Physica C 496 (2014) 35–38

Contents lists available at SciVerse ScienceDirect

Physica C

journal homepage: www.elsevier.com/locate/physc

Position-dependent analysis of nanostripes in bulk light-rare-earth superconductors

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ARTICLE INFO

Article history:
Accepted 18 July 2013
Available online 25 July 2013

Keywords:
Nanostripes
Ternary 123-type superconductors
Bulk materials
AFM
Texture

ABSTRACT

The light rare-earth based bulk superconductors were found recently to exhibit self-organized nanostripes, especially pronounced in the ternary compound (Nd,Eu,Gd)$Ba_2Cu_3O_x$. The nanostripes, consisting of aligned nanoclusters (dimensions 10–50 nm in diameter), are formed during the sample growth, so it is an essential issue for the production of such samples to be able to control the formation of these nanostripes in order to generate effective pinning sites at high magnetic fields. Here, we investigate bulk pellets with a diameter of 18 mm and a height of 10 mm. These pellets are characterized prior to the cutting by means of trapped field measurements. Small samples are cut from the bulk pellet in various growth directions and sample sectors. The nanoscopic investigations are performed using atomic force microscopy and scanning tunneling microscopy in ambient conditions.

1. Introduction

The various light rare-earth (LRE) based bulk superconductors were found recently to exhibit self-organized nanostripes, which are especially pronounced in the ternary compound (Nd,Eu,Gd)$Ba_2Cu_3O_x$ (abbreviated: NEG) [1–4]. The nanostripes, consisting of aligned nanoclusters (dimensions 10–50 nm in diameter), are formed during the sample growth process as demonstrated in Ref. [5], where atomic force microscopy (AFM) topography images of non-oxygenated samples were presented. The nanoclusters correspond to chains of nanoclusters formed by the light rare-earth (LRE)-rich phase [6,7]. These nanostripes may play an important role in flux pinning due to their dimensions in the nanometer range, which is especially important at elevated temperatures (77 K and above) and high applied magnetic fields. The flux pinning properties due to the nanostripes could already be demonstrated by means of *I*/*V*-measurements on the sister compound (Sm,Eu,Gd)$Ba_2Cu_3O_x$ (SEG) [8].

Therefore, it is an essential issue for the production of bulk LRE-123-type samples to be able to control the formation of these nanostripes in order to generate effective pinning sites at high magnetic fields. We investigate in this contribution samples cut from large, bulk pellets with a diameter of 18 mm and a height of 10 mm, while keeping the information of the original position within the sample. The NEG pellets were characterized in full by means of trapped field measurements prior to the microstructure investigations.

2. Experimental procedure

We employed Digital Instruments Nanoscope III and IV controllers in AFM mode and STM mode at ambient conditions. For comparison, AFM scans were performed in contact mode and tapping mode using micro-machined, doped Si-cantilevers (type PPP, Nanoworld Services GmbH, Erlangen, Germany). A Q-control unit was used to improve the signal-to-noise ratio in the tapping mode. STM scans were done using cut Pt/Ir-tips [9]. As both AFM and STM share the same scanner but employ different types of tips, this enables one to exclude influences of the cantilevers. Any effects of the measurement direction are excluded by employing different scan directions in both AFM and STM measurements. The tunneling voltages range between 0.1 and 0.5 V, sometimes up to 1 V. The tunnel currents are for most images below 1 nA.

The melt-textured NEG samples were obtained following the procedure described in Refs. [10,11]. The sample selected for this study is a $(Nd_{0.33}Eu_{0.38}Gd_{0.28})Ba_2Cu_3O_y$ sample with 3 mol% Gd_2BaCuO_5 (Gd-211) particles added together with Pt and 10 wt.% Ag. The exact overall sample size was 17.6 mm in diameter and 10 mm thickness.

In a first step, the bulk sample was cut in half through the center of the seed crystal using a diamond blade saw, and then the resulting surfaces were mechanically polished as described below. Then, a 1 mm thick slice was cut from one half of the bulk sample. From here, we selected the following cuts to be located within the

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http://dx.doi.org/10.1016/j.physc.2013.07.007

a- and *c*-growth sector sections. Small samples with dimensions of $1 \times 1 \times 1$ mm^3 were cut from the big pellet as sketched in Fig. 2; overall a total of 12 samples were selected for the investigation. From all small samples cut, we investigated the top surface in the following experiments. Since the as-grown surfaces of the samples were usually too rough to achieve good scanning results, the sample surfaces 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 suspension (Struers OP-AA) [12]. After that, the samples were cleaned for several minutes in acetone in an ultrasonic bath and then for several minutes in an ethanol bath. Both surface preparation methods serve well for the AFM/STM measurements, as well as for magneto-optic and electron backscatter diffraction (EBSD) analysis [13]. As shown previously, the resulting surface roughness after both mechanical preparation procedures can be of the order of 6–10 nm; additional chemical etching produces a much rougher surface. The influences of the surface preparation methods on the images were studied previously in Refs. [13,14].

3. Results and discussion

Firstly, the bulk sample was characterized by measuring the trapped field distribution using a scanning Hall probe setup. The sample is zero-field cooled to 77 K, and then energized by means of a magnet with a room temperature bore using a field of 2 T. The measured trapped field distribution is illustrated in Fig. 1. As expected, there is a large peak found at the sample center, indicating that the bulk sample is homogenous without microscopic cracks. The peak could be distorted or splitted up in case there would be cracks or other defects in the sample. The distribution of flux pinning sites within the sample influences the peak height and its shape, so the present sample shows a good and homogeneous pinning site arrangement, even though the maximum trapped field achieved here is not very high. A maximum trapped field of 0.22 T could be recorded by means of a scanning Hall probe.

In Fig. 2, we present the cutting scheme of the small pieces from the bulk pellet intended for AFM/STM measurements. In order to illustrate the changes of the nanostripe patterns depending on the position in the bulk sample, the sample was cut through the center, parallel to one edge of the seed crystal. Therefore, we obtain small samples for AFM measurements located in the *a*-growth sector as well as in the *c*-growth sector of the original bulk sample [15]. Furthermore, one sample was taken close to the edge of the original bulk sample where the growth front has ended. A total of 12 samples were cut from the bulk pellet; the exact positions

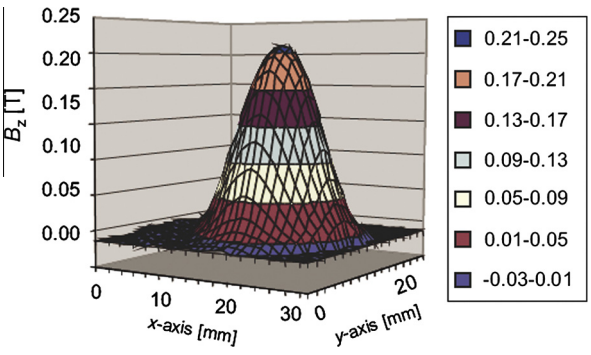

Fig. 1. Trapped field measurement of the bulk pellet before cutting. Note the homogeneous field distribution which indicates a homogeneous distribution of the flux pinning sites within the bulk pellet. A maximum trapped field of 0.22 T is recorded.

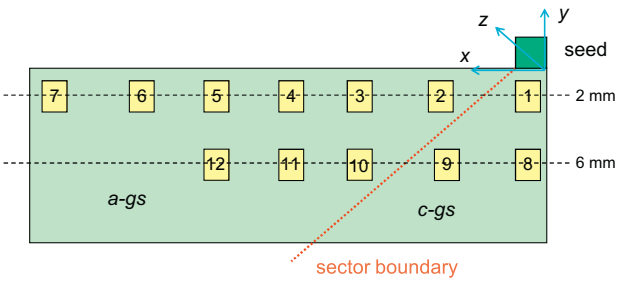

Fig. 2. Schematic drawing of the locations where the small samples were cut from the big pellet. The positions 1, 8 and 9 are located close to the seed crystal within the *c*-growth sector. Position 7 ($x = 17$ mm) is located outside the oriented growth sector. From all samples, we always investigated the top surface.

Table 1
Summary of the observations of nanostructures as function of position within the original bulk sample. The exact sample positions [*x*,*y*] are given in millimeters (see also the drawing in Fig. 2). Position 7 is located outside of the regular growth sector.

Position	Periodicity (nm)	Observation	Position	
			x (mm)	*y* (mm)
1	15	Homogeneous stripes, small periodicity (*c*-gs)	1	2
2	50	Homogeneous stripes	4	2
3	45	Homogeneous stripes	6	2
4	48	Homogeneous stripes	8	2
5	47	Homogeneous stripes, curvy	10	2
6	~45	Stripes show irregular edges, curvy	12	2
7	–	No stripes, only clusters, aligned	17	2
8	15	Homogeneous stripes, small periodicity (*c*-gs)	1	6
9	13	Homogeneous stripes, small periodicity (*c*-gs)	3	6
10	50	Homogeneous stripes	6	6
11	50	Homogeneous stripes	8	6
12	~45	Stripes are curvy, irregular edges	10	6

and the measured properties of all of them are summarized in Table 1.

The AFM and STM topography scans taken in ambient conditions are presented in Figs. 3–6 below.

Fig. 3 shows typical images (AFM, tapping mode) from positions 1, 8, and 9, which are all located within the *c*-growth sector. The investigated surface corresponds to a (*a*,*c*)-plane; the *c*-axis location is indicated in the image. The nanostripe patterns observed in these positions have only a short periodicity of 10–20 nm.

Fig. 4 gives examples of the nanostripe patterns observed at positions 2–5, 10, and 11 stemming from the *a*-growth sector. The selected surface of the samples corresponds to a (*a*,*b*)-plane with the *c*-axis perpendicular to the image plane. The topography images are recorded by tapping mode AFM and also STM for comparison. All nanostripe patterns of this region are found to be very regular. Here, it is important to note that the sample in this area exhibits a well developed texture of the superconducting matrix, and hence, also well developed nanostripe patterns are observed here. The degree of texture of the respective samples was checked by an EBSD analysis, which revealed a dominating (0 0 1)-texture with misorientations not exceeding 5° which is typical for good melt-textured 123-type samples [16,17].

Fig. 5 illustrates the topography (tapping-mode AFM) observed at position 6, which is also a (*a*,*b*)-plane. The nanostripe patterns get diffused and the stripe edges are irregular. These images reveal the nature of the nanostripes being composed of chains of islands.