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CRITICAL CURRENTS, MAGNETIC RELAXATION, AND MICROSTRUCTURE IN

ZONE-MELTED $\text{YBa}_2\text{Cu}_3\text{O}_x$ *

CONF-900944--36

DE91 006594

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To appear in Proceedings of 1990 Applied Superconductivity Conference, September 24-28, 1990, Snowmass, CO, to be published in IEEE Transactions on Magnetics, March 1991.

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*Work supported by the U.S. Department of Energy, BES-Materials Sciences under contract #W-31-109-ENG-38 (DS, JGC, MX, KCG), and the National Science Foundation under Research Grant MSM-871843, and the Indiana Center for Innovative Superconductor Technology through funding from the Indiana Corporation for Science and Technology (PJM, WC).

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CRITICAL CURRENTS, MAGNETIC RELAXATION, AND MICROSTRUCTURE IN ZONE-MELTED $\text{YBa}_2\text{Cu}_3\text{O}_x$ *

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Abstract

Extruded $\text{YBa}_2\text{Cu}_3\text{O}_x$ wires have been zone-melted to develop highly textured microstructures. The zone-melted wires exhibit large magnetic hysteresis and transport J_c values relative to conventional bulk $\text{YBa}_2\text{Cu}_3\text{O}_x$. Transmission electron microscopy observations indicate unique grain boundary features which are likely to be responsible for reduced weak-link effects. Large amounts of intragranular defects such as dislocations and Y_2BaCuO_5 particles have been observed in the sample matrices. The dislocations are possibly connected with the greatly increased magnetic hysteresis.

Introduction

Transport critical current density has been a major issue in high- T_c superconductors. Practical application of high- T_c superconductivity not only requires that the materials possess high current-carrying capability, but also mechanical flexibility and chemical stability. However, due to some natural characteristics of high- T_c ceramics, such as high anisotropy, severe flux creep, and weak-link effects associated with grain boundaries, the materials in the bulk polycrystalline form generally carry only extremely low critical currents, especially in magnetic fields. Various methods, such as orienting of grains and introducing extra pinning centers to the system, have therefore been devised to solve these problems (1-4). In this paper, we report experimental results of superconducting properties and microstructural characteristics in zone-melted $\text{YBa}_2\text{Cu}_3\text{O}_x$ wires. We discuss the possible microstructural effects on transport critical current density, magnetic hysteresis, and flux creep.

Experimental Details

Wires of $\text{YBa}_2\text{Cu}_3\text{O}_x$ were fabricated by a plastic extrusion technique (5). Well-characterized $\text{YBa}_2\text{Cu}_3\text{O}_x$ powder was prepared via a solid-state reaction from appropriate amounts of reagent grade Y_2O_3 , BaCO_3 , and CuO (6). The precursors were mixed by ball milling with ZrO_2 media, calcined for 4 h at 800°C in flowing oxygen at a total pressure of about 2.6×10^2 MPa, followed by low-temperature oxygen annealing in ambient pressure oxygen. The calcined $\text{YBa}_2\text{Cu}_3\text{O}_x$ powder was mixed in a solvent with binder, dispersant, and plasticizer and was homogenized in a mechanical mixer. The resultant plastic mass was forced through a small opening in a steel die to form a wire. The extruded wire was cut into pieces, which were heated very slowly in oxygen to 400°C to remove the organics and then sintered at 950°C in oxygen. Final wire diameters were about 2 mm in diameter.

The sintered wire samples were zone melted in a vertical tube furnace, with a resistance coil providing localized heating of the samples. Typical hot zone temperatures were between 950 and 1050°C . The solidification rate was controlled by varying the sample speed through the hot zone (7). The zone-melting process was carried out in air; a subsequent oxygen anneal at about 450°C was required to increase the oxygen level in the $\text{YBa}_2\text{Cu}_3\text{O}_x$ sample.

The critical current density, J_c , determined with a voltage criterion of 1 mV/cm, was measured by a standard four-terminal method at 77 K. All samples were cut and carefully shaped into pieces with dimensions of $13 \times 1.5 \times 0.3$ mm. The current and voltage leads were directly soldered onto the sample surface with pure indium. An electromagnet was used to supply a steady applied field up to 1.5 T for the J_c measurements. The magnetic hysteresis and relaxation data were taken with a commercial

superconducting quantum interference device (SQUID) at various temperatures up to 5 T. The transmission electron microscopy (TEM) was performed on a Philips 420 electron microscope.

Results and Discussion

Transport J_c data for a zone-melted wire and a sintered pellet are shown in Fig. 1 (the pellets were made from $\text{YBa}_2\text{Cu}_3\text{O}_x$ powder processed identically to that used for the wires (6)). As shown in the figure, the transport J_c of the sintered pellet drops sharply to a value of 20 A/cm^2 at an applied field of only a few hundred gauss. This drop is due to the well-known weak-link behavior associated with misoriented grain boundaries (8). However, J_c in the zone-melted sample remains at considerably higher values near 10^3 A/cm^2 , in applied fields up to 1.5 T. Since severe heating due to high contact resistance at the current leads was encountered during the transport J_c measurements, the values shown in Fig. 1 are the lower limits of J_c ; the values for the zone-melted wire could actually be much higher than those reported. It should be noted that the as-sintered and the zone-melted samples were from different batches of material and transport J_c behavior of the as-sintered sample was typical of all other polycrystalline samples.

Figure 1. Transport critical current density versus applied magnetic field at 77 K for zone-melted and as-sintered samples.

Magnetic hysteresis results for both as-sintered and zone-melted samples are shown in Fig. 2. As can be seen, the hysteresis loop of the as-sintered sample almost vanishes at 77 K, indicating largely reduced flux pinning strength. However, the hysteresis loop of the zone-melted sample remains open up to 1 T. This greatly increased magnetic hysteresis indicates that the microstructure of the as-sintered and zone-melted samples must be essentially different.

Figure 3 shows the magnetic relaxation rate ($dM/d\ln t$) versus applied field (H) data for the zone-melted wire from 10 K to 50 K. As shown in the figure, between 20 K and 50 K, the relaxation rate experiences only small changes with increasing field up to 2 T.

Near 10 K, however, dM/dH is a gradually increasing function of applied field. As can also be seen in Fig. 3, at each fixed field dM/dH increases; at 2 T for example, dM/dH increases from 0.2 emu/cm^3 to 2.7 emu/cm^3 as temperature decreases from 50 K to 10 K.

Figure 2. Magnetization hysteresis at 77 K for zone-melted and as-sintered samples.

Figure 3. Magnetic relaxation rate (dM/dH) versus applied field at temperatures indicated for the zone-melted wire.

According to the flux-creep model, the magnetization $M(t)$ can be written as (9):

$$M(t) = M(0) \{1 - kT/U [\ln(t/t_0)]\},$$

where $M(0)$ is the magnetization at the initial time, U is associated with the condensation energy, and $1/t_0$ as the hopping frequency for the flux lines to jump over the pinning potential. It should be noted that U is not the condensation energy as defined in the classical flux-creep model. We have also measured the magnetic relaxation for the as-sintered sample in the same temperature and field region (the data are not shown here). We have estimated U

value to be 30 meV and 16.8 meV at 10 K for the zone-melted and as sintered samples, respectively.

Figure 4 shows a scanning electron micrograph (SEM) of the zone-melted wire. It can be seen in the figure that the $\text{YBa}_2\text{Cu}_3\text{O}_x$ grains are large and highly elongated and are aligned into a well-textured microstructure. Cracks are present between many of the grains. A considerable amount of Y_2BaCuO_x , formed during the incongruent melting process, is also present in the sample matrix. The Y_2BaCuO_x is present as large, randomly oriented grains.

To find out the detailed intergranular and intragranular microstructural characteristics of the zone-melted sample, extensive TEM was performed. We have observed small-angle grain boundaries in the zone-melted sample (10). It is observed that twin boundaries run smoothly through the grain boundaries and that there is no second phase found in the grain boundary region. The small-angle grain boundaries are typical features in the zone-melted material, which is consistent with the SEM observations. It has been reported that weak-link effects in $\text{YBa}_2\text{Cu}_3\text{O}_x$ are attributed to strong superconducting anisotropy and second phase contamination at grain boundaries (8). The anisotropy problem can be solved by reducing crystal misalignment, and occurrence of small-angle grain boundaries is probably connected with improved transport J_c of the zone-melted wire.

A large concentration of dislocations was observed in the zone-melted sample (Fig. 5). The dislocation density has been estimated to be $10^{10}/\text{cm}^2$. Far fewer dislocations have been observed in conventionally sintered $\text{YBa}_2\text{Cu}_3\text{O}_x$ (11). With the two-beam conditions, bright field image, the Burger's vectors of the dislocations in both types of sample have been determined to be $[100]$ and $[010]$. We have also found that the dislocations are located mostly in the a - b plane, which may be due to the fact that it is relatively easy to slip along a or b directions. The dimension of the dislocation is on the scale of the lattice, which is comparable with the coherence length of the superconductor. Therefore, these dislocations may act as strong pinning centers. It should be noted that the dislocations were rarely observed in the as-sintered samples.

Figure 4. SEM micrograph showing the textured microstructure in the zone-melted sample; the large particles in the matrix are the Y_2BaCuO_x phase.

Figure 5. TEM photograph showing a high density of dislocations in the sample matrix for the zone-melted sample.

It was found that microstructural features such as twin density, oxygen content, and crystal structure were quite similar for the as-sintered and zone-melted samples. However, the samples exhibit a large difference in the magnetic hysteresis loop and magnetic relaxation behavior. The increased hysteresis loop in the zone-melted wire could, on one hand, be due to the enlarged induced current loop size, since the contacts between the superconducting grains are enhanced, but could, on the other hand, be attributed to strong flux pinning associated with high density of crystal defects such as dislocations. The increased U value (i. e. reduced flux creep effects) may also be related to high microstructural inhomogeneity in the zone-melted wire. Further systematic study of flux pinning in the zone-melted wires is underway.

Conclusions

A well-textured microstructure was obtained in zone-melted $\text{YBa}_2\text{Cu}_3\text{O}_x$ wires. The transport J_c in the zone-melted wire, in magnetic fields up to 1.5 T, has been greatly improved at 77 K. TEM evidence suggests that this enhancement of J_c may be connected with small-angle grain boundaries. The magnetic hysteresis loop at 77 K in the zone-melted wire is largely increased relative to that for a sintered specimen. This increase of the hysteresis loop may possibly be attributed to a high density of dislocations acting as strong pinning centers.

Acknowledgments

We thank U. Welp and M. M. Fang for the magnetization measurements and M. T. Lanagan and U. Balachandran for providing the extruded wire samples. This work was supported by the U. S. Department of Energy, Basic Energy Sciences-Materials Science, under Contract W-31-109-ENG-38 (DS, JGC, MX, KCG); the National Science Foundation under Research Grant MSM-871843, and the Indiana Center for Innovative Superconductor Technology through funding from the Indiana Corporation for Science and Technology (PJM, WC).

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