Grain orientation of $YBa_2Cu_3O_x$ high-T_c superconductors studied by OIM

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Abstract

Orientation imaging microscopy (OIM) provides a method for measuring a large number of individual grain orientations and relating them directly to the microstructural features by means of evaluating electron backscatter Kikuchi patterns in scanning electron microscopy. We investigated the grain orientation distributions of various YBCO ceramic samples prepared with different Alkali metal additions. The samples are characterized by pole figures, inverse pole figures, and grain orientation maps. Grain orientation distribution functions are obtained from the measured data. The $KClO_3$-doped samples are shown to exhibit a texture for an addition in the initial batch between 3 and 5 wt.-%. This observation explains the increased critical current density in the doped samples.

Key words: EBSD; $YBa_2Cu_3O_7$; polycrystals; orientation analysis

The investigation of the microstructure of ceramic, polycrystalline high-T_c superconductors plays an important role to understand the behaviour of the critical current density, $j_c(B)$. Especially the performance of bulk samples of the RE-123 type (i.e. $YBa_2Cu_3O_{7-\delta}$ (YBCO) or $NdBa_2Cu_3O_{7-\delta}$ (NdBCO)) relevant for applications (e.g. microwave resonators, trapped field magnets, fault current limiters and wires) are severely limited by the presence of grain boundaries [1] with different misorientation angles. High angle grain boundaries were found early to act as weak-links, but also low angle grain boundaries and subgrain boundaries pose serious problems in large magnetic fields.

Therefore, grain boundaries form severe obstactles for the current flow, thus in the past years a lot of efforts were invested to improve the texture and grain connections within these bulk samples [2]. An important step for the improvement of the transport properties of the grain boundaries is given by chemical doping, as described recently by Mannhart *et al.* [3] By means of doping YBCO thin films with Ca, these authors showed that the current density across the grain boundaries could rise by a factor of about 6. These investigations were, however, performed on YBCO thin films with a single grain boundary. For the development of bulk materials, it is therefore extremely important to study the effects of doping on bulk, polycrystalline samples aimed for applications. Therefore, a direct correlation of the local microstructure (grain size and orientation) with the magnetic properties of the samples is a very important task.

	Dopant	x	Sintering temp. [°C]	T_c [K]
Sample A	pure YBCO	–	980	91.3
Sample B	$KClO_3$	0.30	915	93.8
Sample C	K_2CO_3	0.75	940	93.4
Sample D	Rb_2CO_3	0.40	920	94.6

Table 1
List of the $YBa_2Cu_3O_{7-\delta}$-samples studied here.

An useful tool to achieve this goal is given by the recently developed electron backscatter diffraction (EBSD) or, sometimes called orientation imaging

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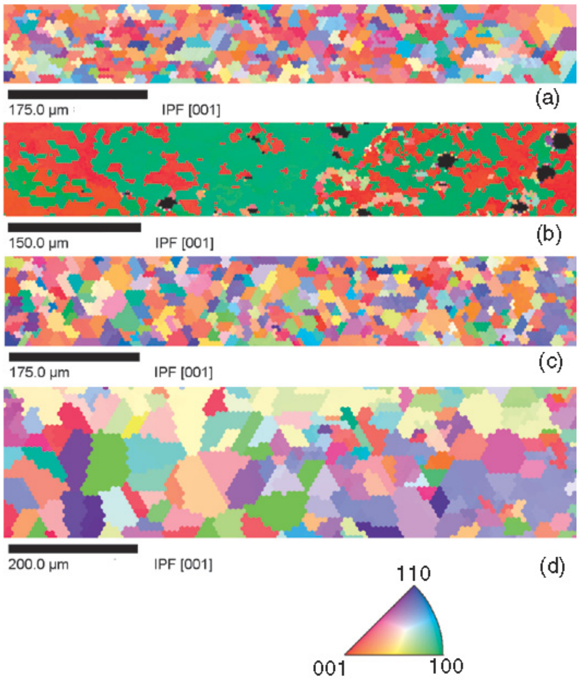

Fig. 1. Inverse pole figure (IPF) maps of the YBCO samples A, B, C and D in (0 0 1) direction, i.e. the "grains" are shaded by their respective crystallographic orientation normal to the sample surface. The orientations are given in the color-coded stereographic triangle.

(OIM) technique [4–6], which enables the orientation mapping of granular samples by means of automatized recording of Kikuchi patterns. EBSD bridges the two traditional methods of studying crystalline materials, X-ray crystallography and transmission microscopy, and adds the benefit of high spatial resolution at any point on the surface of a bulk specimen.

Backscatter Kikuchi patterns are generated in a commercial EBSD system (TSL TeXSEM Laboratories [7]) using a phosphor screen mounted in a commercial SEM microscope (CamScan Series 4) by back-diffraction of a stationary beam of high-energy electrons from an almost perfect volume of crystal. To produce a crystallographic orientation map, the electron beam is scanned over a selected surface area and the resulting Kikuchi patterns are automatically recognized and indexed by the controlling computer [7].

Four samples (samples A - D, see table 1) were chosen for this study, which correspond to 5 wt.-% additions in the initial batch yielding the best superconducting properties of each series. After the final baking, AAS revealed only traces of K (about 4×10^{-2} wt.-%) and Rb (about 2×10^{-2} wt.-%). The details about the sample preparation can be found in Refs. [8,9]. For use with the EBSD analysis, the sample surfaces required mechanical polishing, which was performed on dry polishing papers by 3M and only ethanol as lubricant and for cleaning purposes. Details of this procedure were described in detail in Ref. [10]. Figure 1 shows the so-called inverse pole figure (IPF) map in (0 0 1) direction, i.e. the "grains" are shaded by their respective crystallographic orientation normal to the sample surface. The corresponding grain orientations are given in the associated color-coded stereographic triangle. The pure YBCO sample A (a), and the carbonate-doped samples C and D (c,d) show more or less a random orientation; whereas in stark contrast to this the map of sample B (b) reveals that the two orientations, (0 0 1) and (1 0 0) are dominating. We explained the influence of $KClO_3$ and the carbonates K_2CO_3 and Rb_2CO_3 on the properties of the microstructure in terms of different interaction in which $KClO_3$ and the carbonates participate up to 900 °C [8,9]. We assume that the beneficiary effect of the $KClO_3$-addition on the microstructure of YBCO is a result of the presence of a liquid phase of KCl during the processing stage [9].

In conclusion, EBSD mapping on four different polycrystalline YBCO samples was successfully performed. The pure and the carbonate-doped YBCO samples did not show any preferred grain orientation, whereas on the $KClO_3$-doped YBCO sample, we found an increased texture with the (0 0 1) and (1 0 0) directions dominating. These measurements illustrate nicely the importance of EBSD measurements to provide a deeper understanding of the doping effects in high-T_c superconductors.

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