Critical currents in silver-sheathed (Bi,Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_{10-y}$ superconducting tapes

Donglu Shi, S. Salem-Sugui, Jr., and Zuning Wang
Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439

L. F. Goodrich
Electromagnetic Technology Division, National Institute of Standards and Technology, Boulder, Colorado 80303-3328

S. X. Dou, H. K. Liu, Y. C. Guo, and C. C. Sorrell
School of Materials Science and Engineering, University of New South Wales, P. O. Box 1, Kensington, NSW 2033, Australia

(Received 2 August 1991; accepted for publication 16 September 1991)

Nearly 95 vol % of the 110 K superconducting phase was formed by lead doping in a Bi–Sr–Ca–Cu–O system. The processed 110 K superconducting powders were used to produce long silver-sheathed tapes with a highly textured microstructure by rolling and prolonged sintering. The transport critical current density was measured at 4.0 K to be 0.7×10$^4$ A/cm$^2$ (the corresponding critical current is 74 A) at zero field and 1.6×10$^4$ A/cm$^2$ at 12 T for $J_c$||ab. At 76 K, the critical current density reached a value of ~1×10$^4$ A/cm$^2$ at zero field for $J_c$||ab and gradually decreased to 419 A/cm$^2$ at 1 T. Excellent grain alignment in the a–b plane led to greatly improved critical current densities under a magnetic field. The relationship between the transport properties and the microstructure of the tapes is discussed.

Large-scale application of high-$T_c$ superconductivity depends on successful production of long wires with high current-carrying capability, superb mechanical flexibility, and chemical stability. In the powder-sintered form, high-$T_c$ tapes are capable of carrying a critical current density of $10^4$ A/cm$^2$ at 77 K and 1 T. However, the critical current densities are all obtained from short pieces of textured material, as indicated in Fig. 1. The sintered material contained mostly the Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_x$ (2223) phase (95 vol %), as indicated in Fig. 1. The sintered material was then powdered and poured into a silver tube of 10 mm outside diameter and 8 mm inside diameter. The silver tube was then rolled into tapes 0.1 mm thick and ~2–3 mm wide. The tapes were then pressed into pellets and sintered at 850 °C for 20 h.

The x-ray diffraction data showed that the sintered material contained mostly the Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_x$ (2223) phase (95 vol %), as indicated in Fig. 1. The sintered pellets were powdered and poured into a silver tube of 10 mm outside diameter and 8 mm inside diameter. The silver tube was then rolled into tapes 0.1 mm thick and ~2–3 mm wide. The tapes were then pressed into pellets and sintered at 850 °C for 20 h.

Large-scale application of high-$T_c$ superconductivity depends on successful production of long wires with high current-carrying capability, superb mechanical flexibility, and chemical stability. In the powder-sintered form, high-$T_c$ tapes are capable of carrying a critical current density of $10^4$ A/cm$^2$ at 77 K and 1 T. However, the critical current densities are all obtained from short pieces of textured material, as indicated in Fig. 1. The sintered material contained mostly the Bi$_2$Sr$_2$Ca$_2$Cu$_3$O$_x$ (2223) phase (95 vol %), as indicated in Fig. 1. The sintered pellets were powdered and poured into a silver tube of 10 mm outside diameter and 8 mm inside diameter. The silver tube was then rolled into tapes 0.1 mm thick and ~2–3 mm wide. The tapes were then pressed into pellets and sintered at 850 °C for 20 h.

In this letter, we report on the transport and inductive $J_c$ data in a magnetic field up to 12 T at 4.0 K and, to 1 T at 76 K for silver-sheathed Bi–Pb–Sr–Ca–Cu–O tapes. We discuss the possible relationship between the critical current and the microstructure of the tapes.

The processing method for making the silver-sheathed Bi–Pb–Sr–Ca–Cu–O tapes has been previously reported by Dou et al. The superconducting powders were made by a freeze-drying technique. The solution of Bi$_2$O$_3$ in nitric acid was mixed with Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$·4H$_2$O, Sr(NO$_3$)$_2$, and Cu(NO$_3$)$_2$·3H$_2$O in distilled water in the ratios Bi:Pb:Sr:Ca:Cu = 1.6:0.4:1.6:2.3. The solutions were then quickly frozen by spraying into a liquid nitrogen bath. The frozen mixtures of the nitrates were placed in a freeze drier and dried under vacuum for 48 h. The dried powders were then pressed into pellets and sintered at 850 °C for 20 h.

In this letter, we report on the transport and inductive $J_c$ data in a magnetic field up to 12 T at 4.0 K and, to 1 T at 76 K for silver-sheathed Bi–Pb–Sr–Ca–Cu–O tapes. We discuss the possible relationship between the critical current and the microstructure of the tapes.

The processing method for making the silver-sheathed Bi–Pb–Sr–Ca–Cu–O tapes has been previously reported by Dou et al. The superconducting powders were made by a freeze-drying technique. The solution of Bi$_2$O$_3$ in nitric acid was mixed with Pb(NO$_3$)$_2$, Ca(NO$_3$)$_2$·4H$_2$O, Sr(NO$_3$)$_2$, and Cu(NO$_3$)$_2$·3H$_2$O in distilled water in the ratios Bi:Pb:Sr:Ca:Cu = 1.6:0.4:1.6:2.3. The solutions were then quickly frozen by spraying into a liquid nitrogen bath. The frozen mixtures of the nitrates were placed in a freeze drier and dried under vacuum for 48 h. The dried powders were then pressed into pellets and sintered at 850 °C for 20 h.
FIG. 1. X-ray diffraction plot for a calcined Bi$_2$Pb$_2$Sr$_2$Ca$_2$Cu$_3$O$_y$ powder showing that 95 vol % of the 110 K superconducting phase was formed in the material.

$J_c$ value remains approximately the same ($> 1 \times 10^4$ A/cm$^2$) as the field reaches 12 T at 4.0 K for both $H_{||ab}$ and $H_{||c}$ [Fig. 2(b)]. However, $J_c(H_{||ab})$ is about 20% higher than $J_c(H_{||c})$ at 12 T and $T = 4.0$ K. It should be pointed out that the total critical current at 4.0 K and zero field has reached a maximum value of 74 A.

Magnetic hysteresis curves were obtained from 4.2 to 60 K up to the applied field of 5 T for $H_{||c}$. By applying a Bean critical state model, we calculated the magnetization $J_c(A/cm^2)$ using the formula $M = a J_c \left(1 - \frac{a_2}{3a_1}\right)/20$, where $M$ is the magnetic hysteresis difference in emu/cm$^3$, and $2a_1 \times 2a_2$ is the cross-sectional area of the sample ($a_1 > a_2$). The $J_c$ versus $H$ data are shown in Fig. 3; the magnetization $J_c$ at 4.2 K is considerably higher than the transport $J_c$. This is associated with the choice of the dimensions of the sample in the Bean model formula for $J_c$ Nevertheless, the magnetization $J_c$ at 4.2 K also exhibits weak field dependence on critical current density, which is consistent with the transport data.

We attribute the high critical current density to the textured microstructure developed by tape rolling and subsequent heat treatment. In Fig. 4 we show the x-ray diffraction plot of the textured sample. As shown in the figure, the material is highly textured, and therefore only the (001) peaks are present in the diffraction pattern [a few non-(001) peaks are low in intensity]. Figure 4 also indicates that the material is relatively free of second phases (the amount of second phase is estimated to be less than 5%).

To confirm the textured microstructure in the silver-sheathed tapes, we performed scanning electron microscopy (SEM) experiments on the cross-sectional areas of the tapes, the results are shown in Fig. 5. For comparison, we also show the SEM photo of the powder-sintered sample [Fig. 5(a)]. As can be seen, the powder-sintered sample has relatively large grains, and the microstructure exhibits randomly oriented grains. In contrast, as can be seen in Fig. 5(b), the Ag-sheathed tape has a highly textured microstructure. Most of the plate-like grains are well oriented along the rolling direction parallel to the surface of the tape.

We found that the texturing in the silver-sheathed tapes could be greatly enhanced by prolonged heat treatment after rolling. As previously observed, the plate-like grains tend to grow much more rapidly along the $a$-$b$ plane than along the $c$ axis. Although some degree of texturing can be obtained after rolling, extended sintering (150 h at 820 °C) is required to further improve the grain alignment for achieving an optimized critical current density.

It has been well reported that flux-creep effects are strong in the bismuth-based system and that the “irreversibility line” lies in the low regions of temperature and field compared to those of the YBa$_2$Cu$_3$O$_y$ compound. For well-textured (Bi,Pb)$_2$Sr$_2$Ca$_2$Cu$_3$O$_y$ tapes, the transport $J_c$ exhibits behavior dominated by flux pinning at both 4.0 and 76 K. The difference in transport $J_c$ between $H_{||c}$ and $H_{||ab}$ (shown in Fig. 2) indicates that the Cu-O planes in the layered structure are responsible for enhanced flux pinning at 4.0 and 76 K.
In conclusion, we have successfully processed a Bi-Pb-Sr-Ca-Cu-O compound and obtained bulk samples with a majority of the 110 K phase by using freeze-dried powders. With the powder-in-tube technique, we have produced silver-sheathed superconducting tapes with a highly textured microstructure. The processed tapes possess high flexibility after sintering and can carry high critical current density under high applied magnetic field at 4.2 K. Our experimental data indicate that the silver-sheathed superconducting tapes show promise for practical applications.

We are grateful to T. C. Stauffer for assistance with the measurements and instrumentation. This work is supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under Contract No. W-31-109-ENG-38 (D. S., S. S. S.). L. F. G. acknowledges the National Institute of Standards and Technology high-\(T_c\) program for supporting this research. Support from the Australian Metal Manufacture Ltd. (S. X. D.), and from the Australian Commonwealth Department of Industry, Technology and Commerce (H. K. L. and Y. C. G.) is gratefully acknowledged. S. S. S. acknowledges his fellowship from Fundação de Amparo a Pesquisa do Estado de São Paulo, FAPESP, Brazil.

\[ J_c(H\|c) \text{ and } J_c(H\|ab) \text{ has been shown to be associated with the degree of grain alignment.} \]

In conclusion, we have successfully processed a Bi-Pb-Sr-Ca-Cu-O compound and obtained bulk samples with a majority of the 110 K phase by using freeze-dried powders. With the powder-in-tube technique, we have produced silver-sheathed superconducting tapes with a highly textured microstructure. The processed tapes possess high flexibility after sintering and can carry high critical current density under high applied magnetic field at 4.2 K. Our experimental data indicate that the silver-sheathed superconducting tapes show promise for practical applications.

We are grateful to T. C. Stauffer for assistance with the measurements and instrumentation. This work is supported by the U.S. Department of Energy, Basic Energy Sciences-Materials Sciences, under Contract No. W-31-109-ENG-38 (D. S., S. S. S.). L. F. G. acknowledges the National Institute of Standards and Technology high-\(T_c\) program for supporting this research. Support from the Australian Metal Manufacture Ltd. (S. X. D.), and from the Australian Commonwealth Department of Industry, Technology and Commerce (H. K. L. and Y. C. G.) is gratefully acknowledged. S. S. S. acknowledges his fellowship from Fundação de Amparo a Pesquisa do Estado de São Paulo, FAPESP, Brazil.


