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MICROSTRUCTURES AND dc CRITICAL CURRENTS IN
TEXTURED Y-Ba-Cu OXIDES

J. Orchotsky, H. Wiesmann, A. R. Moodenbaugh and M. Suenaga
Division of Materials Science
Brookhaven National Laboratory
Upton, NY 11973

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and
H.-G. Wang and H. Herman
State University of New York at Stony Brook
Stony Brook, NY 11794

Abstract

Sintered bars of Y-Ba-Cu oxide were partially melted using a quartz halogen lamp zone heating apparatus. The resulting multiphase samples showed varying degrees of "melt texturing." It was found that the addition of $\text{YBa}_2\text{Cu}_3\text{O}_7$ powders to the sintered $\text{YBa}_2\text{Cu}_3\text{O}_7$ prior to the zone melting process enhanced the growth of large textured grains. The best value of the critical current density was $\sim 10^3 \text{ A/cm}^2$ at 1.0 T and 77 K for the starting composition of $\text{YBa}_2\text{Cu}_3\text{O}_7 \cdot (0.2) \text{ Y}_2\text{BaCuO}_5$.

Introduction

The low current carrying capacity of bulk high T_c superconductors is still a major disappointment for those interested in the application of these materials to practical devices. It is widely accepted that this property of the oxides is due to the presence of very weakly coupled superconducting regions at grain boundaries. The intragranular current density J_c has been shown to be sufficiently large even at 77 K. Thus, in order to circumvent the grain boundary problem, Jin et al.¹ demonstrated that bulk $\text{YBa}_2\text{Cu}_3\text{O}_7$ (123) with improved values of critical current density J_c can be produced by a "melt texturing" technique. Furthermore, Murakami et al.² showed that the growth of large textured grains of 123 can be fabricated by quenching molten 123, reheating to a partial-melt condition and slowly cooling. These samples also sustained large screening currents. A number of other groups have also reported increased values of J_c in 123 produced by texturing in various ways.³⁻¹⁰

We constructed a quartz halogen lamp zone-melting apparatus to investigate the possibility of fabricating textured 123 in the form of a composite tape by a continuous process. Earlier reports,^{5,6,9} suggested that the starting composition of the sintered bar has a strong influence on the microstructure and J_c of the resulting textured specimen. We studied the influence of the nominal starting compositions and effects due to the rate of motion and temperature of the partially molten zone on the morphology and dc transport critical currents of bulk textured 123 solid bars.

Experimental Procedures

For preparation of specimens for texturing, a nominally stoichiometric commercial powder of 123 was pressed to a disk 50 mm in diameter and ~ 3 mm in thickness, and then heated to 950°C for several hours in flowing oxygen in order for it to be sufficiently sintered for slicing. Those specimens containing Y_2BaCuO_5 (211) powders were similarly produced by mixing the 123 powders with separately prepared 211 powders before pressing into a disk. Then, the sintered disks were sliced into rectangular bars ($\sim 1 \text{ mm} \times \sim 3 \text{ mm} \times \sim 50 \text{ mm}$) for texturing.

The zone heating apparatus was constructed using a pair of linearly focused quartz halogen lamps which were positioned to face each other at a common horizontal focal line. A quartz tube with

flowing oxygen was placed vertically between the lamps and a specimen was suspended from the top in the tube. For this study, the lamps were moved upward with the speed of 3.2 or 6.5 mm/h. The exact temperature of the partially molten zone of the specimen is difficult to determine. The estimated temperature range as measured by an optical pyrometer is ~ 1030 - 1100°C .

After completion of the zone melting, the specimens were heated in a flowing oxygen atmosphere for ~ 1 h at 950°C , then slowly cooled to 500°C and held for 24 h at this temperature before final cooling. At this point, the specimens were cut in half lengthwise. Since the specimens did not always remain straight, the length of the specimens cut for the critical current measurement varied from ~ 10 mm to ~ 30 mm long. Strips of Ag were deposited by sputtering onto the bar. The specimens were again heated to $\sim 500^\circ\text{C}$ for ~ 1 h in oxygen to reduce the contact resistance.¹¹ After this process, the resistance of the current contacts was $\sim 10^{-5}$ to $\sim 10^{-6} \Omega$. Current and voltage leads were then soldered to the Ag using Pb-Sn solder.

Critical currents were measured in a copper-wound split-pair magnet which produced a maximum field of 1.2 Tesla at 100 W power dissipation in a liquid nitrogen bath. All measurements of J_c were performed in a geometry where the applied current was perpendicular to the field. The criterion for determining J_c is $2 \mu\text{V/cm}$ over the length (5-10 mm) of the specimen.

ResultsMicrostructures

Development of texture microstructures depends crucially on a number of factors including (1) temperature, (2) rate of lamp motion, (3) composition, (4) temperature gradient, (5) ambient atmosphere, and (6) thickness of the specimen. Here, we discuss the effect of the first three variables. In regard to the composition of the starting material, we only discuss the effect of the 211 additions. The temperature gradient was determined by the properties of the lamp and no attempt was made to change it.

In Figs. 1(a) and 1(b) an example of microstructure in a textured 123 sample is shown. This was made from nominally pure 123 powder at a growth rate of 3.2 mm/h and at 1050 - 1100°C . The heated surfaces are the top and the bottom of Fig. 1(a), and the height in Fig. 1(a) represents the thickness of the specimen. Since these pictures were taken under polarized light, the regions with different contrast indicate different relative orientations. Thus, under this growth condition, large grains are grown, but along the length, the orientation of these grains varies. Figure 1(b) is a magnified view of a region in Fig. 1(a) and shows the presence of 211 precipitates as well as nearly parallel low angle grain boundaries within a grain. Although not clearly shown in Fig. 1(b), in those specimens made from a nominally stoichiometric powder, there are large voids which are an indication of the presence of a Ba-Cu-O compound that was etched away during the polishing operation. These voids tend to be at the boundaries of the large oriented regions.

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Figure 1. Optical micrographs taken using polarized light showing the cross section of a melt textured (123) grown at a speed of 3.2 mm/h and at 1050-1100°C for (a) an entire thickness and (b) for the enlarged view of (a).

Figure 3. As described in Fig. 1 for the specimen grown at a speed of 6.5 mm/h at 1050°-1100°C.

Figure 2. As described in Fig. 1 for the specimen grown at 3.2 mm/h and at slightly lower temperature.

Figure 4. As described in Fig. 1 for the specimen (123)-20% (211) grown at the speed of 3.2 mm/h and at 1050-1100°C. (a) is the side, (b) is the top view of the specimen and (c) is an enlarged side view.

Another sample was prepared using a reduced lamp heating current which we estimate would result in a process temperature reduced $\sim 20^\circ\text{C}$ from that above. The lamp speed was 3.2 mm/hr. The main difference in the specimen produced is a reduced grain size [see Figs. 2(a) and 2(b)]. The morphology within the grain is unchanged, however.

A third sample was prepared using a higher lamp speed, ~ 6.5 mm/hr, and the lamp power used to produce the sample in Fig. 1. The effect on the morphology is drastic; see Figs. 3(a) and 3(b). Although a substantial fraction of the specimen is oriented in a similar crystallographic direction [the bright region covers a large area in Fig 3(a)], the individual oriented regions are small and separated from each other by grains oriented in another direction. In addition, the aligned platelets within the oriented regions are shorter in this specimen [Fig. 3(b)] than in those specimens grown at lower speeds.

Earlier investigators concluded that the starting compositions of the specimens are important for various texturing processes.^{5,6,9,10} Here, we show the effect of the addition of the 211 powder to the sintered bars of 123. As shown in Figs. 4(a), 4(b), and 4(c), a specimen with a nominal starting composition of (123) + 0.2 moles (211), which was grown at a rate of 3.2 mm/h and at $1050\text{--}1100^\circ\text{C}$, has larger grains than those without 211 additions (Fig. 1). In fact, in this particular segment of the specimen, the entire cross section of the material is aligned to the same direction, although the micrograph of a section cut perpendicular to Fig. 4(a) of an adjacent section shows large regions oriented in different directions. The other differences between the nominally pure and the 211-added specimen are the size and the amount of the 211 precipitates as well as the width of the parallel platelets in the large grains. The reason for this difference is not known at this time.

Critical Current Densities

The measured values of J_c in these specimens are not always consistent. This is partly due to existence of microcracks and nonuniform microstructures. With a sufficient number of measurements, a trend in J_c can be identified, which is related to the morphology. The field dependence of J_c for several starting compositions (123 + various amounts of 211) is shown in Fig. 5. For the sample with no added 211, the J_c is poor, even though the size of oriented regions is substantial (see Fig. 1).

For the case of no 211 addition, the results for the sample grown at 3.2 mm/hr can be compared with that grown more quickly (6.5 mm/hr). The sample grown quickly shows relatively poor grain orientation (compare Fig. 3 and Fig. 1). In spite of this, the magnitude and field dependence of J_c are comparable ($J_c \approx 430$ A/cm² at $H=0$ for the 6.5 mm/hr sample). This suggests that perhaps, in samples without 211 addition, the boundaries between the large oriented regions are contaminated in such a way as to reduce J_c .

As illustrated in Fig. 5 for samples grown at 3.2 mm/h at 1050°C , the values of J_c were significantly improved with increasing amounts of 211 in the starting material, particularly at higher fields. This suggests that large aligned regions in the specimens are helpful in increasing J_c . However, it is interesting to note that a specimen with 20% 211, which was grown at the speed of 6.5 mm/h at $\sim 1050^\circ\text{C}$ rather than at 3.2 mm/h, also carries a substantial current density, approximately 1/2 of that in Fig. 4 although the microstructure is similar to that in Fig. 3 rather than in Fig. 4. These results suggest that the increased J_c accompanying the addition of 211 is not only due to the enhanced growth of the oriented region, but also to other factors, such as decreased areas of uncontaminated grain boundaries. In this connection, it is interesting to note that the additions of 211 nearly eliminate large BaCuO₂ and CuO precipitates at the boundaries of the oriented regions, as originally suggested by Kase et al.⁵ In order to confirm the suggestion that a larger fraction of the grain boundaries in the materials with 211 additions is cleaner than those without, transmission electron microscopy of the grain boundaries is planned.

Figure 5. The critical current densities as a function of magnetic fields at 77 K for (123), (123) + 5 mol.% (211) and (123) + 20 mol.% (211). All grown at 3.2 mm/h and at $1050\text{--}1100^\circ\text{C}$.

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