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EBSD characterisation of $Y_2Ba_4CuUO_x$ phase in melt-textured YBCO with addition of depleted uranium oxide

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Abstract. Melt-textured YBCO samples processed with added Y_2O_3 and depleted uranium oxide (DU) contain nano-particles, which have been identified previously as $Y_2Ba_4CuUO_x$ (U-2411). This phase has a cubic unit cell, which is clearly distinct from the orthorhombic Y-123 and Y-211 phases within the YBCO system. In samples with a high amount of DU addition (0.8 wt-% DU), U-2411 particles have sizes between 200 nm and several µm, so identification of the Kikuchi patterns of this phase becomes possible. Together with a parallel EDX analysis, the particles embedded in the Y-123 matrix can be identified unambiguously. In this way, a three-phase EBSD scan becomes possible, allowing also the identification of nanometre-sized particles in the sample microstructure.

1. Introduction

The recent development of melt-textured Y-Ba-Cu-O (YBCO) samples with embedded $Y_2Ba_4CuUO_x$ (U-2411) nanoparticles [1,2] produces new types of specific microstructures. In former experiments, the crystallographic orientations of the embedded Y_2BaCuO_5 (Y-211) particles were studied by means of EBSD, requiring a two-phase scan [3,4]. This analysis resulted in important information about the interplay between the Y-123 and the Y-211 phase [5]. Now, in order to enable a three-phase EBSD analysis, the Kikuchi pattern of the cubic U-2411 phase must be identified. Here, we show that this identification became possible using YBCO samples with a high amount of depleted uranium oxide (DU) addition (0.8 wt-% DU), so that the resulting U-2411 particles have sizes between 200 nm and several µm. Together with a parallel performed EDX analysis, the particles embedded in the Y-123 matrix can be identified unambiguously.

2. Experimental procedure

Melt-textured YBCO samples were produced using a standard top seeded melt growth (TSMG) procedure [6], but with various additions of DU (0.1 − 0.8 wt-%) and 30 mol-% Y_2O_3 to the starting powder prior to melt-processing. The fully melt processed samples were oxygenated in a separate process in the usual way to obtain the superconducting $YBa_2Cu_3O_{7-x}$ phase (Y-123). The surfaces were subsequently dry-polished using 3M polishing papers as described in Ref. [7], using only ethanol for cleaning purposes. This procedure yields high image quality Kikuchi patterns and has been demonstrated to work well even on various polycrystalline YBCO samples [7,8].

The EBSD system consists of a FEI dual beam workstation (Strata DB 235) equipped with a TSL OIM analysis unit [9]. The Kikuchi patterns are generated at an acceleration voltage of 20 kV, and are recorded by means of a DigiView camera system, allowing a recording speed of the order of 0.1 s/pattern. The time may be slightly longer in the case of a multi-phase scan. 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 (i.e. the Kikuchi bands are detected by means of the software). An image quality (IQ) parameter and a confidence index (CI) are recorded for each such Kikuchi pattern. A detailed description of the measurement procedure can be found in Refs. [7,8].

3. Results and discussion

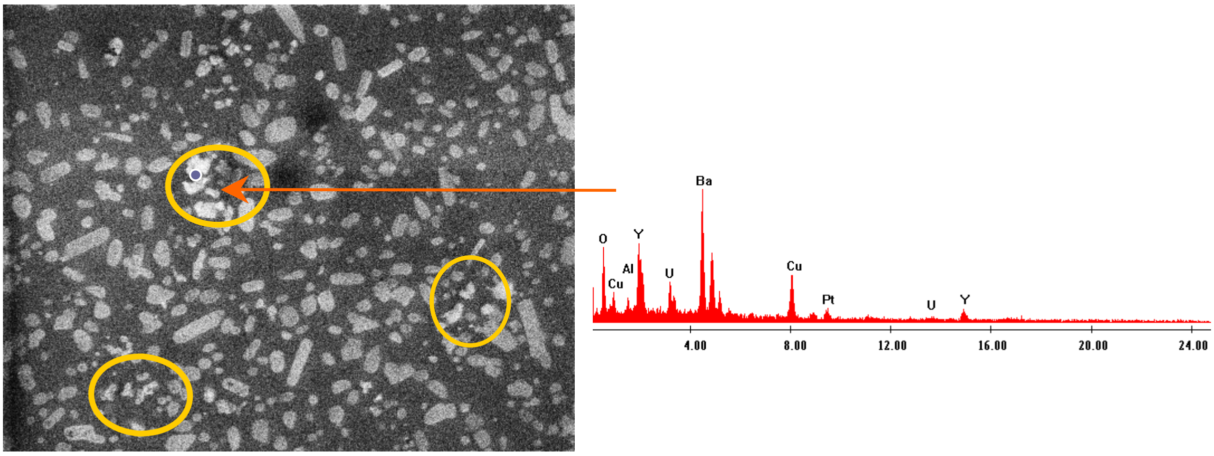


Figure 1. SEM/EDX-analysis of the YBCO sample with 0.8 wt.-% DU addition. The encircled areas are found to contain U-2411 particles. On the left, the corresponding EDX analysis is presented.

Figure 1 gives the SEM/EDX-analysis of the YBCO sample with 0.8wt.-% DU addition. Here, some large U-2411 particles are found which can also be observed using optical microscopes [10] due to their specific shape. The EDX analysis reveals the presence of U, Ba, Y, Cu within these particles. Therefore, these particles were used to obtain the Kikuchi patterns of the U-2411 phase.

The interesting areas were then investigated by EBSD using the manual interactive mode. In this way, the Kikuchi pattern of the U-2411 phase can be unambiguously identified. In fig. 2, the detected Kikuchi patterns of the U-2411 phase are shown, together with the indexing obtained using an adapted X-ray data (PDF) file no. 480120 [11].

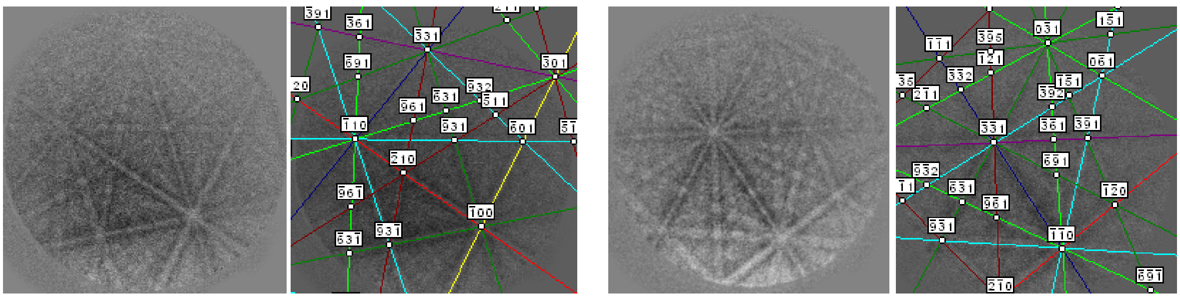
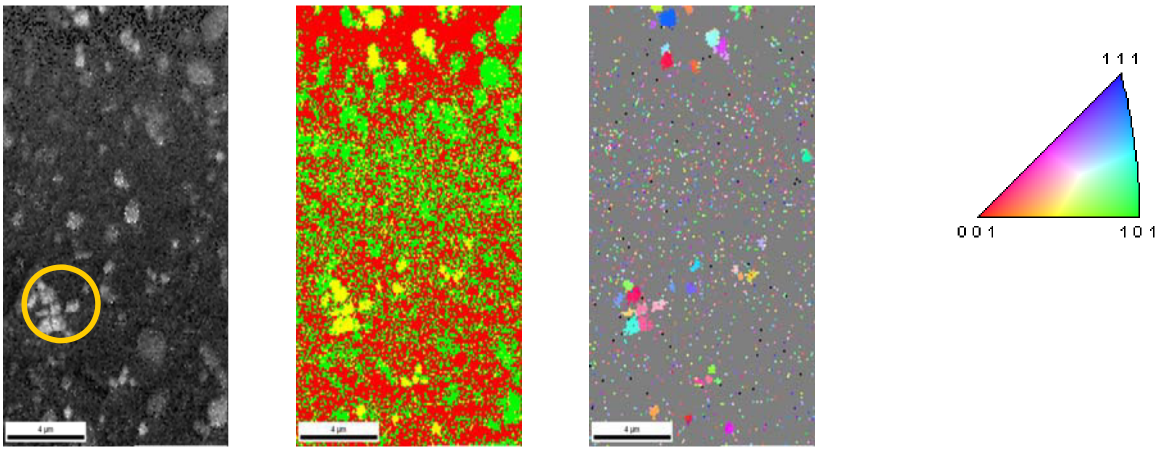


Figure 2. Kikuchi patterns with the indexing of the U-2411 phase.

The U-2411 phase is cubic, with a unit cell length a = 8.71 Å, which makes the Kikuchi pattern of this phase clearly distinct from the orthorhombic Y-123 and Y-211. Therefore, together with the high image quality values achieved, an automated three-phase EBSD scan becomes possible.

Finally, Fig. 3 shows the results of the EBSD three-phase mappings. The leftmost map is a so-called image quality (IQ)-map, which gives an information about the Kikuchi pattern quality to each measured datapoint. The encircled area was identified before by means of EDX as U-rich particles, and the corresponding EBSD phase map (middle) clearly shows the presence of the U-2411 phase (indicated in yellow). The Y-123 phase is shown as red, and the embedded Y-211 particles as green. Most of the U-2411 particles are indeed found to be very small with sizes down to 200 nm. Finally, on the right an inverse pole figure (IPF map) presents the crystallographic orientations of the U-2411 particles in [0 0 1] direction; the corresponding orientations are indicated in the stereographic triangle. The U-2411 particles do not show a preferred orientation, analogous to the embedded Y-211 particles as found earlier [3,4].



IQ map **Phase map** **IPF (001) map U-phase**

Figure 3. EBSD three-phase mapping of an area identified before via EDX to contain U-2411 particles. These particles are well identified by the cubic Kikuchi pattern. The left map is an image quality (IQ) map, the yellow circle denotes particles identified by EDX to be U-2411. The middle map gives the phase map (Y-123 – red, Y-211 – green and U-2411 – yellow). The right map is an orientation (IPF) map of the detected U-2411 particles.

From the measured EBSD datapoints, the grain sizes of the U-2411 phase can be determined. Provided that the EBSD datapoints are measured on a fine grid (typical stepsize 50 nm), the size distribution of the embedded nanoparticles can be determined. Figure 4 shows that the size of most U-2411 nanoparticles is around 500 nm, while some particles have sizes around 100-150 nm. This allows them to be already quite effective flux pinning sites as compared to the Y-211 particles.

4. Conclusions

We have presented the identification of the Kikuchi patterns of the U-2411 phase, embedded in a superconducting Y-123 matrix. In this way, a true automated EBSD three-phase becomes possible. The crystallographic orientation of the U-2411 particles in Y-123 samples with addition of DU is found to be random, similar to the Y-211 particles.

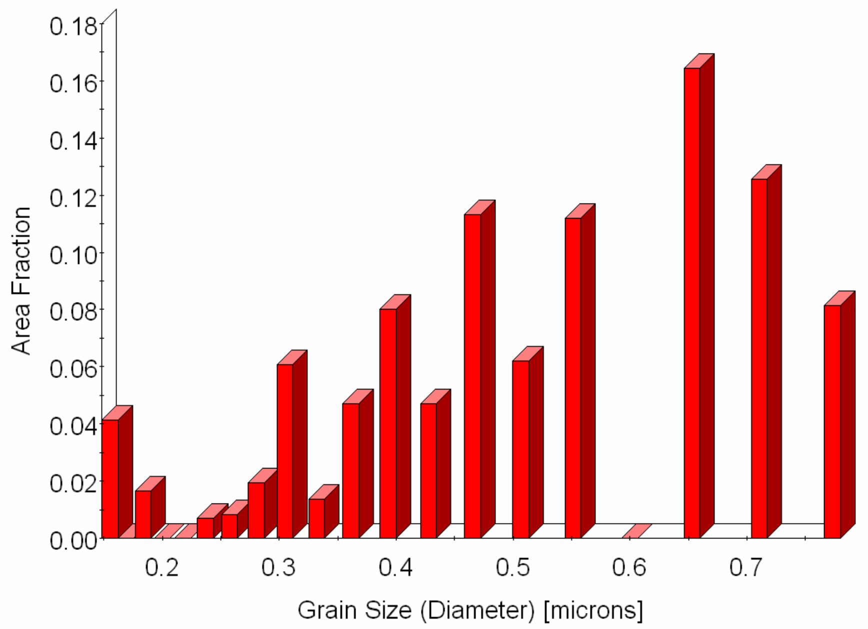


Figure 4. Grain-size histogram of the U-2411 phase as determined by the EBSD scan.

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