

New Experiments Elucidating the Current Limiting Mechanisms of Ag-Sheathed (Bi,Pb)₂Sr₂Ca₂Cu₃O_x Tapes

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Abstract: Multiple current limiting mechanisms exist from the nanometer to millimeter scale in Ag-sheathed (Bi,Pb)-2223 tapes. Recent studies of the zero-field critical current density (J_c (0T, 77K)), the irreversibility field (H^*) and the crack microstructure elucidate these properties. We show that H^* (77K) can vary significantly over the range ~120-260 mT, independently of J_c (0T, 77K). Cracks, actual or incipient, exist on the sub to several hundred micron scale. Surface magneto optical imaging of whole tapes, correlated to subsequent ultrasonic fracture analysis of the bare 2223 filaments extracted by dissolving away the Ag shows that even composites having J_c (0T, 77K) values of 60 kA/cm² exhibit strong signs of unhealed rolling damage. These combined studies show that today's very best 2223 tapes are still far from full optimization.

Keywords: BSCCO-2223, critical current density, irreversibility fields, cracks, ultrasonic fractures.

INTRODUCTION

It is now well accepted that several current limiting mechanisms (CLM) operate simultaneously in Ag-sheathed (Bi,Pb)₂Sr₂Ca₂Cu₃O_x (2223) tapes. These CLM operate on multiple length scales from the nanometer scale of vortex pinning and grain boundary structure to the millimeter scale of cracks and filament sausageing irregularities. Because of this complexity, it is still not clear what the true limits to J_c of this system are. Thin film 2223 samples have been reported to have J_c (0T, 77K) values from 10⁵-10⁶ A/cm² [1-3], while analysis of resistivity data on bulk 2223 and 2212 single crystals has suggested that well above 100 kA/cm² is possible [4]. As of now, the largest reproducible (and thus verified) values of J_c (0T, 77K) are in the 70 kA/cm² range [5]. Single filaments extracted from 50 kA/cm² samples showed [6] a clear crossover from junction-control of J_c in self field (produced by occasional c-axis current flow) to vortex depinning within ab-planes in small applied fields. Magneto optical (MO) imaging also showed quasi-periodic, flux-leakage channels, which cross whole filaments transverse to the rolling axis, suggesting significant, but not total, interruption of the supercurrent by only

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partially healed ("incipient") cracks. We call such cracks "incipient", because the filaments do not fragment when the Ag is removed, but only after ultrasonic treatment, which does cause filament fragmentation into lengths consistent with the "incipient MO crack" separation [6,7]. Such experiments suggest at least three simultaneous strategies for raising J_c . At the nanoscale, these include: (1) improving c-axis misalignment so as to diminish the need for c-axis current flow; (2) raising flux pinning and the irreversibility field, $H^*(77K)$, which varies by at least a factor of 2 in "optimized" composites [8,9]; and (3) raising the effective current-carrying cross-section [6-7,10] by both reducing and healing fabrication damage.

EXPERIMENTAL DETAILS

The starting tapes were prepared in various ways as described in refs. 5-10. Experimental details of the single filament testing are given by Cai et al. [6-7], of the irreversibility field measurement by Anderson et al. [8], of variable Pb composition composite properties by Anderson et al. [9], and of intermediate deformation effects by Anderson et al. [10].

RESULTS

Irreversibility Fields of Ag-sheathed Bi(Pb)-2223 Composites. It is now becoming clear that the self-field J_c of 2223 tapes is rather sensitive to their size, aspect ratio and their test configuration [11]. Since all applications of 2223 require some in-field performance, there should be greater interest in the slope of the $J_c(H)$ characteristic and a recognition that $J_c(0T, 77K)$ is no longer valid as the only metric of 2223 performance. We have proposed [8] that the field-dependent properties can be usefully characterized by the irreversibility field, $H^*(77K)$, which is primarily determined by the flux pinning properties of the transport current path. Reference 8 describes methods of measuring H^* reliably, even in the presence of damage that causes ohmic components to the E-J traces [12]. Figure 1 shows the correlation between $H^*(77K)$ and $J_c(0T, 77K)$ for a variety of representative good quality tapes, taken from

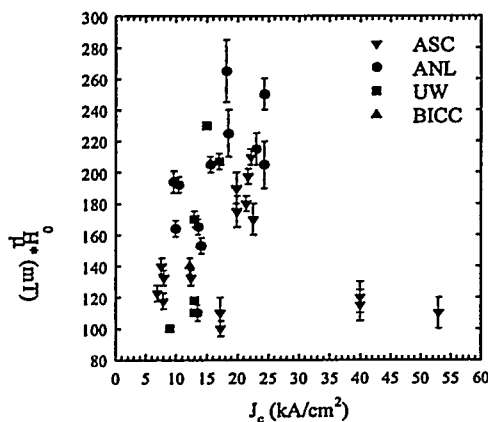


Fig. 1: Irreversibility fields $H^*(77K)$ measured in transport and $J_c(0T, 77K)$ for representative 2223 composite conductors.

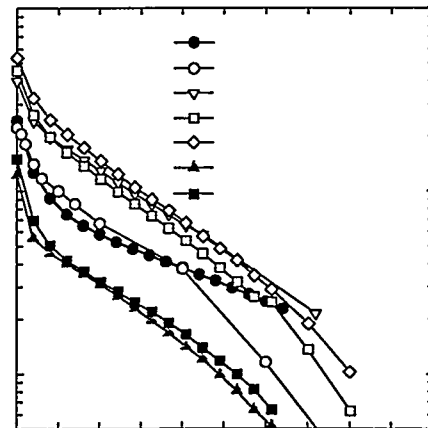


Fig. 2: $J_c(H, 77K)$ taken at $1 \mu V/cm$ for some tapes from figure 1. A crossover in J_c at higher

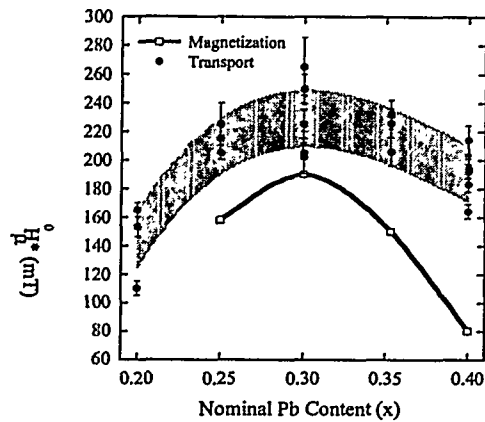


Fig. 3: $H^*(77K)$ for $(Bi_{1.8}Pb_x)_2Sr_2Ca_2Cu_3O_x$ composites with variable Pb content.

composite plots convey a very different message than those typically generated by the optimization of one single composite, where it is common to see that $H^*(77K)$ and $J_c(0T, 77K)$ both increase together as the process is optimized [13,14]. The potential for raising H^* is indicated by a recent experiment [9] on the influence of varying the Pb from 0.2 to 0.4 in a $(Bi_{1.8}Pb_x)_2Sr_2Ca_2Cu_3O_x$ composition series. The mean of multiple measurements of $H^*(77K)$ varied from 140 mT (0.2Pb) to 230 mT (0.3 Pb) to 200 mT (0.4 Pb), with individual maxima above 260 mT. Since so few measurements of H^* have been made in 2223 tapes, we believe that there is still considerable untapped potential in understanding its variation. Measuring not just $J_c(0T, 77K)$, but also $H^*(77K)$ is a vital first step towards understanding this potential.

Magneto-optical imaging (MO) and ultrasonic fracturing studies. Fig. 4a shows a plan-view MO image of a 19 filament, 60 kA/cm² tape. The individual shape of the five upper filaments lying closest to the imaging film is quite apparent [15]. A considerable local variability of each filament's flux shielding and a quasi-periodic transverse flux penetration on a scale of a few hundred microns, which in many cases cross the whole filament, is also visible. We believe that these are partially healed cracks [6] that form during rolling, probably during the intermediate rolling step. Subsequent heat treatment partially heals this damage, such that when the Ag is etched away with a NH_4OH/H_2O_2 mixture, the filaments do not fall apart into small pieces. However, after putting the bare filaments into an ultrasonic bath for 15 minutes, they fracture into a relatively monosize distribution with a characteristic length $\sim 200 \mu m$, as shown in figure 4b and 4c.

The influence of varying the intermediate rolling (IR) conditions on the MO and ultrasonic fracture (UF) response is shown in Figure 5. We compared large (152 mm) and small diameter rolls (38 mm), since larger diameter rolls shift the largest principal stress towards the transverse axis, thus favoring cracks which lie parallel to the rolling axis, whereas the smaller diameter rolls tend to favor transverse cracking [16,17]. It is indeed clear from Figure 5 that the mean size after UF is smaller for the larger rolls and, from the MO images, that the larger rolls exhibit fewer "incipient" transverse cracks [10]. We stress, however, that the incipient cracks seen in

representative sources. Figure 1 shows that, although there is a general tendency for both $J_c(0T, 77K)$ and $H^*(77K)$ to increase together, there is no universal correlation between the two. Particularly striking is the fact that the higher $J_c(0T, 77K)$ tapes have only moderate $H^*(77K)$ values of ~ 120 mT, while other composites with much lower $J_c(0T, 77K)$ can have H^* exceeding 250 mT. As noted in figure 2, the higher values of H^* produce much less field-dependent J_c values and consequently exhibit a J_c crossover at higher fields. The implication of these data is that the connectivity, which determines the local current-carrying cross-section and thus I_c , and the flux pinning, which determines the nanoscale, local intragrain J_c , are quite independent parameters, which need independent optimization. Such multi-

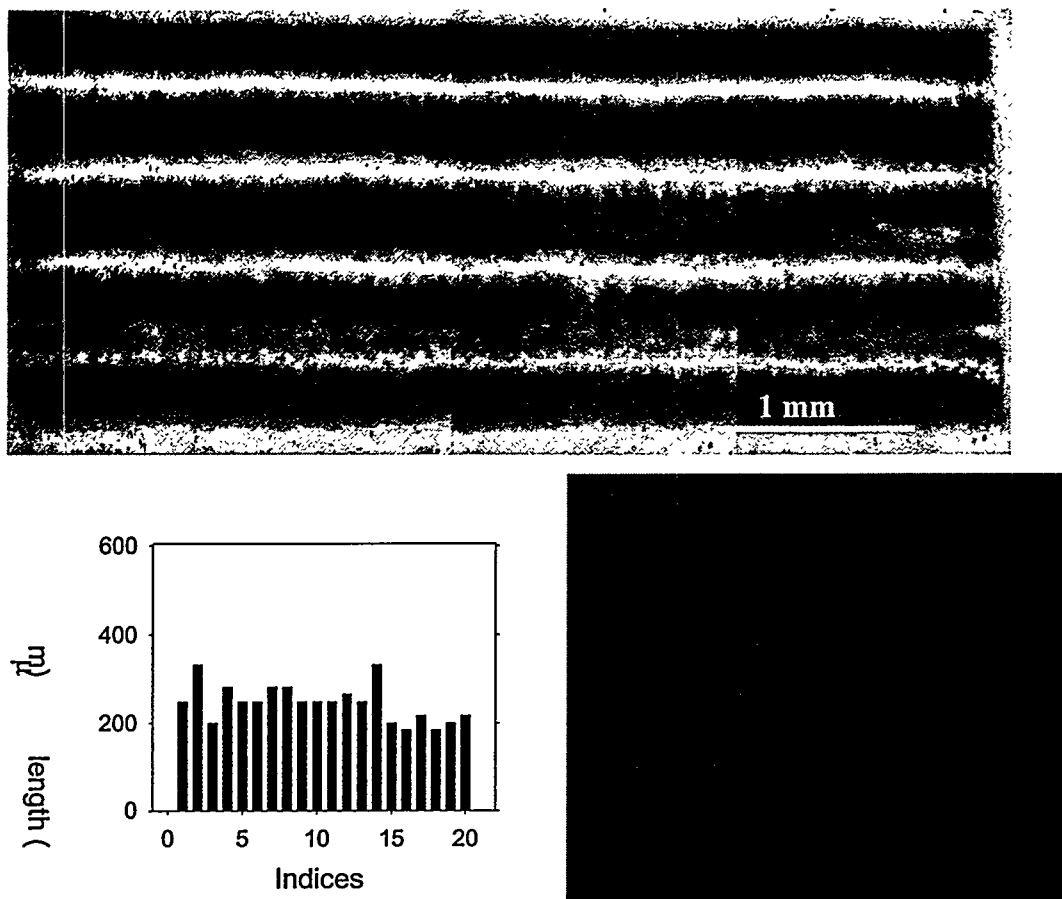


Fig. 4: a. Plan view magneto optical image taken at 10K after zero field cooling and the application of 40 mT. The imaging film was placed directly on top of the Ag sheath of the 19 filament composite, for which $J_c(0T, 77K)$ at $1 \mu V/cm$ was 60 kA/cm^2 ; b. Distribution of piece lengths after ultrasonic treatment of exposed filaments; c. Plan view of pieces remaining after ultrasonic fracturing.

the MO image do not cause complete fracture when the filaments are exposed, nor do they reduce the J_c to zero. They are weak links of unknown strength at this time.

DISCUSSION

The data presented here, obtained largely on very high J_c tapes, suggest that there are considerable opportunities to improve the $J_c(H)$ of BSCCO-2223 tapes by several times. Figures 1 and 2 show that $J_c(0T, 77K)$ and $H^*(77K)$ can vary independently. Although the connectivity of any individual tape largely determines the magnitude of $J_c(77K)$ (which is experimentally defined by I_c/A) over quite a significant range of field, it is also clear from figure 2 that a significant improvement in J_c at all fields would be obtained if optimum H^* and $J_c(0T, 77K)$ properties could be combined. For example, the linear combination of the least field-dependent and the highest $J_c(0T, 77K)$ composites from Fig. 2 would raise $J_c(0.1T)$ from 13.5 to $\sim 20 \text{ kA/cm}^2$. However, the complexity of the factors controlling H^* is only partially

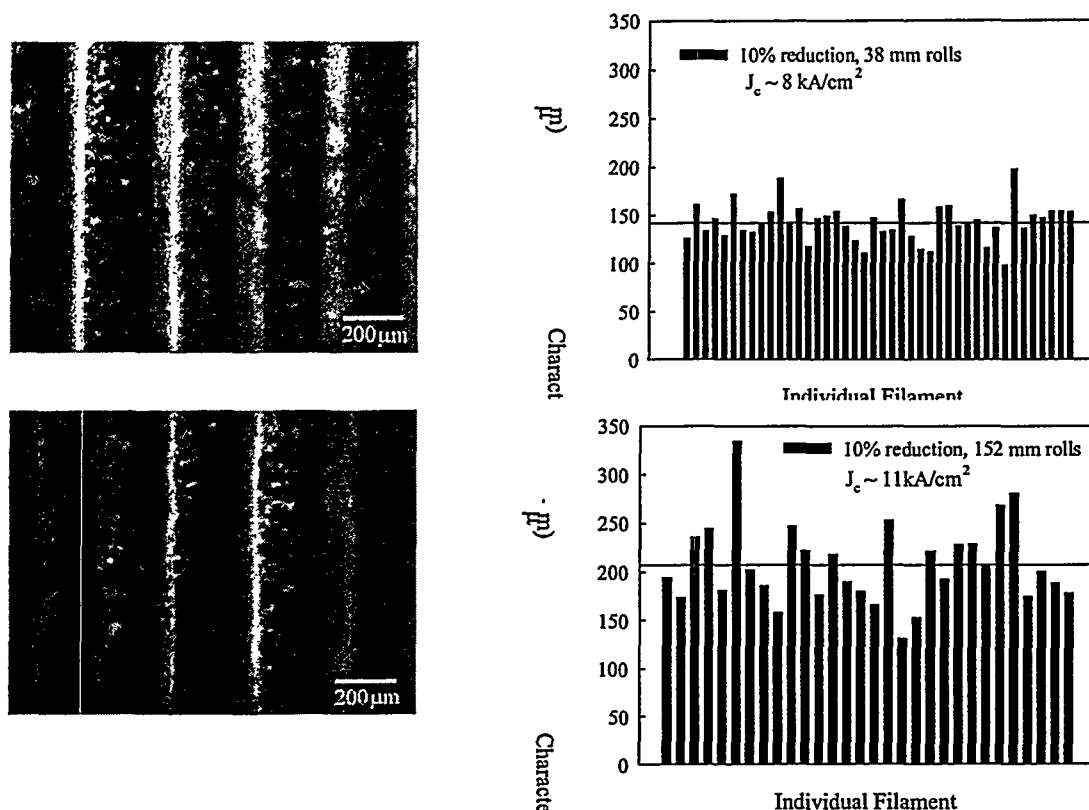


Fig. 5: At left, plan view magneto-optical images taken at 12K and 40 mT after zero field cooling for 61 filament tapes rolled with 38 mm (above) and 152 mm (below) diameter rolls. A distinctly greater transverse flux penetration is seen in the sample rolled with smaller rolls and this correlates well to the average fracture size after ultrasonic fracturing. These data are shown at right. The characteristic length is defined as the square root of the area of the fractured pieces, as determined by plan view digital image analysis [10].

described by Figs. 1-3. Recent STEM-EDS examination of similar, very high J_c composites by Holesinger et al. [18] showed that the local Pb content of 2223 grains fell to as little as $\text{Pb}_{0.13}$ in the vicinity of Pb-rich phases and 2212 grains, values well below the starting $\text{Pb}_{0.3}$ of the tape and the optimum Pb content of Fig. 3 but consistent with a low H^* . However, we have measured $H^*(77\text{K})$ as high as 340 mT on 100 μm long regions of extracted filaments of similar 60 kA/cm^2 tapes. Thus variations of H^* of order 2 appear to be associated with the phase heterogeneity of the filaments. It is also clear from Fig. 2, however, that the zero field J_c dominates $J_c(H)$ over a large field range, thus emphasizing the need to address the connectivity aspects of J_c , which are largely determined by the void and crack population through their influence on the effectively connected cross-section $A_{\text{effective}}$. The quasi-periodic large-scale flux penetrations (incipient cracks) of Figs. 4 and 5 are tantalizing, for they signal the presence of significant larger-scale fabrication damage (ref. 18 also shows that there is local cracking on the $\sim 0.1 \text{ μm}$ scale too), which locally minimizes $A_{\text{effective}}$, thus both limiting I_c and serving as strain-induced crack-initiation sites. These multi-scale cracks, perhaps fractal in nature, suggest that the central issue of BSCCO-2223 processing is to find means of raising the filament mass density, without introducing too much cracking [19, 20]. The intermediate rolling steps do not

appear to be able to do this as effectively as is needed, since optimum reduction is of order 15% [10], while the fractional porosity is significantly greater [21]. The quasi-periodic damage seen in Figs. 4 and 5 suggest that additional factors of several times in J_c are potentially available for better densification or crack-healing treatments. Increases in J_c of this order at $\sim H^*/2$ would go far towards developing truly cost- and performance-effective BSCCO-2223 conductors [22,23] capable of broad application.

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