Transport critical current density of Bi–Sr–Ca–Cu–O/Ag superconductor tapes with addition of magnetic nanopowder $\gamma$-Fe$_2$O$_3$

S.Y. Yahya$^1$, M.H. Jumali, K.T. Lau$^2$, R. Abd-Shukor$^*$

School of Applied Physics, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Received 29 November 2004; revised 13 January 2005; accepted 3 February 2005

Available online 21 June 2005

Abstract

The effect of sintering temperature on the transport properties of Ag-sheathed-(Bi$_{1.6}$Pb$_{0.4}$)Sr$_2$Ca$_2$Cu$_3$O$_{10-\delta}$($\gamma$-Fe$_2$O$_3$)$_{0.01}$ superconductor tapes prepared by the powder-in-tube technique with sintering time fixed at 50 h has been investigated. The maximum transport critical current density, $J_c$ of 6490 A/cm$^2$ at 77 K, was observed at sintering temperature of 845°C. A further single intermediate rolling step increases $J_c$ to 9560 A/cm$^2$. Sintering temperature from 830 to 845°C increases the 2223 phase content and resulted in improved $J_c$. At 850°C, the content of 2223 phase decreased resulting in a corresponding decrease in $J_c$. X-ray diffraction patterns suggest that the 2212 phase reacts with non-superconducting phase such as CaCuO$_2$, (SrCa)$_2$CuO$_3$, CaO, and CuO to form the 2223 phase. Samples without $\gamma$-Fe$_2$O$_3$ prepared under the same condition showed a lower $J_c$ with maximum at 1560 A/cm$^2$. Our results show that nanomagnetic $\gamma$-Fe$_2$O$_3$ addition improved $J_c$ which supports previous calculations on the possibility of frozen flux superconductor with nanomagnetic addition in this class of materials.

© 2005 Elsevier Ltd. All rights reserved.

Keywords: Superconductor tapes; Transport critical current density; Flux pinning; Magnetic nanopowder

1. Introduction

Wide application of (Bi,Pb)-2223/Ag tapes is still a challenge despite many years of research effort to improve their critical current density, $J_c$. This is partly due to their weak flux pinning capability. The microstructure of partial melt processed Ag/Bi-2223 tape exhibits a high degree of texture and uniformity along the Ag-superconductor interface. Its micaceous properties support self-aligning of the grains in the deformation process and crystal growth which makes the tape production process easier and more straightforward compared to other high $T_c$ superconductors [1].

In order to enhance their flux pinning capabilities many attempts have been made by introducing artificial pinning sites. Some of the techniques include precipitates of 0.1 μm Y$_{211}$ in single crystals or cylindrical tracks created by heavy ion bombardment [2], amorphous tracks produced in Bi$_2$Sr$_2$CaCu$_2$O$_8$ by proton irradiation [3], neutron irradiation of HgBa$_2$CaCu$_2$O$_{6+x}$ [4] and various atomic substitutions [5]. However, each of these techniques has its own difficulties when applied in large-scale production [6].

To overcome these problems, addition of nanometer size particles as pinning centers has been performed. Previous study on the conventional superconductors showed that defects in dimensions comparable to the superconducting coherence length are much more effective in pinning flux lines. Nanosize MgO increases the critical current when the maximum heat treatment temperature was 910°C [7] but no significant enhancement in $J_c$ was observed after nanometric SnO$_2$ powder was added into YBCO [8].

Flux line network and magnetic texture can interact effectively if their characteristic scales have the same order of magnitude. The characteristic scales for flux line network is the coherence length $\xi$ which is about 10 nm in the high $T_c$ superconductor materials. Another characteristic length is the penetration depth $\lambda$ which is in the range of 60–1000 nm. In a magnetic system with characteristic length $L$, where $\xi < L < \lambda$, strong interaction
between flux line network and magnetic subsystem can be expected [9]. It is interesting to experimentally investigate further the possibility of a frozen flux superconductor in such a superconductor-magnet hybrid system.

Based on the above considerations, magnetic nanopowder $\gamma$-Fe$_2$O$_3$ has been introduced into Ag/BSCCO system to act as flux pinning centers. The effect of sintering temperature on the $J_c$ of these tapes are studied. The results are compared with tapes without $\gamma$-Fe$_2$O$_3$ addition.

2. Materials and methods

Precursor powders with nominal 2223 composition such as Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ was prepared by solid-state reaction method. In this method, high-purity powders (>99.9%) of Bi$_2$O$_3$, SrO, PbO, CaO, and CuO were mixed and heated to produce BSCCO powder with 2212 phase as the majority phase, 2223 phase and other phases. The powders were ground and added with 0.01wt% $\gamma$-Fe$_2$O$_3$ before being packed into Ag tube with outer diameter of 6.03 mm and inner diameter of 4.43 mm. This wt% is chosen based on our previous studies on bulk 2223 samples where 0.01wt% of $\gamma$-Fe$_2$O$_3$ optimized the superconducting properties of the system [10].

The tube was grooved rolled, drawn into wire and then flat rolled into tape form. All deformations processed were carried out using a rolling cylinder 20 mm in diameter and rolling speed of about 0.6 m/min. The tapes were cut into 2 cm long sections and divided into four groups. Each group was sintered for 50 h at different sintering temperature (Table 1). The S1 sample was sintered at 830 °C, S2 at 840 °C, S3 at 845 °C, and S4 at 850 °C. S3** are tapes which underwent intermediate rolling and sintered for 100 h at 845 °C. S5, S6, and S8 are tapes without $\gamma$-Fe$_2$O$_3$ addition sintered at various temperatures for 50 h. S7 are tapes without $\gamma$-Fe$_2$O$_3$ which underwent intermediate rolling and sintered for 100 h at 845 °C. All sintering process was carried out in air.

The phase of the tapes were identified using the powder X-ray diffraction (XRD) method using a Bruker D8 Advance Diffractometer with a CuKα source. The transport critical current $I_c$ was measured by a DC four probe method using the 1 µV/cm criterion at 77 K and $J_c$ was calculated by dividing the critical current ($I_c$) with the corresponding superconductor core cross-sectional area of the tape.

3. Results and discussions

Fig. 1 shows randomly oriented rod-like shape of $\gamma$-Fe$_2$O$_3$ with opposite length of about 200 nm and radius of 50 nm. Table 1 shows $J_c$ for different sintering temperature. It is clearly seen that $J_c$ is strongly correlated with the sintering temperature with optimum value at 845 °C. Fig. 2 shows the XRD patterns for the Ag/BSCCO-$\gamma$-Fe$_2$O$_3$ tapes sintered in air for 50 h at different temperature. This figure indicates that the peak intensity for the (00l) plane of the 2223 phase increased as the sintering temperature is increased from 830 to 845 °C. This suggests that the c-axis orientation (texture of grains) increased with sintering temperature. At higher temperature of 850 °C, the 2223 phase decomposes into non-superconducting phase that may result in the corresponding decrease in the superconducting properties. The volume fraction of the 2223 phase (Bi-2223) and 2212 phase (Bi-2212) was determined as ratio of X-ray peak intensity of the (0 0 14) plane of the 2223 phase to (0 0 8) plane of 2212. This ratio was measured by a DC four probe method using the 1 µV/cm criterion at 77 K and $J_c$ was calculated by dividing the critical current ($I_c$) with the corresponding superconductor core cross-sectional area of the tape.

The 2223 content increases with sintering temperature up to 845 °C followed by a decrease at higher temperature. This increment in 2223 phase is believed to be the result of the conversion of 2212 phase to the 2223 phase through appropriate reactions between 2212 and minor phase such as

![Fig. 1. Scanning electron micrograph of rod-like structure of $\gamma$-Fe$_2$O$_3$.](image)
CuO, CuCuO₂, (SrCa)₂CuO₃, and CaO. At higher temperatures, the 2223 phase decreased by decomposing into non-superconducting phase. This result shows that the presence of higher percentage of the 2223 phase contributed to better \( J_c \).

In this work maximum \( J_c \) of 6490 A/cm² has been obtained for S3. A further single intermediate rolling step increases \( J_c \) to 9560 A/cm² (for sample S3**). The \( J_c \) for Ag/BSCCO tapes without \( \gamma\)-Fe₂O₃ addition sintered at 845 °C for 50 h show a maximum at 1560 A/cm². This shows that the addition of magnetic nanopowder \( \gamma\)-Fe₂O₃ can improve the flux pinning ability in superconductor tape.

The \( J_c \) for sample S7 which is not added with \( \gamma\)-Fe₂O₃ is much lower than sample S3 even though S7 has a higher amount of the 2223 phase. No plate-like feature or alignment is observed in the microstructure of all samples. For example, Fig. 3 shows the SEM for S3. Hence, increase in \( J_c \) for the \( \gamma\)-Fe₂O₃ added samples cannot be due to the microstructure or the volume fraction of the 2223 phase. We attribute this increase as due to enhanced flux pinning as a result of addition of magnetic nanopowder \( \gamma\)-Fe₂O₃. This is consistent with previous calculations which show that with certain amount of magnetic nanorod in a superconductor matrix, a frozen flux superconductor can be achieved [9].

In general, most \( J_c \) values of Bi-based tapes at 77 K are much higher than the one reported here. Higher \( J_c \) in many of the previous reports are achieved through various preparation steps which is not the main objective of this work. The main objective of this work is to make a direct comparison between tapes with and without \( \gamma\)-Fe₂O₃ prepared under the most elemental preparation condition. We believe that \( \gamma\)-Fe₂O₃ can further enhance the \( J_c \) of the Bi-based tapes if prepared under the optimum condition.

Magnetic impurities generally suppress superconductivity. However, our result shows that magnetic \( \gamma\)-Fe₂O₃ can be employed to enhance the flux pinning capability of Ag/BSCCO superconductor tapes and improve the transport critical current density. This is in line with previous calculation on frozen flux superconductor with magnetic particles as pinning centers.

Acknowledgements

This work has been supported by the Ministry of Science, Technology and Innovation of Malaysia under IRPA Grant No. 09-02-02-0072EA199.

References

[1] U.P. Trociwitz, P.R. Sahm, R.E. Korital, L. Brandao, C. Bacaltchuk, J. Schwartz, The influence of BaO₂ additions on microstructure and superconducting properties of Bi₁₂Sr₀₈Ca₂Cu₃O₁₀₋ₓ, Physica C 366 (2002) 80–92.
[2] L. Civale, A.D. Marwick, T.K. Worthington, M.A. Kirk, J.R. Thompson, L.K. Elbaum, Y. Sun, J.R. Clem, F. Holtzberg, Vortex confinement by columnar defects in yttrium barium copper oxide (YBa₂Cu₃O₇) crystals: enhanced pinning at high fields and temperatures, Phys. Rev. Lett. 67 (1991) 641–651.
[3] L.K. Elbaum, J.R. Thompson, R. Wheeler, A.D. Marwick, C. Li, S. Patel, D.T. Shaw, P. Lisowski, J. Ullman, Enhancement of persistent currents in Bi₁₂Sr₀₈Ca₂Cu₃Oₓ tapes with splayed columnar defects induced with 0.8 GeV protons, Appl. Phys. Lett. 64 (1994) 3331–3333.
[4] J. Schwartz, S. Nakamae, G.W. Raban Jr., J.K. Heuer, S. Wu, J.L. Wagner, D.G. Hinks, Large critical current density in
neutron-irradiated polycrystalline HgBa₂CuO₄+δ. Phys. Rev. B. 48 (1993) 9932–9934.

[5] H.T. Ren, K.N.R. Taylor, Y.J. Chen, J.A. Xia, He Qing, Enhanced critical current density in melt-textured (Y₁₋ₓPrₓ)Ba₂Cu₄O₈, Physica C 216 (1993) 447–452.

[6] Kristian Fossheim, Ellen D. Tusset, Thomas W. Ebbesen, Michael M.J. Treacy, Justin Schwartz, Enhanced flux pinning in Bi₂Sr₂CaCu₂O₉+δ superconductor with embedded carbon nanotubes, Physica C 248 (1995) 195–202.

[7] K. Christova, A. Manov, J. Nyhus, U. Thisted, O. Herstad, S.E. Foss, K.N. Haugen, K. Fosheim, Bi₂Sr₂CaCu₂O₉ bulk superconductor with MgO particles embedded, J. Alloys Compd 340 (2002) 1–5.

[8] Z.H. He, T. Habiserether, G. Bruchlos, D. Litzkendorf, W. Gawalek, Investigation of microstructure of textured YBCO with addition of nanopowder SnO₂, Physica 356 (2001) 277–284.

[9] I.F. Lyuksyutov, D.G. Naugle, Frozen flux superconductors, Mod. Phys. Lett. B 13 (1999) 491–508.

[10] S.Y. Yahya, M.H. Jumali, C.H. Lee, R. Abd-Shukor, Effects of γ-Fe₂O₃ on the transport critical current density of (Bi₁₋ₓPbx)₁₋ₓSrₓCa₂Cu₃O₁₀ superconductors, J. Mater. Sci. 39 (2004) 7125–7128.

[11] J. Joo, J.P. Singh, R.B. Poeppel, Effects of thermomechanical treatment on phase development and properties of Ag-sheathed Bi(Pb)-Sr-Ca-Cu-O superconducting tapes, Supercond. Sci. Technol. 6 (1993) 421–428.