Critical currents in silver-sheathed $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10-y}$ superconducting tapes

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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 0.7×10^5 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 $H\|ab$. At 76 K, the critical current density reached a value of $\sim1\times10^4$ A/cm^2 at zero field for $H\|ab$ 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 superconductors can carry only extremely low transport critical current density J_c especially in a low magnetic field ($\sim$10 mT).[1–3] The materials are also very brittle and the measured mechanical strength is low.[4]

To improve these properties, researchers have concentrated on increasing the critical current densities and enhancing the mechanical strength of high-T_c superconductors. Various methods have been developed to produce highly textured microstructures suitable for carrying high currents. Zone melting and melt texturing are the two most successful techniques used to process $YBa_2Cu_3O_x$ samples which possess a good grain-oriented microstructure and are capable of carrying a critical current density of 10^4 A/cm^2 at 77 K and 1 T.[5–7] However, the critical current densities are all obtained from short pieces of textured samples (10 to 20 mm long).

The metal sheathed powder-in-tube technique has proved successful for making long high-T_c superconductor wires that can carry high critical current densities.[8–17] Specifically, Bi-based superconducting tapes have been developed that can carry a critical current densities greater than 1×10^4 A/cm^2 at high magnetic field (>20 T) at 4.2 K.[8–17]

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.*[8] The superconducting powders were made by a freeze-drying technique.[8] The solution of Bi_2O_3 in nitric acid was mixed with $Pb(NO_3)_2$, $Ca(NO_3)_2\cdot4H_2O$, $Sr(NO_3)_2$, and $Cu(NO_3)_2\cdot3H_2O$ 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 calcined in air at 830 °C for 10 h. The calcined powders 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 $Bi_2Sr_2Ca_2Cu_3O_x$ (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 $\sim$2–3 mm wide. The tapes were heat treated at 820 °C for 150 h in a mixture of oxygen and nitrogen with varying O_2 partial pressure. The heat treatment was repeated twice to optimize grain alignment.

The transport critical current density J_c of the silver-sheathed tapes was measured at 4.0 K up to 12 T and 76 K up to 1 T. The measurements were performed using a standard four-probe method with a voltage criterion of 1 μV/cm. The direction of the transport current was perpendicular to the applied field. For comparison, we also measured magnetization critical current density in a wide temperature regime using a vibrating sample magnetometer.

As shown in Fig. 2(a), the $J_c(H\|ab)$ reaches a value of $\sim1\times10^4$ A/cm^2 at 76 K and gradually decreases to $\sim$400 A/cm^2 at 1 T. For $H\|c$, strong field dependence of the J_c has been observed. J_c is significantly reduced to less than 10 A/cm^2 at 1 T [see Fig. 2(a)]. In agreement with most of the previously reported transport data, the high

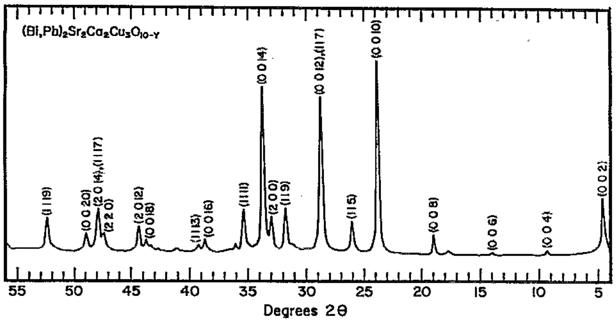

FIG. 1. X-ray diffraction plot for a calcined $Bi_{1.6}Pb_{0.4}Sr_{1.6}Ca_2Cu_3O_x$ powder showing that 95 vol % of the 110 K superconducting phase was formed in the material.

J_c value remains approximately the same ($>1\times10^4$ 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[18] $\Delta M = a_2 J_c(1 - a_2/3a_1)/20$, where ΔM is the magnetic hysteresis difference in emu/cm^3, and $2a_1\times2a_2$ 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.[8]

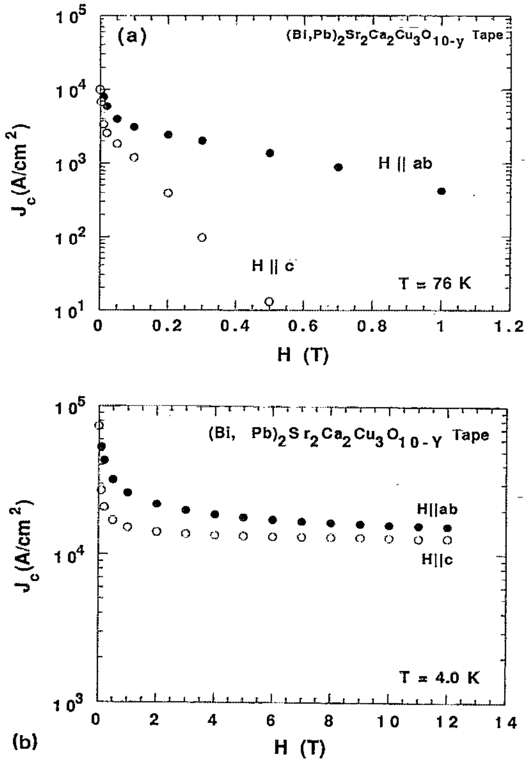

FIG. 2. (a) Transport J_c vs H at 76 K, and (b) J_c vs H at 4.0 K.

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 $YBa_2Cu_3O_x$ compound. For well-textured $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10-y}$ 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 pin-

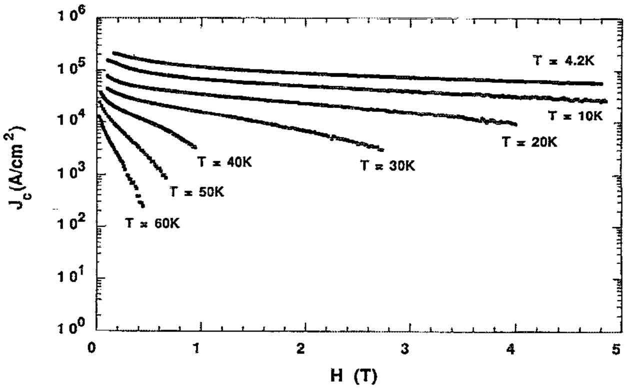

FIG. 3. Magnetization J_c vs H at various temperatures indicated.

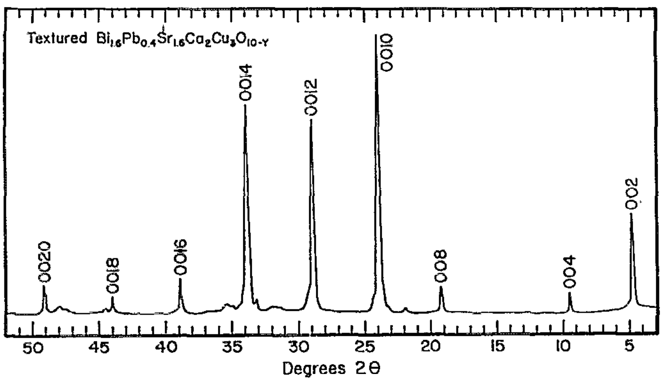

FIG. 4. X-ray diffraction plot showing highly *c* axis-oriented superconducting grains after rolling and prolonged sintering.

ning. In particular, this difference becomes pronounced at higher temperature (76 K). At 4.0 K, the difference in the J_c for two configurations is almost the constant between 2 and 12 T, indicating that the increasing rates of the pinning force with the increasing field are the same. However, J_c drops more rapidly for $H \| c$ at 76 K. This fact may suggest that pinning by Cu–O planes is not important at low temperatures where intrinsic pinning by oxygen vacancies dominates.[19] However, as a result of high flux-creep rates at liquid nitrogen temperature, the Cu–O planes can act as major pinning centers. It should be noted that the anisotropy between $J_c(H\|c)$ and $J_c(H\|ab)$ has been shown to be associated with the degree of grain alignment.[20]

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.

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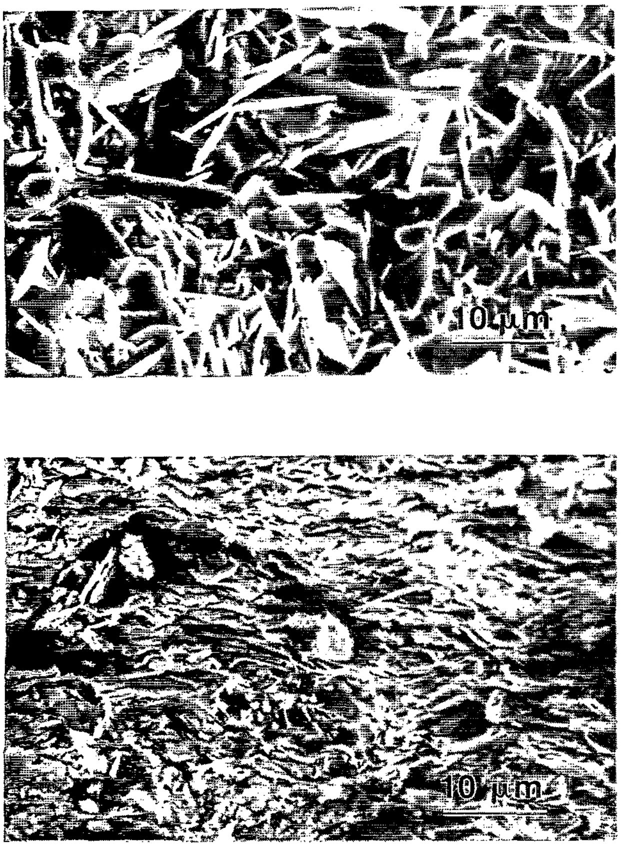

FIG. 5. Scanning electron microscopy photographs showing (a) randomly oriented grains in the powder-sintered sample, and (b) highly *c* axis-oriented grains in the rolled and sintered tapes.

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