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CRITICAL CURRENT DENSITY ENHANCEMENT IN  
ROLLED MULTIFILAMENT BI-2223 HTS COMPOSITES

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# CRITICAL CURRENT DENSITY ENHANCEMENT IN ROLLED MULTIFILAMENT BI-2223 HTS COMPOSITES

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## ABSTRACT

At 77 K and self-field, we report transport critical current density ( $J_c$ ) of 58 kA/cm<sup>2</sup> in rolled 19 and 85 filament Bi-2223 HTS tapes. For an 85-filament tape with transport  $J_c$  of 50 kA/cm<sup>2</sup>, transport  $J_c$  of greater than 80 kA/cm<sup>2</sup> has been measured in the individual filaments after etching off the Ag sheath. To achieve such high self-field electrical performance, increasing the number of the strong links between the 2223 colonies is believed to be the most important factor. Microstructure studies on these samples suggest that there is still great potential for further  $J_c$  enhancement.

## INTRODUCTION

During the past few years, ASC rolled multifilament Bi-2223 composites made by the powder-in-tube technology have advanced rapidly in performance[1-2]. Self-field  $J_c$  of 55 kA/cm<sup>2</sup> was measured at 77 K in a number of 85-filament composites[2]. Such a level of performance has begun to show promise for many commercial applications such as transmission cables, transformers, and large motors.

To further enhance self-field  $J_c$  for the Bi-2223 composites, the connectivity between grains must be improved by eliminating microcracks and by increasing the number of strong links. This is achieved by a high degree of c-axis texture, clean grain boundaries, and reducing secondary phase and porosity[2-6]. To our knowledge, most c-axis texture and microstructure studies so far were done on pressed monofilament samples with  $J_c$  values in range of 10 - 30 kA/cm<sup>2</sup>[5, 7-8].

In this paper, we present  $J_c$  progress with laboratory-scale, rolled multifilament Bi-2223 composites. Also, we show detailed microstructures and c-axis texture of these high performance tapes. Finally, we discuss the potential  $J_c$  enhancements related to c-axis current transport.

## EXPERIMENTAL

19 and 85 filament composites were fabricated using the powder-in-tube technique. The powder stoichiometry is Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>1.9</sub>Ca<sub>2.0</sub>Cu<sub>3.1</sub>. A thermomechanical process consisting of a sequence of roll deformation and heat treatment steps was used to promote 2223 phase formation, texture, and densification. Transport critical current (1  $\mu$ V/cm) measurements at 77 K and self-field were performed using a standard four probe technique. Transport  $J_c$  values were determined by dividing  $I_c$  by the total cross-sectional area of the filaments, determined by optical image analysis. Transport measurements were also performed on individual filaments of the multifilament samples by etching off the silver (details see Cai et al. in [9]). The c-texture of the 2223 phase was characterized by XRD rocking curves and resistivity measurements in directions parallel and perpendicular to the tape surface. Detailed microstructures of the high  $J_c$  tapes were studied using optical microscope and TEM.

## RESULTS AND DISCUSSION

Performance of ASC rolled multifilament Bi-2223 tapes has continued to advance with  $J_c$  of 58 kA/cm<sup>2</sup> (77 K and self-field) obtained in rolled 85-filament composites. For many 19-filament tapes,  $J_c$  values of 56 kA/cm<sup>2</sup> have been reached. In addition, individual filaments extracted from an 85-filament tape ( $J_c$  of 58 kA/cm<sup>2</sup>) have transport  $J_c$  values of greater than 80 kA/cm<sup>2</sup> at 77 K and self-field. The fact that a distribution of  $J_c$  values was found for different filaments extracted from a multifilament sample demonstrates that opportunities continue to improve the overall  $J_c$  of Bi-2223 tapes.

Achieving high  $J_c$  values is believed to stem in part from an improvement of the 2223 grain connectivity.

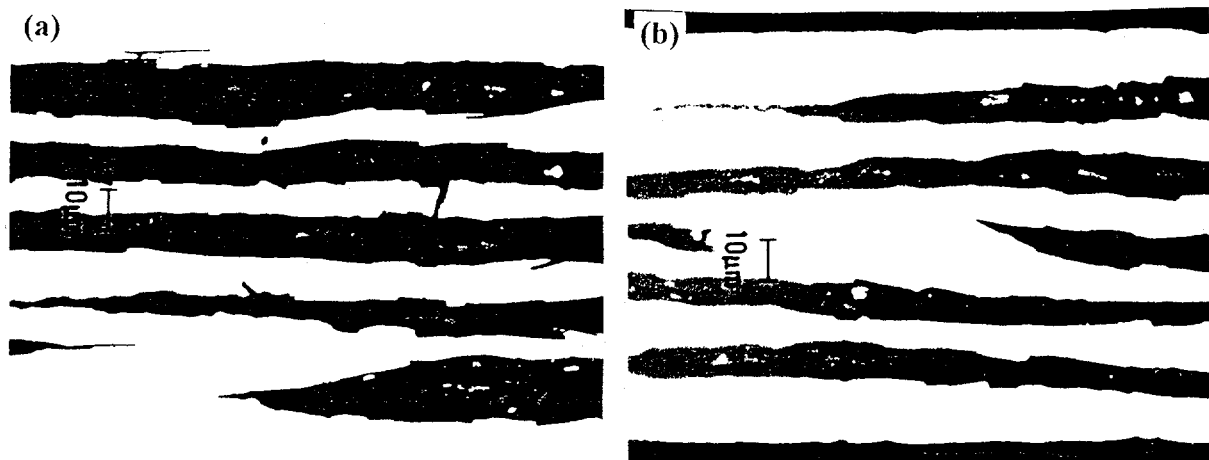


Figure 1. Optical micrographs from short transverse cross-sections of 19-filament tapes (a)  $J_c$  of 33 kA/cm<sup>2</sup> and (b)  $J_c$  of 56 kA/cm<sup>2</sup>, showing porosity reduction and texture improvement.

The microstructures of two 19-filament tapes with  $J_c$  of 33 and 56 kA/cm<sup>2</sup> are given in Figure 1a and 1b, respectively. Near the Ag-oxide interface, both samples are very dense and have well textured microstructures. However, the central regions of each filament for the low  $J_c$  sample has much more porosity and randomly aligned 2223 phase than for the high  $J_c$  tape. Although the  $J_c$  values have improved significantly, the amount of the secondary phases in the low  $J_c$  samples is similar to that in the high  $J_c$  samples.

For an 85-filament tape with  $J_c$  of 55 kA/cm<sup>2</sup>, the full width high maximum (FWHM) c-axis tilt misorientation angles determined from XRD rocking curves are 13.5° for the sample after exposing the Ag-oxide interface by etching and 15.1° for the same sample after polishing into the center of the tape (Fig. 2a). The texture difference in these two regions has been proved statistically significant by multiple sample studies. Surprisingly, these values are not much less than those reported for lower  $J_c$  samples determined by the same technique[5, 7-8]. In contrast, Kobayashi et al.[10] showed that the  $J_c$  values for multifilament tapes increase from 15 to 33 kA/cm<sup>2</sup> as the FWHM values decrease from 22 to 16°. These suggest that there may be other factors besides texture that influence intergranular current flow such as microcracks or gaps in 2223 colony boundaries. The degree of c-axis texture can also be estimated by measuring the resistivity ratio in directions perpendicular ( $\rho_n$ ) and parallel ( $\rho_r$ ) to the tape surface (Fig. 2b). Because the normal state resistance of each 2223 grain is highly anisotropic, a large  $\rho_n/\rho_r$  ratio at  $T_c$  suggests a higher degree of c-axis texture[11]. For an 85-filament sample with  $J_c$  of 50 kA/cm<sup>2</sup>, the  $\rho_n/\rho_r$  ratio is about 25. In contrast, Cho et al.[11] have measured that the  $\rho_n/\rho_r$  ratio is only in range of 4 - 10 for monofilament samples with  $J_c$  values of 12 - 24 kA/cm<sup>2</sup>.

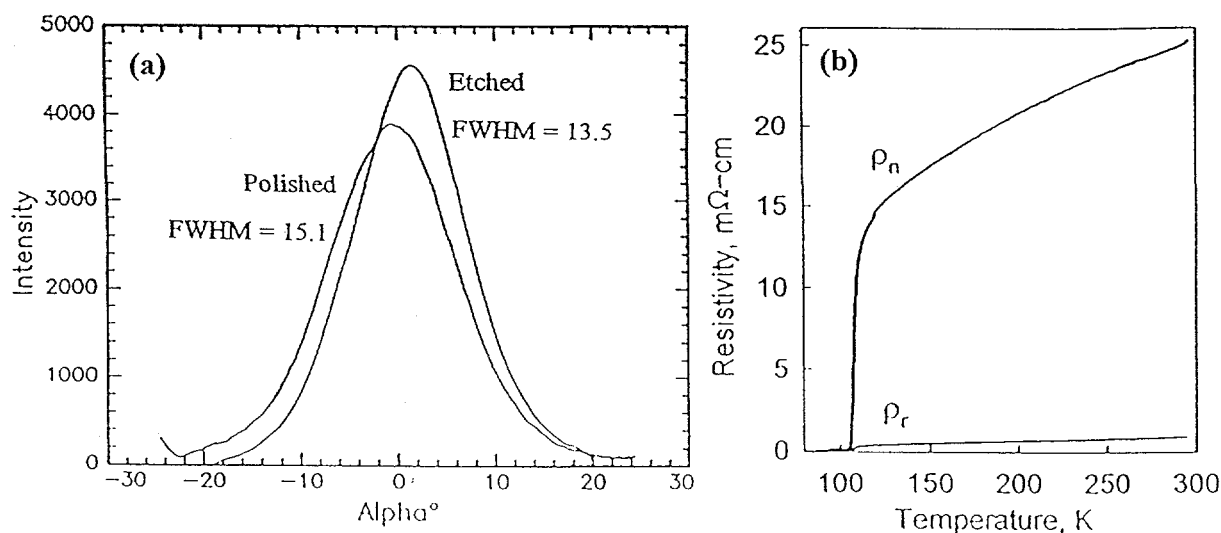


Figure 2. (a) XRD rocking curves of the (0024) peak of the 2223 phase and (b) resistivity curves of perpendicular and parallel to the tape surface of the high  $J_c$  85-filament sample

TEM micrographs showing typical microstructures of an 85-filament sample ( $J_c = 55 \text{ kA/cm}^2$ ) are given in Figure 3. Dense, highly-textured colonies dominate the interfacial region near silver (Fig. 3a and 3b). In this region, the c-axis tilt misorientation angles between colonies appear to be small with a high degree of connectivity, although some cracks are present as indicated by the arrows in Figure 3a and 3b. Colony boundaries are primarily ab-basal plane grain boundaries (BGB[12] or OABTWIST[13]) and edge colony boundaries (ECB[12] or ECTILT[13]). Some small angle c-axis tilt colony boundary (SCTILT[13] or SCB[12]) exists in these regions, apparently resulting from the need to accommodate changes in the filament thickness, especially near the filament ends. Overall, the colony microstructure appears to resemble that described by the brick-wall model[14] or the freeway model[12], and less like the railway-switch model[13].

In contrast to the interfacial region, the middle of the filament is quite porous and contains more secondary phases. As shown in Figure 3c, many of the 2223 colonies are heavily bent and the number of SCB and ECB boundaries are now comparable. The degree of c-axis tilt misorientation between colonies has also increased significantly. Figure 3d shows a porous region in which all boundary types (BGB, ECB, and SCB) can be found. To our knowledge, most previous microstructure studies by SEM[13,15] and TEM[15-16] have been carried out on monofilament samples with  $J_c$  in the range of 10 - 30  $\text{kA/cm}^2$ . Proportionally, these monofilament tapes contain less interfacial area. As a result, most of the samples tends to be porous and have poor texture, and SCB type boundaries predominate in these monofilament tapes. In our high  $J_c$  multifilament tapes, the interfacial regions account for the majority of the microstructure, comprising approximately 2/3 of the total filament cross section (see Fig. 3c). Our results indicate that high  $J_c$  is associated with the highly-textured and well-connected interfacial region. To further improve  $J_c$ , it is necessary to eliminate microcracks and to promote the microstructure of the interfacial region through the entire thickness of each filament by reducing porosity and secondary phases.

There is another microstructure feature that may play a role in limiting current in our high  $J_c$  samples. Our XRD results show that there is still a small amount of 2212 phase in the sample with  $J_c$  of 55  $\text{kA/cm}^2$ . In a series of studies on samples made a number of years ago in our laboratory, 2212 intergrowth regions with thickness values greater than one unit cell were predominantly localized at the (001) twist grain boundaries within the 2223 colonies[15-16]. Moreover, it was found that transport  $J_c$  increases as the frequency of these types of 2212 intergrowths decreases[16-17]. As a result, Umezawa et al.[16] suggested that such intergrowth

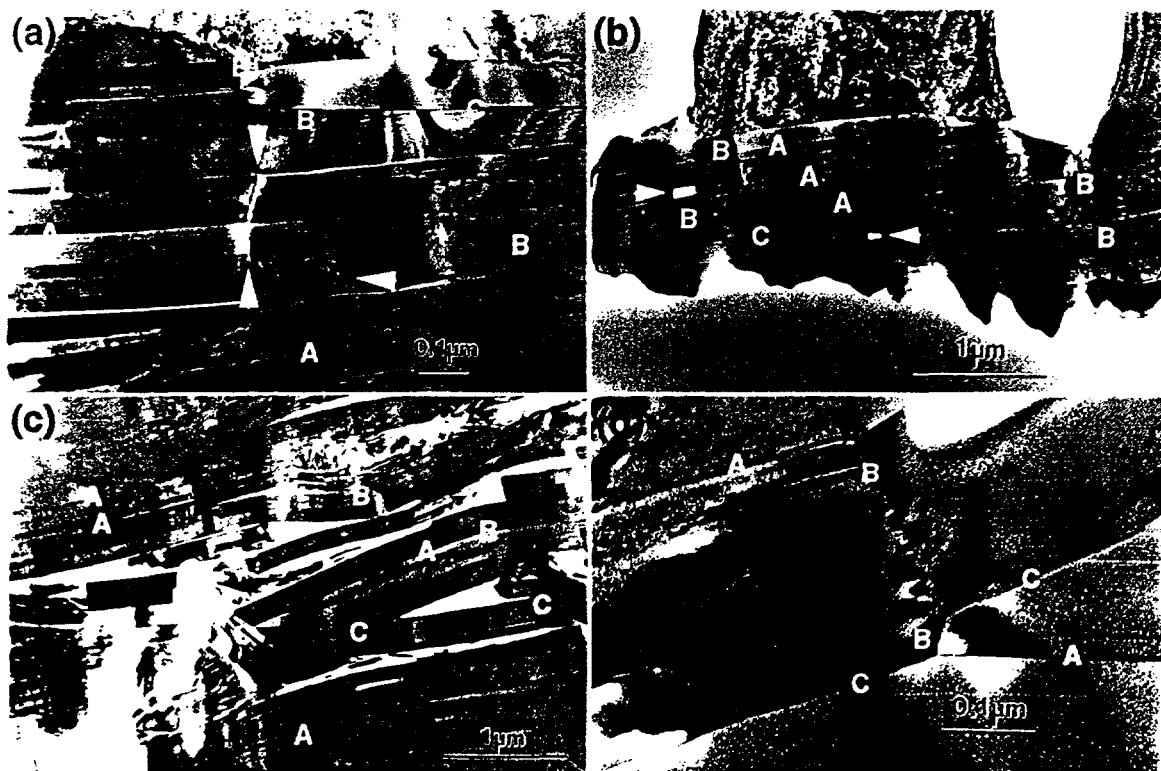


Figure 3. TEM micrographs, showing typical microstructures from an 85-filament sample ( $J_c$  of 55  $\text{kA/cm}^2$ ). (a) and (b) cracks and colonies in the interfacial regions, (c) and (d) colonies near the porous center regions. Arrow — microcrack, and A — BGB, B — ECB, C — SCB colony boundaries.

types control current flow along the c-axis. However, it is less clear how a 2212 intergrowth with a thickness of a single unit cell would influence current flow. Recently, Riley et al.[12] proposed a new 'freeway' model for current transport in BSCCO materials, in which intracolony current transport across high angle c-axis twist boundaries is invoked to account for the apparent relaxation of the biaxial alignment criterion for intergranular current flow in HTS materials. Based on this model, current flow in the c-axis direction and ab-plane transport play important roles in Bi-2223 composites. Depending on the relative current density and grain dimensions transport in either the c-axis direction or ab-plane may be limiting. Recent experimental evidence suggests that c-axis transport does occur in Bi-2223 composites under certain conditions[9]. In the context of the freeway model, the gating mechanism in high fields should be c-axis transport in the presence of 2212 intergrowths. Therefore, 2212 intergrowths could play a key role in limiting the intracolony current flow in Bi-2223 composites. An alternative perspective, based on relaxation of the biaxial alignment criterion at edge type colony boundaries with low c-axis tilt and high c-axis twist components, would suggest that Bi-2212 intergrowths would not play a role in limiting intercolony transport. Thus there appears to be opportunity to build upon the early work by [16], and develop new insight into current flow in high  $J_c$  Bi-2223 tapes.

## CONCLUSION

High  $J_c$  values of more than 58 kA/cm<sup>2</sup> at 77 K and self-field have been achieved in rolled multifilament Bi-2223 tapes. A transport  $J_c$  value of greater than 80 kA/cm<sup>2</sup> has been measured for individual filaments extracted from a multifilament sample. Microstructure and c-axis texture studies on these samples suggest that the high  $J_c$  values are associated with the highly textured and well connected Ag-interfacial regions. The colony boundaries in the dense interface region are mostly BGB and ECB types, while those in the porous center region are mostly SCB type. To further improve  $J_c$ , the most important factors are to eliminate microcracks and 2212 intergrowths within the colonies and improve c-axis texture by reducing porosity and secondary phases in the filament center.

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