Analysis of melt-textured YBCO with nanoscale inclusions

M R Koblischka\textsuperscript{a}, M Winter\textsuperscript{a}, U Hartmann\textsuperscript{a}, A Koblishka-Veneva\textsuperscript{b}, N Hari Babu\textsuperscript{c} and D A Cardwell\textsuperscript{c}

\textsuperscript{a} Institute of Experimental Physics, University of Saarbrücken, P.O. Box 151150, D-66041 Saarbrücken, Germany.
\textsuperscript{b} Institute of Functional Materials, University of Saarbrücken, P.O. Box 151150, D-66041 Saarbrücken, Germany.
\textsuperscript{c} IRC in Superconductivity, University of Cambridge, Madingley Road, Cambridge CB3 0HE, U.K.

m.koblischka@mx.uni-saarland.de

Abstract. Recently, particles with the chemical composition Y\textsubscript{2}Ba\textsubscript{4}Cu\textsubscript{M}O\textsubscript{x}, where \textit{M} = U, Nb, Zr, etc., and sizes in the range of 50 – 200 nm have been generated within the YBCO matrix of bulk, melt-processed superconductors in order to serve as effective flux pinning sites. By means of AFM and electron backscatter diffraction (EBSD) measurements, we analyse the spatial distribution and the size distribution of these nanoparticles within the superconducting YBCO matrix.

1. Introduction

Y-Ba-Cu-O (YBCO) is currently still the best suited high-\textit{T}_c superconductor for most bulk applications, but a higher critical current density would be desirable. To achieve this goal, attempts have been made to refine the size of the embedded Y\textsubscript{2}BaCuO\textsubscript{5} (Y-211) particles within the superconducting YBa\textsubscript{2}Cu\textsubscript{3}O\textsubscript{7} (Y-123) phase matrix \cite{1-3}. However, these particles are generally too big to be effective flux pinning sites, except in low magnetic fields. Furthermore, there is a negative influence of these particles on the growth of the superconducting Y-123 matrix, which could be clearly demonstrated by electron backscatter diffraction (EBSD) analysis \cite{4}. Recently, particles with the chemical composition Y\textsubscript{2}Ba\textsubscript{4}Cu\textsubscript{M}O\textsubscript{x} (\textit{M}-2411), where \textit{M} = U, Nb, Zr, etc., have been generated within the YBCO matrix \cite{5,6}. These true nanoparticles have sizes in the range of 50 – 200 nm, and provide therefore a much better approach to generate effective flux pinning sites within the bulk, melt-textured YBCO samples.

In this contribution, we analyse the spatial distribution and the size distribution of these particles by means of AFM and EBSD measurements.

2. Experimental procedure

We employed Digital Instruments Nanoscope III and IV controllers in atomic force microscope (AFM) mode at ambient conditions. For comparison, AFM scans were performed in contact mode and tapping mode using doped Si-cantilevers. A Q-control unit was used to improve the signal-to-noise ratio in the tapping mode. EBSD analysis was carried out using a FEI dual-beam microscope equipped with a TSL EBSD analysis unit as described elsewhere \cite{7}.
The melt-textured (LRE)BCO [(LRE) = light rare earths) samples were produced using the standard procedure [8]. Nanopowders of Nb-2411 and Zr-2411 were added prior to the melt-texturing. For this investigation, a sample with 2.5 mol% Nb-2411 and a sample with 30 mol-% Zr-2411 were chosen.

Since the as-grown surfaces of the samples were usually too rough to achieve good scanning results, the samples were polished prior to scanning, either dry from 12 µm to 0.5 µm diamond paper or wet from 320 grain SiO paper to 4000 grain SiO paper and then from 3 µm diamond polishing solution down to 40 nm colloidal silica (OP-S) [9]. After that, the samples were cleaned for several minutes in acetone in an ultrasonic bath and then for several minutes in an isopropanol bath. This surface preparation method serves well for both types of measurements.

3. Results and discussion

Figure 1. AFM scans of the ab-plane of the melt-textured YBCO samples. The upper image shows a YBCO sample containing Zr-2411 nanoparticles. The lower two images present a YBCO sample containing Nb-2411 nanoparticles.
Figure 1 presents the results of AFM scans on the polished \textit{ab}-planes of the melt-textured YBCO samples. The embedded nanoparticles are clearly visible within the YBCO matrix. The upper image shows a YBCO sample containing Zr-2411 nanoparticles with an average particle diameter of 40 nm and an area density of 35 µm\(^2\); the average particle height is 15 nm. The lower images present a YBCO sample containing Nb-2411 nanoparticles with an average particle diameter of 30 nm and area density of 280 µm\(^2\); the average particle height is 6 nm. The distribution of the nanoparticles is still relatively inhomogeneous; there are several areas without any nanoparticles in the samples.

Figure 2. AFM scans of on a YBCO sample with U-2411 nanoparticle inclusions. These particles are somewhat bigger (~200 nm diameter). Here, it is clearly visible that the particles are arranged in stripes due to the twin boundaries within the superconducting matrix.

Figure 2 presents detail measurements, indicating the size and shape of the embedded nanoparticles. As already observed earlier, the nanoparticles are influenced by the arrangement of the twin boundaries within the superconducting matrix, leading to a stripe-like arrangement.
Finally, figure 3 presents an EBSD three-phase analysis (Y-123, Y-211 and Zr-2411) of the sample with Zr-2411 inclusions. Map (a) is a SEM image of the selected area for analysis, map (b) is a so-called inverse pole figure (IPF) map giving the crystallographic orientation for all three phases. Clearly visible are here the twins in the superconducting Y-123. Map (c) represents the phase map, where Y-123 is indicated as red, Y-211 as green and the Zr-2411 nanoparticles as yellow. Finally, map (d) shows only the crystallographic orientation of the Zr-2411 nanoparticles, which are oriented in (001) direction, corresponding to the orientation of the YBCO matrix. This is in strong contrast to the observations on YBCO samples containing U-2411 nanoparticles [9]. Map (d) also reveals that the distribution of the nanoparticles is inhomogeneous, and strongly influenced by the presence of the twin structure within the YBCO matrix. The EBSD mappings reveal that the M-2411 nanoparticles do not disturb the growth of the superconducting Y-123 matrix, which makes these particles better suited as additional pinning sites.

4. Conclusions

By means of AFM analysis, we have observed embedded nanoparticles within the superconducting YBCO matrix in melt-textured superconductor samples. The nanoparticles have an average diameter of $30 \text{ -- } 40 \text{ nm}$, which is nearly optimum considering that the best flux pinning is achieved at a diameter of $2 \text{ nm}$ The arrangement of the nanoparticles is influenced by the twin boundaries within the superconducting matrix, as revealed by EBSD analysis.

![Figure 3](image)

**Figure 3.** EBSD-mappings on the sample with embedded Zr-2411 particles. (a) is a SEM picture, (b) the crystallographic orientation (IPF) map and (c) the phase map. (d) gives the orientation of the embedded nanoparticles, which are homogeneously oriented in (001)-direction.
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