Dependence of Critical Current Density on Microstructure and Processing of High- T_c Superconductors

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ABSTRACT

Microstructural origins for reduced weak-link behavior in high- J_c melt-processed YBCO, spray pyrolyzed thick films of Tl-1223, metallic precursor Y-124 polycrystalline powder-in-tube (PIT) wires and PIT Bi-2212/2223 are discussed. Since the materials studied are the highest J_c , polycrystalline, high- T_c superconductors fabricated worldwide, the results provide important guidelines for further improvements in superconducting properties, thereby enabling practical applications of these materials. It is found that strongly linked current flow within domains of melt-processed 123 occurs effectively through a single crystal path. In c-axis oriented, polycrystalline Tl-1223 thick films, local in-plane texture has been found to play a crucial role in the reduced weak-link behavior. Formation of "colonies" of grains with a common c-axis and modest in-plane misorientation was observed. Furthermore, a colony boundary in general has a varying misorientation along the boundary. Large regions comprised primarily of low angle boundaries were observed. Percolative transport through a network of such small angle boundaries appears to provide the non-weak-linked current path. Although powder-in-tube BSCCO 2212 and 2223 also appear to have a "colony" microstructure, there are some important differences. Colonies in BSCCO consist of stacks of grains with similar c-axis orientation in contrast to colonies in Tl-1223 films where few grains are stacked on top of one another. Furthermore, most grains within a colony in BSCCO have the same lateral dimensions as that of the colony, resulting largely in "twist" boundaries. Further microstructural characterization of high- J_c PIT 2212 and 2223 is currently underway. In the case of Y-124 wires, weak macroscopic in-plane texture is found. Additional measurements are underway to determine if a sharper, local in-plane texture also exists. It is found that in three of the four types of superconductors studied, reduced weak-link behavior can be ascribed to some degree of biaxial alignment between grains, either on a "local" or a "global" scale.

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1.0 INTRODUCTION

It is now well established that the transport critical current density (J_c) of bulk, sintered, unoriented, polycrystalline, high T_c superconductors is extremely low (~ 0.1 – 1 kA/cm^2) even in the absence of magnetic fields, and drops significantly in the presence of a few hundred Gauss. The established microstructural features dictating the transport J_c include chemical and structural variations at grain boundaries, micro-cracking, and the inherent anisotropy of the superconducting properties. Unlike conventional low-critical-temperature (T_c) superconductors where the grain boundaries act as pinning centers, grain boundaries in high- T_c superconductors are weak-links due to the extremely short coherence lengths in these materials. The effect of intrinsic anisotropy of the superconducting properties and the presence of structural disorder at grain boundaries on J_c is well documented by the results of Dimos et al. on artificially fabricated $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (123) grain boundaries on bi-crystals of SrTiO_3 [1]. Intergranular critical current densities were found to decrease rapidly with increasing misorientation angle, even for a symmetric [100] tilt boundary. For such a boundary, the c-axes of the two grains are parallel and current flow is predominantly in the basal plane. Such a boundary is most favorable with respect to the intrinsic anisotropy in the superconducting properties, particularly the coherence length, which is much larger in the basal plane than along the c-direction for all the layered ceramic superconductors. Measurements on Tl- and Bi- bicrystals indicate a similar behavior [2-4]. These results indicate that a high degree of biaxial alignment may be a prerequisite for high transport J_c . Exceptions to this are special low energy boundaries as discussed by Babcock et al. [5].

Here we report on microstructural characterization of four types of superconductors: domains of melt-processed 123 [6-11], $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ (Tl-1223) spray-pyrolyzed thick films [12], metallic precursor $\text{Ca}_{0.1}\text{Y}_{0.9}\text{Ba}_2\text{Cu}_4\text{O}_8$ (Y-124) powder-in-tube (PIT) multi-filamentary wires [13] and PIT $\text{Bi}_2\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_x$ (Bi-2212) and $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ (Bi-2223) polycrystalline wires [14]. These materials are the *only* forms of polycrystalline, high- J_c superconductors fabricated so far world-wide. It is found that three of these superconductors contain some degree of biaxial alignment between grains, either on a macroscopic scale (tens of mm) or a microscopic ($< 1 \text{ mm}$) scale. The results are suggestive of model microstructures for polycrystalline superconductors which may lead to strongly-linked current paths without complete biaxial alignment of grains.

2.0 EXPERIMENTAL

Domains of melt-processed 123 were fabricated using a schedule reported previously [6,7]. In addition, materials from other investigators including, Salama et al. [9], McGinn et al. [10], Armstrong et al. [11] and Neal et al. [15] were examined. All the materials examined had high- J_c 's, typically 10^4 A/cm^2 in 1T (H//c) at 77K.

Spray-pyrolyzed Tl-1223 thick films were obtained from Deluca et al. and were fabricated using procedures reported previously [12]. Transport J_c of the samples examined are 10^5 A/cm^2 and 10^4 A/cm^2 in self-field and 1T (H//c) respectively, at 77K[13].

Metallic precursor, Y-124 PIT wires were obtained from Masur et al. [13]. J_c values of the tapes examined are $\sim 7 \times 10^4 \text{ A/cm}^2$ and $3 \times 10^4 \text{ A/cm}^2$ in self-field and 0.1T respectively at 4.2K [13].

PIT Bi-2212 and Bi-2223 wires were obtained from American Superconductor Corporation [14]. The J_c values of the Bi-2223 tapes examined at were $\sim 2 \times 10^4 \text{ A/cm}^2$ in self-field [14] at 77 K.

Microstructural examinations were made using a combination of optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), Electron backscatter diffraction (EBSP) and X-ray diffraction (XRD) techniques.

3.0 RESULTS AND DISCUSSION

3.1 Melt-Processed Domains of 123

Convergent beam electron diffraction has shown that platelets within domains of melt-processed 123 are simply portions of a common single crystal [6-8]. No orientation change was detected between adjacent platelets comprising a domain [6-8]. These platelets are however separated and the intervening region is filled, primarily with crystalline Cu and Ba-Cu- oxides. However, it is found that these planar platelet boundaries are not continuous and terminate within the domain. Hence, a non-weak-link current path can readily be envisioned through continuous, interconnected, single

crystalline regions. Nucleation and growth mechanisms resulting in formation of such a unique microstructure have been proposed [6,7]. In summary, microstructural analysis of melt-processed 123 domains indicates that mitigation of weak-links for current flow in the c-direction is a result of near *complete biaxial texture* and the discontinuous nature of the platelet boundaries.

3.2 Tl-1223 Spray Pyrolyzed Thick Films ($\sim 3\mu\text{m}$)

As formed films were found to exhibit a high degree of c-axis texture. Conventional x-ray rocking curves of the (004) Bragg reflection show that the macroscopic c-axis alignment of the films is 2 deg FWHM. No macroscopic in-plane texture was found. TEM of cross-sectional samples, SEM on fracture samples and EBSD measurements on polished cross-sections and tapered plan-view samples indicate that the films are comprised of plate-like grains $\sim 0.5\text{-}2\mu\text{m}$ thick in the c-direction, with basal plane dimensions in the range of $3\text{-}10\mu\text{m}$. Typical aspect ratios were found to be ~ 10 , intermediate between those seen in 123 and Bi-2212, 2223. In addition, TEM indicated that most of the grain boundaries were clean in the sense that no second phases were present.

Local texture was determined from XRD azimuthal scans (i.e., ϕ scans). In these experiments, the intensity of a non-00l reflection is monitored as the sample rotates about its surface normal (i.e. the c-axis). Thus, ϕ scans are effectively distribution plots of the a-axis of the grains. Figure 1a&b show ϕ scans using effective X-ray beam sizes of $4 \times 8 \text{ mm}^2$ and $0.5 \times 1 \text{ mm}^2$, generated using a conventional source and Figure 1c shows a ϕ scan using a circular beam of 0.13 mm^2 , generated using a much brighter synchrotron X-ray beam at the beam-line X-14 of the National Synchrotron Light Source (NSLS) [16]. The intensity distribution obtained using the largest beam used (Figure 1a) consists of several peaks superimposed on an average intensity which is well above the background, suggesting that basal plane orientations are largely random, but there is a tendency for clustering about several specific orientations. A very different intensity distribution is obtained when a smaller beam is used as indicated in Figure 1b. This scan shows that in the region illuminated by the beam, the a-axis clusters around two orientations. Such peaks in the ϕ scan indicate the presence of locally aligned regions or "colonies" of bi-axially aligned grains. Figure 1c shows a ϕ scan obtained using a high intensity 0.13mm^2 circular beam generated at NSLS. The intensity distribution indicates the presence of only one colony. The high intensity of the X-ray beam results in a high signal/noise ratio

compared to that obtained using a conventional X-ray source (Figure 1b). Experiments were performed on several films, wherein, the X-ray ϕ scans were taken as a function of position on the film. This results in a complete mapping of the a-axis distributions of the regions measured. Such X-ray contour maps were compared to the measured transport J_c 's of various segments in the films [17,18]. The results show excellent correlation between regions of high- J_c and high in-plane texture. In addition, it was found that within colonies the c-axis texture was also sharper, typically 1.2 degrees FWHM.

The presence of in-plane texture was further confirmed using EBSD measurements, using a technique described elsewhere [19]. Diffraction patterns were obtained from a large number of points within a rectangular area on the sample surface. In some cases the samples were lightly polished to remove silver and secondary phase particles. While observing the diffraction pattern the electron beam was moved in a line over the sample surface. When a shift in the pattern was observed the new pattern was recorded and analyzed to obtain the local crystalline orientation. Figure 2a shows a (100) pole figure of the patterns recorded in one area. The presence of only one maximum in the (100) pole figure suggests that the grain orientations in the region examined cluster around one orientation. An X-ray ϕ -scan from such a region would correspond to that shown in Figure 1c. Such clustering of grain orientations around a single orientation is indicative of a "colony" of locally aligned grains. Fig. 2b shows an "arrow map" of the a-axis directions for a $44 \times 120 \mu\text{m}^2$ area. Since the c-axes of the grains are very well aligned (FWHM ~ 1.2 deg), the in-plane orientation can be graphically represented by a single "arrow" in the a-direction of the grains. During the measurement, the electron beam was moved along parallel lines separated by $12 \mu\text{m}$ to obtain data over a rectangular grid. An arrow is plotted only where a shift in the diffraction pattern was seen, i.e., at a grain boundary. The spacing between arrows is roughly indicative of the grain size. Clearly the area mapped in Figure 2b contains only one colony as grain orientations cluster around a single orientation. No large angle grain boundaries were detected. The structure of colony intersections is shown in Figures 3 and 4. One kind of colony intersection which results in largely high angle boundaries is indicated in Figure 3. Figure 3b shows the arrow map of the region examined and Figure 3a shows the corresponding (100) pole figure. The presence of two colonies can clearly be seen from the (100) pole figure. The total spread in a-axis orientations within a colony is 15-20 degrees (as determined from the (100) pole figure). A line has been hand-drawn to indicate the approximate position of the colony boundary in Figure 3b. The average a-axis orientations of the two colonies differ by ~ 35 -40 degrees (as revealed by the (100)

pole figure). For a c-axis aligned tetragonal material, the maximum a-axis misorientation is 45 degrees, so the misorientation between these colonies is a maximum. The arrow map in Figure 3b shows there appear to be few small angle grain boundaries in the portion of the colony intersection sampled. Figure 4 shows another type of colony intersection. The (100) pole figure in Figure 4a shows the presence of two colonies. Figure 4b shows the corresponding arrow map. Although the average a-axis misorientation between the two colonies is again large (~ 30 deg, as indicated by the (100) pole figure), the structure of the colony intersection is quite different from that in Figure 3. Figure 4b shows that the change in orientation of the grains across the colony intersection is gradual. Hence, it is difficult to locate the position of the colony intersection. This is an example of a colony intersection where a series of small angle boundaries accommodates the bulk misorientation between colonies which is ~ 30 degrees.

These results are suggestive of long-range current transfer through a *percolative network of small angle boundaries* at colony intersections. The number of such small angle boundaries should increase with the spread in orientation of grains within a colony (i.e. the FWHM of a colony) and with local bias of colony orientations, as may result from certain processing conditions, e.g., an imposed thermal gradient.

3.3 METALLIC PRECURSOR Y-124 WIRES CONTAINING 962,407 FILAMENTS

Figure 5a shows a high resolution SEM image of an Y-124 sample from which the silver was removed by chemical etching. No damage to the superconductor from the etching procedure was detected. Individual filaments are found to be $\sim 0.5\mu\text{m}$ thick and 1- $1.5\mu\text{m}$ wide. Excellent uniformity of filament shape is observed over long lengths. The filaments are comprised of grains $< 1\mu\text{m}$ in size. In many areas the thin filaments are seen to accommodate a large curvature or bending (presumably resulting from the iterative deformation sequence of rolling and heat-treatment) without any evidence of microcracking, perhaps a result of the fine grain and filament size. It is also found that grains are in general rectangular. In many areas these rectangular grains have their long sides oriented parallel to the filament length (Figure 5b). However, there exist many areas where the sides of the grains make high angles to the axis of the filament.

X-ray pole-figures in Figure 6a indicate that the c-axes of the grains tends to tilt about and along the axis of current flow. The distribution is 40 degrees FWHM about the

direction of current flow, and 20 degrees FWHM in the direction of current flow. The grains appear to tilt more about the axis of the filaments in many regions (as seen in SEM), resulting in a c-axis FWHM of ~ 40 degrees in this direction. In order to determine the presence of macroscopic in-plane texture, (017) and (108) pole figures were obtained. These pole figures shown in Figures 6a&b, indicate the presence of a weak macroscopic in-plane texture. The (017) pole figure has the highest intensity along the direction of current flow and the (108) pole figure peaks 90 degrees to the direction of current flow. This indicates a weak b-axis macroscopic in-plane texture along the length of the filament, consistent with SEM morphological examinations, indicating that one side of the rectangular grains was parallel to the axis of the filaments in many cases (Figure 7).

At this point, it is however not clear what happens at the "local" scale. Previous TEM examinations on these wires have however indicated a high degree of in-plane texture in the areas examined [13]. Future EBSP experiments are planned to shed additional light on local correlations between grains over extended lengths. In summary, the results obtained so far clearly indicate that some degree of biaxial texture exists in the filaments. X-ray measurements indicate that there is a weak global biaxial texture. There appears to be a sharper local texture, however, additional measurements are needed to determine the length scale of this local texture.

3.4 Bi-2223 and 2212 PIT WIRES

The microstructures of these tapes appear to be the most complicated of the four classes of materials studied. Typically, Bi-2223 and 2212 samples also contain "colonies" of grains. Colonies in BSCCO are however quite different from those in Tl-1223 thick films. Colonies in BSCCO consist of stacks of grains with similar c-axis orientation resulting in largely twist boundaries. Whereas in Tl-123 deposits, which are only a few grains thick, colonies extend laterally over many grains and contain primarily tilt boundaries. In the case of BSCCO, it is not clear if the grains comprising a colony have any correlation in orientations. Detailed TEM investigations of 2212 colonies by Gao et al. [20] have shown that most boundaries are small angle (< 10 degrees) c-axis twist boundaries. Twin boundaries (resulting in 90 degree c-axis twist boundaries) are also present.

In order to obtain further information about grain correlations, X-ray diffraction studies on Ag-removed samples were undertaken. It is found that while the tapes are

highly c-axis aligned, the texture is far from perfect. (0 0 20) rocking curves show a FWHM ~ 18 -25 degrees. ϕ -scans taken with effective beam sizes of $0.5 \times 1 \text{ mm}^2$ and 0.13 mm^2 indicate random in-plane texture. In addition, complete pole figure measurements of the (1 1 19) reflection show a random texture on the size scale of the beam (typical basal plane grain dimensions are 20 - $40 \mu\text{m}$, so that 25 - 50 grains are sampled using a beam of 0.13 mm^2). It is possible that in-plane texture exists on a finer scale. Furthermore, since X-ray penetration depth is much larger than a typical grain thickness ($\sim 0.1 \mu\text{m}$) within a colony, diffraction from a number of platelets within a single c-axis aligned, colony is obtained. Since c-axis twist boundaries exist at grain boundaries, a random texture may be obtained even if there is some local correlation between the upper most grain of each colony abutting the Ag sheath. Valuable information may however be obtained by EBSP measurements which are currently underway.

4.0 SUMMARY

Microstructural examination of high- J_c , melt-processed Y-123, Tl-1223 spray-pyrolyzed thick films, metallic precursor, Ca-substituted Y-124 PIT wires, and PIT Bi-2212 and 2223 wires, was conducted. It is found that in three of the four classes of materials studied, reduced weak-link behavior can be ascribed to some degree of biaxial alignment between grains, either on a macroscopic scale (tens of mm) or a microscopic scale ($< 1 \text{ mm}$). The microstructure of the Tl-1223 films suggest that global biaxial texture is not required to obtain well-linked transport properties. "Local order" of grains, resulting in long-range percolative current paths through a network of small angle boundaries at colony intersections provides a working model for the microstructure of a practical, polycrystalline conductor. The microstructure of the BSCCO PIT materials is the most complicated of the materials studied and is yet to be fully understood.

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- Fig. 1. X-ray ϕ scans on a c-axis textured Tl-1223 thick film using a) $4 \times 8 \text{ mm}^2$, b) $0.5 \times 1 \text{ mm}^2$ and c) 0.1 mm^2 effective X-ray beam spot sizes. The first two spot sizes were generated using a conventional source while the smallest spot size was generated using a much brighter synchrotron X-ray beam at the beam-line X-14 of NSLS. Inset shows the schematic of the diffraction geometry.
- Fig 2. EBSP measurements on a Tl-1223 thick film. Figure 2a shows the (100) pole figure of the absolute orientations of all the grains measured. Since the c-axis is well aligned (FWHM ~ 1.2 deg), the orientation of the grains can be represented by a single arrow in the a-direction (Figure 2b). "R" and "T" represent rolling and transverse directions respectively and in this case correspond to the two sides of the substrate.
- Fig. 3. EBSP measurements showing an example of a region containing mainly high-angle grain boundaries at a colony intersection. Figure 3a shows the (100) pole figure and the corresponding arrow map is shown in Figure 3b.
- Fig. 4. EBSP measurements showing an example of a region containing mainly low angle grain boundaries at a colony intersection. Figure 4a shows the (100) pole figure and the corresponding arrow map is shown in Figure 4b. A gradual change in orientation across the intersection from one colony to another can be seen.
- Fig. 5. SEM micrograph showing the morphology of a single Y-124 filament.
- Fig. 6. a) (0010) pole figure from a Ag-removed Y-124 tape; b) (017) pole figure from the same region; c) (108) pole figure from the same region.
- Fig. 7. High magnification SEM micrograph of a region showing that the grain edges are parallel to the filament axis.

Pole figure (100)

(a)

(b)

Pole figure (100)

(a)

T

0 13.4 26.8 40.2 53.6 67 μm

(b)

Pole figure (100)

(a)

(b)

(a)

(0014)

(b)

(017)

(c)

(108)

current

Rolling
Direction

4392 2.0 kV X15.0K 2.00μm