

CRITICAL CURRENT DENSITY, IRREVERSIBILITY LINE, AND FLUX CREEP
ACTIVATION ENERGY IN SILVER-SHEATHED $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$
SUPERCONDUCTING TAPES*

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Abstract--Transport data, magnetic hysteresis and flux creep activation energy experimental results are presented for silver-sheathed high- T_c $\text{Bi}_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_x$ superconducting tapes. The 110 K superconducting phase was formed by lead doping in a Bi-Sr-Ca-Cu-O system. The transport critical current density was measured at 4.0 K to be $0.7 \times 10^5 \text{ A/cm}^2$ (the corresponding critical current is 74 A) at zero field and $1.6 \times 10^4 \text{ A/cm}^2$ at 12 T for Hllab. Excellent grain alignment in the a-b plane was achieved by a short-melting method, which considerably improved the critical current density and irreversibility line. Flux creep activation energy as a function of current is obtained based on the magnetic relaxation measurements.

I. INTRODUCTION

Large-scale application of high- T_c superconductivity depends on the successful production of long wires with high current-carrying capability, superb mechanical flexibility, and chemical stability. 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 [1-4]. Specifically, Bi-based superconducting tapes have been developed that can carry critical current densities greater than $1 \times 10^5 \text{ A/cm}^2$ at high magnetic field ($> 20 \text{ T}$) at 4.2 K [5]. In this paper, we report on the critical current density and irreversibility data for silver-sheathed Bi-Pb-Sr-Ca-Cu-O tapes processed by a novel method. We discuss the possible relationship between the critical current and the microstructure of the tapes.

II. EXPERIMENTAL PROCEDURE

The processing method for making the silver-sheathed Bi-Pb-Sr-Ca-Cu-O tapes was previously reported by Dou et al. [1]. The superconducting powders were made by a freeze-drying technique [1]. The solution of Bi_2O_3 in nitric acid was mixed with $\text{Pb}(\text{NO}_3)_2$, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Sr}(\text{NO}_3)_2$, and $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ 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 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 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 $\sim 2\text{-}3 \text{ mm}$ wide. Two types of heat treatment were used in this study. Sample 1 was heat treated at $\sim 820^\circ\text{C}$ for 150 h in a mixture of oxygen and nitrogen with varying O_2 partial pressure; the heat treatment was repeated twice to optimize the grain alignment. Sample 2 was heat treated at $\sim 830^\circ\text{C}$ for 70 h in a similar atmosphere; in addition, it underwent a partial melting at 860°C for 20-30 min. The detailed processing procedure for partial melting of Sample 2 can be found elsewhere [2].

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 by using a standard four-probe method with a voltage criterion of $1 \mu\text{V/cm}$. 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. By applying a Bean critical state model, we calculated the magnetization $J_c(\text{A/cm}^2)$ using the formula $\Delta M = a_2 J_c (1 - a_2/3a_1)/20$, where ΔM is the magnetic hysteresis difference in emu/cm^3 , and $2a_1 \times 2a_2$ is the cross-sectional area of the sample ($a_1 > a_2$).

III. RESULTS AND DISCUSSION

In agreement with most of the previously reported transport data, the high J_c value of Sample 1 remained approximately the same ($> 1 \times 10^4 \text{ A/cm}^2$) as the field reached 12 T at 4.0 K for both Hllab and Hllc (Fig. 1). However, the J_c (Hllab) was about 20% higher than the J_c (Hllc) at 12 T and $T = 4.0 \text{ K}$. It should be pointed out that the total critical current at 4.0 K and zero field reached a maximum value of 74 A [3].

We found that the texturing in the silver-sheathed tapes was greatly enhanced by the short-melting process. As can be seen in Fig. 2, Sample 2 has a higher degree of texture compared with the previously obtained microstructure [3].

Although some degree of texturing can be obtained by extended sintering (150 h at 820 °C), the short-melting process at partial melting temperature is required to further improve the grain alignment for achieving an optimized critical current density.

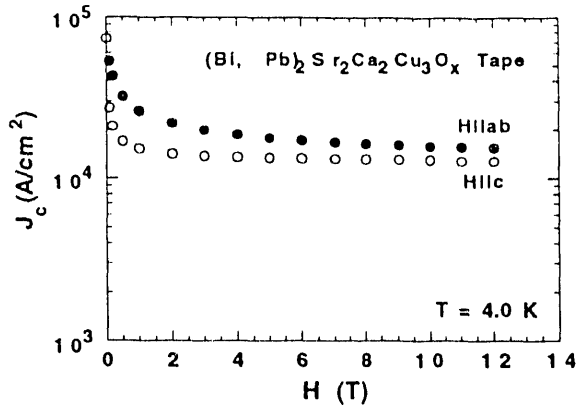


Fig. 1. Transport J_C vs. H at at 4.2 K for sample 1.

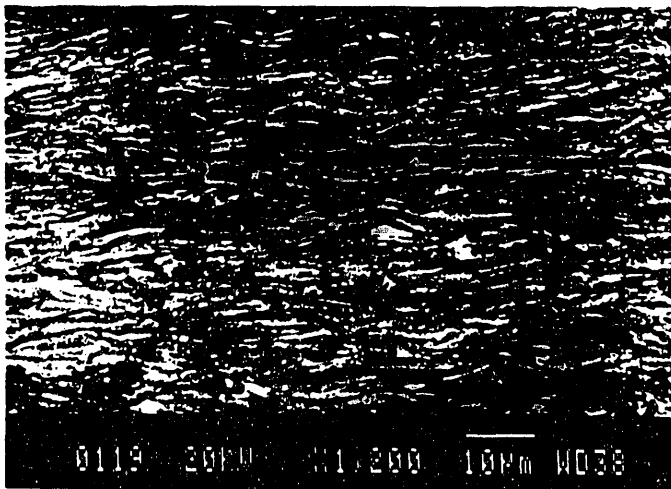


Fig. 2. Scanning electron microscopy photograph showing highly c axis-oriented grains in a tape processed by short-melting method.

The magnetic hysteresis curves were obtained from 4.2 K to 90 K up to the applied field of 5 T for Hllc. Fig 3. Shows the temperature dependence of the J_C at 0.5 T for Samples 1 and 2. As can be seen, the J_C of Sample 2 is considerably increased with increasing temperature, particularly in the high temperature range. However, in the temperature range below 50 K, the J_C values of both samples are comparable.

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 with those of the $YBa_2Cu_3O_x$ compound.

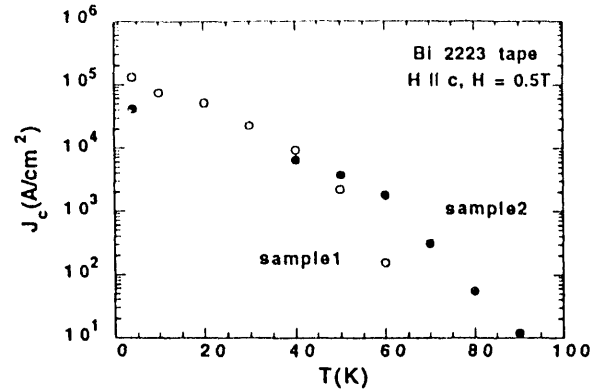


Fig. 3. Magnetization J_C vs. temperature, T , at 0.5 T for sample 1 and 2.

Fig. 4 shows the irreversibility lines of Samples 1 and 2. A similar effect to the temperature dependence of J_C shown in Fig. 3 is observed. It has been found that partial melting generates a high density of dislocations in the superconducting phase and that these dislocations can act as pinning centers [2]. Moreover, as is true of also of J_C , the shift of the irreversibility line is more pronounced at high temperatures. Below 10 K and at high fields, the irreversibility lines of Samples 1 and 2 are located in the nearby regions.

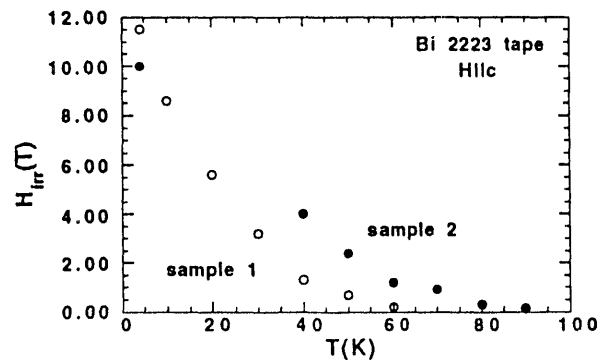


Fig. 4. Irreversibility lines determined based on the magnetic hysteresis data for samples 1 and 2.

It is yet not clear why the J_C and irreversibility line are more pronounced at high temperatures. A possible interpretation is that the flux pinning increases with temperature as a result of the short-melting method, although the pinning mechanism is still to be identified. On the other hand, the difference in anisotropy of J_C at 4.2 K and 77 K suggests that pinning by Cu-O planes is not important at low temperatures, where the superconducting system is more uniform. As observed in Ref. 3 and shown in Fig. 1, the J_C is not much different for both Hllc and Hllab, while a large difference in J_C is observed at 77 K for these configurations.

As a result of high flux-creep rates at liquid nitrogen temperature, the Cu-O planes can act as major pinning centers. The enhanced pinning may also be associated with better texturing in Sample 2.

We consider here that U is a function of J , which is temperature and field dependent [6]. We expand the effective activation energy $U(J)$ about some current J_0 at time t_0 to obtain

$$U(J) = U(J_0) + [\partial U/\partial J]_0(J - J_0) + (1/2)[\partial^2 U/\partial J^2]_0(J - J_0)^2 + \dots \\ \sim U(J_0) + \alpha(J - J_0) + (1/2)\beta(J - J_0)^2 + \dots \quad (1)$$

Considering the second-order term and assuming that the preexponential factor A is constant in the temperature range considered, one obtains that

$$J(t) = J_0 + (kT/c) \ln(t/t_0) - (k^2 T^2 \beta / 2\alpha^3) \ln^2(t/t_0). \quad (2)$$

Using Bean's model¹², one can rewrite above equation as

$$M(t) = M_0 + a \ln(t/t_0) + b \ln^2(t/t_0), \quad (3)$$

where $a = (kT)[\partial M/\partial U]_0$ and $b = -(k^2 T^2 / 2)[\partial^2 U/\partial M^2]_0 [\partial M/\partial U]_0^3$. Experimentally, one can determine the constants a and b from the magnetic relaxation measurements and use them to calculate α and β . Substituting α and β into Equation (1), a smooth U - j curve can be obtained. Fig. 5 shows the U - j relationship for Sample 1. As can be seen, at large driving force, U varies gradually while a rapid increase is observed as j is reduced to a small level. We found that this behavior is typical for most of the type-II superconductors including A-15 Nb₃Sn.

In conclusion, we have found that the J_c and irreversibility line are considerably enhanced in silver-sheathed Bi-based superconducting tapes by employing a short-melting method developed previously. The flux-pinning strength has been found to increase with temperature, resulting in more pronounced enhancement of J_c in the partial-melted sample tape at high temperatures. The pinning mechanism associated with enhancement of the J_c and irreversibility line in Sample 2 is under investigation. We have obtained flux-creep activation energy of the silver-sheathed tape based on magnetic relaxation data.

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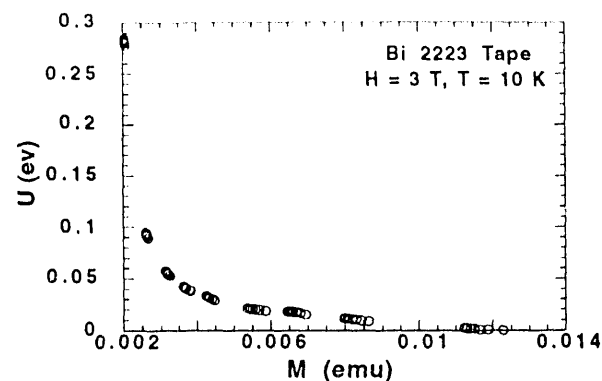


Fig. 5. U vs. J curve for Sample 1.

REFERENCES

- [1] S.X. Dou, H.K. Liu M.H. Apperley, K.H. Song, and C.C. Sorrell, "Critical current density in superconducting Bi-Sr-Ca-Cu-O wires and coils", *Supercon. Sci. Technol.*, Vol. 3, pp. 138-142, November 1990.
- [2] S.X. Dou, H.K. Liu and Y.C. Guo, "Improvement of flux pinning in the Ag-clad Bi-Sr-Ca-Cu-O wires through the use of a short period melting process," *Appl. Phys. Lett.*, Vol. 60, pp. 2929-2931, June 1992.
- [3] D. Shi, S. Salem-Sugui, Jr., Z. Wang, L.F. Goodrich, S.X. Dou, H.K. Liu, Y.C. Guo, and C.C. Sorrell, "Critical currents in silver-sheathed (Bi,Pd)₂Sr₂Ca₂Cu₃O_x superconducting tapes", *Appl. Phys. Lett.*, Vol. 59, pp. 3171-3173, Dec. 1991.
- [4] P. Halder, J.G. Hoehn, Jr., J.A. Rice, and M.S. Walker, "Transport critical current densities of silver clad Bi-Pb-Sr-Ca-Cu-O tapes at liquid helium and hydrogen temperatures," *Appl. Phys. Lett.*, Vol. 61, pp. 604-606, August 1992.
- [5] K. Heine, J. Tenbrink, and M. Thoner, "High field critical current densities in (Bi₂Sr₂Ca₂Cu₃O_{8-x})," *Appl. Phys. Lett.*, Vol. 55, pp. 2441-2443, December 1989.
- [6] S. Sengupta, D. Shi, S. Salem-Sugui, Jr., Z. Wang, P. J. McGinn, and K. DeMoranville, "Current density dependence of the activation energy in type II superconductors," *J. Appl. Phys.*, Vol. 72, pp.592-595, 1992.

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