Significantly enhanced critical current density in Ag-sheathed (Bi,Pb)\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x} composite conductors prepared by overpressure processing in final heat treatment

Y. Yuan,\textsuperscript{a}) J. Jiang, X. Y. Cai, D. C. Larbalestier, and E. E. Hellstrom

Applied Superconductivity Center, University of Wisconsin–Madison, 1500 Engineering Drive, Madison, Wisconsin 53706

Y. Huang and R. Parrella

American Superconductor Corporation, Two Technology Drive, Westborough, Massachusetts 01581

(Received 5 November 2003; accepted 21 January 2004)

Overpressure (OP) processing otherwise fully treated, commercial Ag-sheathed multifilament (Bi,Pb)\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x} (2223) composite conductors increased the critical current density \(J_c\) (0.1 T, 77 K) by 37% to 30.8 kA/cm\textsuperscript{2} and the self-field \(J_c\) (SF, 77 K) to 69.6 kA/cm\textsuperscript{2}. These improvements were obtained on full-size high current conductors such that critical current \(I_c\) (0.1 T, 77 K) reached 80.6 A and \(I_c\) (SF, 77 K) 181.7 A, even though there was a very strong self-field suppression of \(I_c\). Estimated values for the non-self-field-limited \(I_c\) and \(J_c\) (0 T, 77 K) reached 235 A and 90 kA/cm\textsuperscript{2}. Scanning electron microscopy and superconducting quantum inference device measurement revealed that OP processing effectively suppressed cracks, porosity, and the volume fraction of the Bi\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{x} (2212) phase, which are all major current-limiting mechanisms in present 2223 conductors. © 2004 American Institute of Physics. [DOI: 10.1063/1.1682675]

Even though Ag-sheathed 2223 and 2212 tapes are currently the only first-generation high temperature superconductors available in lengths suitable for large-scale electrical applications, major current-limiting mechanisms limit their performance. Typical critical current density \(J_c\) (self-field—SF, 77 K) for the more widely studied 2223 tapes ranges from 30 to 40 kA/cm\textsuperscript{2} and \(J_c\) (0.1 T, 77 K) from 14 to 20 kA/cm\textsuperscript{2} for the best commercial 2223 tape produced by the oxide-powder-in-tube (OPIT) method.\textsuperscript{1–3} However, current reconstructions of magneto-optical images show local regions of 2223 tape with \(J_c\) as high as 300 kA/cm\textsuperscript{2},\textsuperscript{4} while \(J_c\) up to 1000 kA/cm\textsuperscript{2} has been achieved in 2223 thin films,\textsuperscript{5,6} suggesting there is still much headroom to improve \(J_c\) (SF, 77 K) in 2223 tape.

The currently practiced OPIT fabrication of Ag-sheathed 2223 composites is a complicated process,\textsuperscript{7,8} in which a mixture of 2223 precursor powders with nominal composition of (Bi,Pb)\textsubscript{2}Sr\textsubscript{2}Ca\textsubscript{2}Cu\textsubscript{3}O\textsubscript{c} packed into a silver tube, then drawn and rebundled to a multifilamentary composite, and rolled into tape. This as-rolled tape subsequently goes through a thermomechanical cycle that consists of a first heat treatment (HT1), an intermediate rolling (IR), and a final heat treatment (FHT). The FHT includes a second heat treatment (HT2) and a separate low temperature “post anneal” (PA) or a heat treatment combining HT2 and PA.\textsuperscript{9,10} About 80% of the precursor oxide powder reacts to 2223 phase in HT1. IR helps densify the superconducting filaments while HT2 and any FHT further increase 2223 formation. However, the IR creates an extensive network of cracks that cannot be fully healed in the subsequent heat treatments.\textsuperscript{11,12} A significant amount of porosity, ~20% as indicated by mass density measurement, remains in multifilament 2223 tape after HT2 or FHT at 1 bar.\textsuperscript{13,14} These residual cracks and porosity interrupt the grain connectivity and current paths, thus reducing \(J_c\). It is generally accepted that 2223 forms from 2212 and alkaline earth cuprate (AEC) phases in the presence of a transient liquid phase. Residual 2212, present as half-cell intergrowths in 2223 grains and as grains in 2223 colonies, was detected by multiple techniques in 2223 after HT2 or FHT.\textsuperscript{2,15,16} Increases in \(J_c\) were found to correlate strongly with decreases in the volume fraction of 2212.\textsuperscript{2,10,15} A central question in further improving the performance of 2223 tapes is how to eliminate the current-limiting mechanisms of porosity, cracks, and residual 2212 to further improve the \(J_c\).

Previously, overpressure (OP) processing was used to heat treat as-rolled 2223 tape, as well as 1 bar processed HT1 and IR tapes.\textsuperscript{17} We showed that OP processing reduced the crack density and porosity by 11%–16% and increased \(J_c\) (SF, 77 K) by 75% to 59 kA/cm\textsuperscript{2} and \(J_c\) (0.1 T, 77 K) by 82% to 22 kA/cm\textsuperscript{2} compared to samples processed identically at 1 bar. The mass density of OP processed samples reached 97% of 2223 theoretical density. This letter shows that OP processing also works very well when applied only as the final heat treatment (FHT) after all other processing (HT1, IR, and HT2) is done at 1 bar.

Samples up to 8 cm long of a fully processed (FHT), high \(J_c\) (159.6 A) multifilamentary production tape produced by American Superconductor Corp. subsequently received an additional heat treatment at 1 bar or 148 bar overpressure processing (FHT + 1b or FHT + OP). The external cross-section dimensions of the tape were \(~4.0\text{ mm}\times0.21\text{ mm}\). These FHT + 1b and FHT + OP samples were annealed using a heat treatment cycle similar to that published elsewhere.\textsuperscript{17} The total OP pressure of the Ar/O\textsubscript{2} mixture was 148 bar. The calculated oxygen partial pressure (pO\textsubscript{2}) was 0.081 bar.

\textsuperscript{a})Electronic mail: yongwenyuan@wisc.edu
Figure 1 shows SEM images of the three samples. The common feature of FHT and FHT+1b samples was a large amount of residual porosity and cracks. In contrast, the FHT+OP sample was dense, the fabrication cracks were well healed, and the porosity was effectively removed. Measurements of the BSCCO cross section showed that the total cross-sectional area of the filaments was reduced from 2.99 × 10⁻³ cm² for FHT and FHT+1b samples to 2.61 × 10⁻³ cm² for FHT+OP samples. The micrographs also show that all three samples contain current limiting phases, including discrete 2212 grains within the 2223 colonies, AECs, and (Bi,Pb)₂Sr₂Ca₂Cu₃O₉ (3221). The FHT+1b sample [Fig. 1(b)] had the most and largest grains of 3221.

The $V-I$ characteristics of the samples were measured using the four-probe technique in liquid nitrogen with the external magnetic field applied perpendicular to the broad sample surface, which is approximately parallel to the $c$ axis of the sample. Using the 1 μV/cm criterion, the $I_c$ (77 K) values of the FHT+OP sample in self-field and 0.1 T were 181.7 and 80.6 A, respectively. The corresponding $J_c$ values in self-field and 0.1 T are 69.6 and 30.8 kA/cm². Figure 2 shows that these $J_c$ values are 30% and 37% higher than the corresponding $J_c$ values of 53.4 and 22.5 kA/cm² for the FHT sample and 23% and 30% higher than those of 56.4 and 23.7 kA/cm² for the FHT+1b sample. 30.8 kA/cm² is the highest $J_c$ (0.1 T, 77 K) value that has been reported so far for 2223 tape.

The measured $I_c$ at self-field for the FHT+OP sample was 181.7 A. However, Fig. 2 shows the $J_c$-$B$ curves become flattened at low field due to very large self-field suppression effects on $J_c$. The inset shows the extrapolation of the critical current for the FHT+OP sample to zero field $I_c$ (0 T, 77 K) based on the model proposed by Kim et al. where $1/I_c(B) = B/I_c(0)B_0 + 1/I_c(0)$. $B_0$ is the characteristic field and $I_c(0)$ is the critical current at 0 T. The extrapolated $I_c$ (0 T, 77 K) for the FHT+OP sample is 235 A, corresponding to $J_c$ (0 T, 77 K) = 90 kA/cm².

Figure 3 shows the magnetization moment as a function of temperature taken in a Quantum Design superconducting magneto...
quantum interference device (SQUID) magnetometer for the samples before and after they were rolled with a 30% reduction in thickness. As shown in Fig. 3(a), the unrolled samples all had a sharp transition, which is due to the strong shielding by the 2223. After rolling, the magnetization transition was much broader and had a kink at about 80 K [Fig. 3(b)], which is close to the \( T_c \) for 2212. The broadening and kink at \( \sim 80 \) K are both thought to be caused by 2212 in the sample that is exposed when the 2223 is crushed in the rolling process.\(^2\)\(^{10}\)\(^{20}\)

A method has been proposed\(^2\)\(^{10}\)\(^{20}\) to evaluate the relative amount of 2212 in the tape, which defines a 2212 SQUID index. As shown in Fig. 3(b), the 2212 SQUID indices are 0.32, 0.29, and 0.11 for FHT, FHT+1b, and FHT+OP samples, respectively. The much smaller value of 0.11 for the OP sample shows that OP processing decreased the 2212 content relative to the FHT and FHT+1b samples. In fact, the value of 0.11 is the smallest 2212 SQUID index that we have measured for 2223 samples from a variety of sources.\(^2\)\(^{10}\)\(^{20}\)

\( J_c \) was enhanced by reduction of cross-section area and by improved connectivity. OP processing reduced porosity and cracking from \( \sim 20\% \) of the cross-section area in the FHT and FHT+1b samples to less than 5% in the FHT+OP sample, which proportionally enhanced the active cross-section area that carried current. The 30% enhancement of \( J_c \) (0.1 T, 77 K) in FHT+OP samples compared to FHT+1b samples was partially due to the 13% reduction in cross-section area, but the remaining 17% enhancement must be due to the improvement in connectivity through eliminating current-blocking cracks and pores, reducing long-range obstructions of 2212 streaks and removing wetting phases at grain boundaries by the precipitation of 3221 seen in Fig. 1. Gurevich and Friesen\(^21\) have shown that nonlinear effects on critical currents are due to removing dissipation obstructions in superconductors, some of which may be pores and grain-boundary wetting phases. 2212 grains and intergrowths can play a very important role in limiting \( J_c \), as pointed out earlier by Umezawa et al.\(^15\) Evidently OP processing reduces all these obstructions and thus results in the \( J_c \) increase in FHT+OP samples. Present studies using local probes such as magneto-optical imaging are investigating the local current obstructions in exceptionally high \( J_c \) samples such as these FHT+OP samples.

In conclusion, we have shown that OP processing 2223 tape after FHT results in a significant increase in transport properties. \( J_c \) (0.1 T, 77 K) of 30.8 kA/cm\(^2\) and \( I_c \) (SF, 77 K) of 181.7 A, with \( J_c \) (SF, 77 K) of 69.6 kA/cm\(^2\) and an extrapolated \( J_c \) (0 T, 77 K) of 90 kA/cm\(^2\) have been achieved. Even though \( J_c \) (SF, 77 K) of 74 kA/cm\(^2\) was previously reported in a Bi-2223 tape,\(^8\) as commented in the literature,\(^2\) the external cross-section dimensions (1.78 mm×\( \sim 0.08 \) mm) and critical current \( I_c \) (\( \sim 30 \) A) of that tape were small, so the self-field suppression of \( I_c \) was also very small. The increase in \( J_c \) in FHT+OP samples is attributed to the OP process decreasing the density of cracks, pores, and residual 2212 content.

This work was supported by the Department of Energy-Energy Efficiency and Renewable Energy program and also benefited from facilities supported by the National Science Foundation—MRSEC.