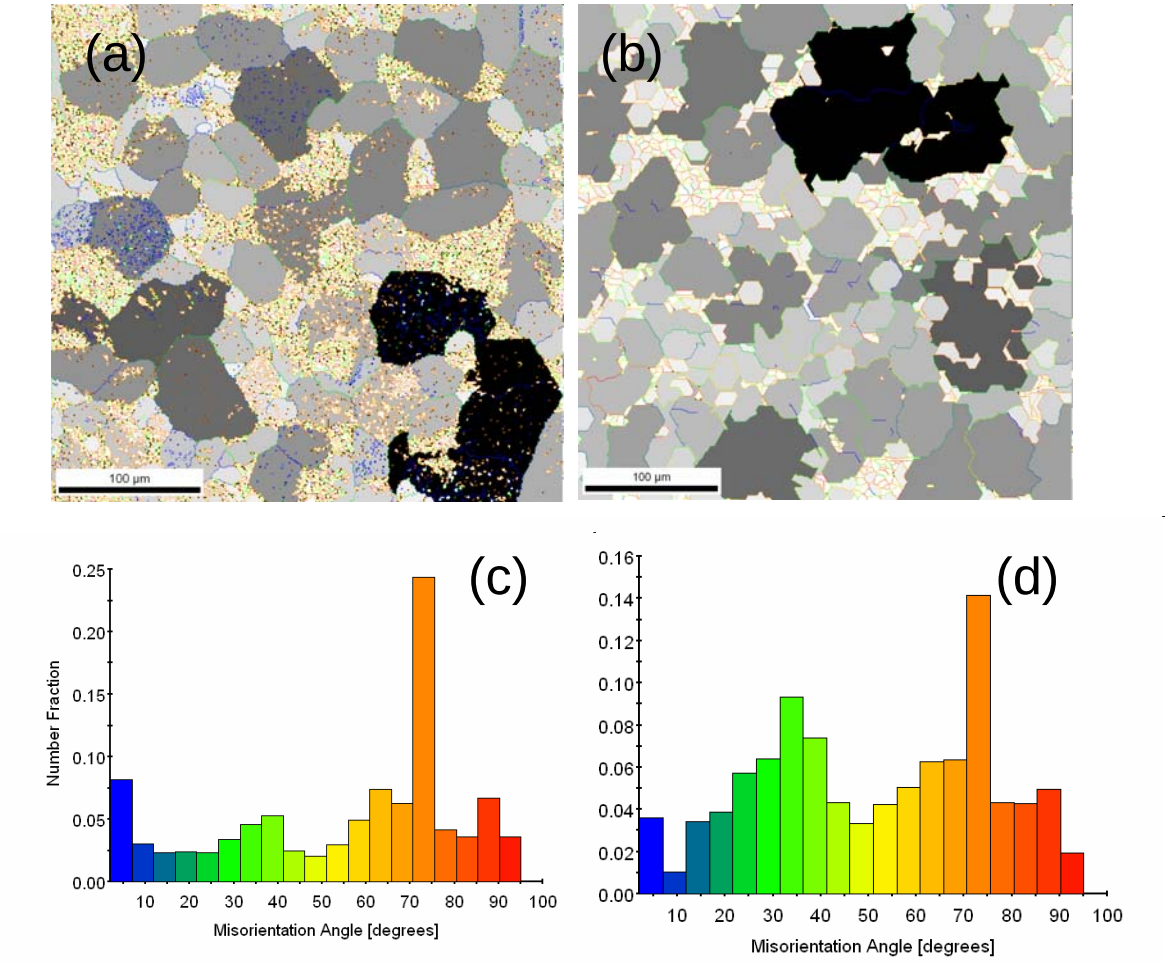

Figure 2. Grain size maps (a,b) of both samples (black – large, ranging to white – small, see also Fig. 3), together with the misorientation boundaries marked in colour according to the misorientation angle histograms (c,d) given below the maps. Note also the different scale of the two histograms.

In Fig. 1 (d), the situation is much more homegenous, which becomes even more obvious regarding the maps of Fig. 2. Here, grain size maps of both samples are given (a,b), together with the misorientation boundaries marked in colour, according to the histograms (c,d). The large ferrite grains of sample N2 exhibit a large number of small misorientations (blue, green), and around the small grains are many boundaries in the range between 40° and 60° (yellow, orange). In contrast to that, the ferrite grains in sample N3 do not show any misoriented subgrains. The higher angle grain boundaries (orange, red) are only occurring in between the remaining smaller grains, so that the overall grain structure is quite homogeneous. Between the ferrite grains mostly 30° misorientation boundaries (green) are observed. These observations are summarised in the histograms shown in Fig. 3. The misorientation histogram shows that sample N2 exhibits a large number of small angle misorientations, while sample N3 exhibits a maximum at 35°. Otherwise, the two histograms look similar. The grain size histogram indicates the large amount of small grains (2 – 5 µm), while sample N3 shows a large number of grains in the range between 60 and 100 µm.

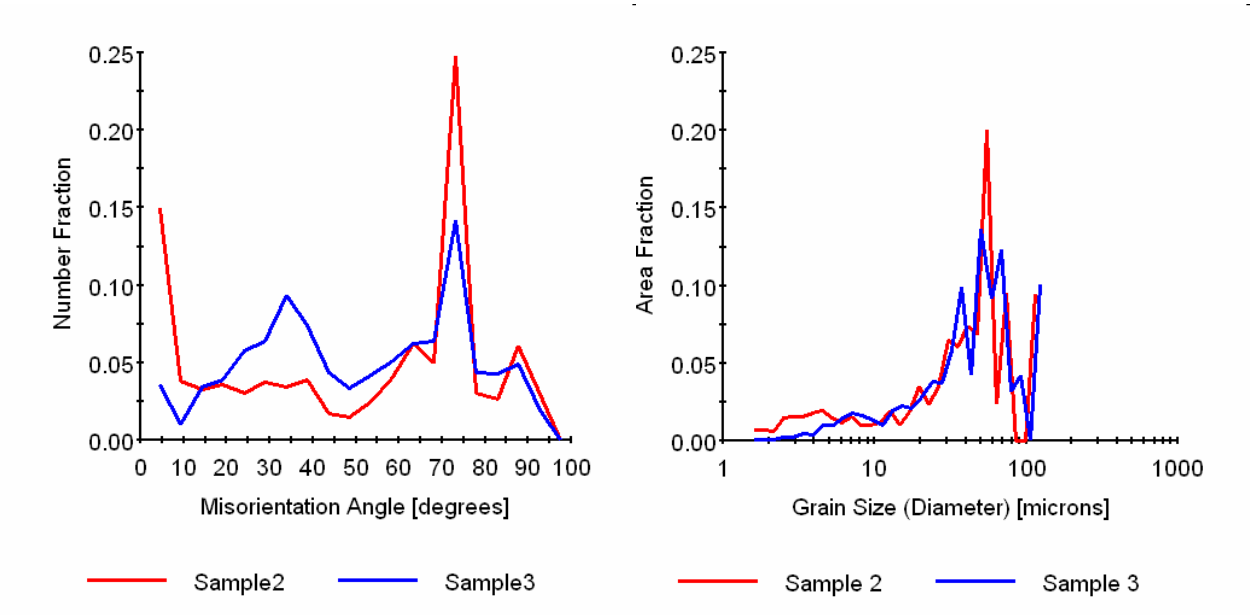

Figure 3. Misorientation angle histogram for both samples (left), and grain size histogram for both samples (right).

In this way, the achieved microstructures can be characterized in detail, which provides important information additionally to the magnetic characterisation.

The analysis of the local distribution of misorientations is the advantage of the EBSD technique, providing spatially highly resolved data. Therefore, important conclusions concerning the various fabrication methods can be drawn.

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