

FABRICATION OF Yb-123 TAPES*

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Fabrication of Yb-123 Tapes

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Abstract—While Bi-2223 tapes have been the workhorses of the superconductor industry, their poor performance in applied magnetic fields restrict their use to below 30 K. Melt-processing of Ag-clad Yb-123 PIT tapes offers a simple and scalable technique for fabricating long-length HTS conductors capable of being used at 77 K. Under reduced oxygen partial pressure, the peritectic temperature of Yb-123 is below the melting point of Ag, and this facilitates the adaptation of melt-texturing methods for fabricating these tapes. The effect of melt-processing temperature on current density was also explored; a temperature of 965°C yielded optimal critical current values. The critical current density achieved at 4.2 K was 20,000 A/cm², corresponding to a critical current of 52 A. Based on the above results, an optimal processing zone for melt-processing of Ag-clad Yb-123 tapes was determined. These results hold promise for melt-processing of Ag-clad Yb-123 tapes as an alternative to Bi-2223 PIT technology.

Index Terms—high growth rates, melt-texturing, powder-in-tube, Yb-123.

I. INTRODUCTION

TO realize the potential of high-temperature superconductors (HTS) for replacing copper in current-carrying cables, it is imperative that cost-effective processing techniques be developed to manufacture long-length cables that do not compromise the performance of the HTS conductors. While Ag-clad Bi-2223 tapes made by the powder-in-tube (PIT) technique have been the workhorses of the HTS industry so far, their poor flux-pinning properties limit their critical current density (J_c) in the presence of an applied magnetic field [1,2]. Consequently, second-generation wire technology development has focused on YBa₂Cu₃O_x (Y-123) coated conductors through novel approaches such as IBAD and RABiTS [3,4]. These techniques offer enhanced performance compared to first-generation conductors, but require cost-intensive processing steps that cannot be offset by the superior properties of the conductors. Melt-texturing has been shown to be a successful processing method for achieving high J_c values in RBa₂Cu₃O_x (R-123) compounds for applications such as trapped-field magnets and levitators [5]. However, the melt-texturing technique has not been adapted to conductor fabrication due

to the nature of its processing conditions. The high temperatures associated with the heat treatment, and the presence of a high volume fraction of liquid phase at high temperatures, necessitate the use of a flexible metallic substrate or cladding material. The only economical material benign to R-123 is Ag, but its use is curtailed in melt-processing because its melting point is lower than the heat treatment temperatures for most R-123 systems. Among the R-123 systems, YBa₂Cu₃O_x (Yb-123) has the lowest peritectic decomposition temperature, and hence the lowest expected processing temperature [6]. In order to use Ag as cladding material in melt-processing, one is therefore limited to Yb-123 as the choice of HTS material.

Very little work has been done on melt-processing of Yb-123 thus far, primarily because of the apparent difficulty in forming the Yb-123 phase [7]. Recently, Athur et al. [8] reported synthesizing phase-pure Yb-123 powder by a simple solid-state sintering technique. This paper reports on the fabrication of Ag-clad Yb-123 tapes by the PIT process, and the effects of melt-processing variables such as growth rate, processing temperature, and atmosphere on the electrical properties of the tapes.

II. EXPERIMENT

Fabrication of the tapes required four steps: powder synthesis, tape fabrication, melt-processing, and annealing.

A. Powder Synthesis

The powder was synthesized through solid-state sintering by using precursor Yb₂O₃, CuO, and BaCO₃ powders. The sintering atmosphere was a flowing gas mixture of Ar + 1% O₂, and the sintering temperature was 825°C. Details of the synthesis route are provided in Ref. 8.

B. Tape Fabrication

A silver tube measuring 6.35 mm OD x 4.35 mm ID x 52.5 mm length was crimped at one end, filled with Yb-123 powder, hand-tapped to a packing density of ≈35%, and closed. The tube was then subjected to a mechanical deformation step that consisted of two stages, groove-rolling during which the billet was reduced in cross-sectional area, and a final flat-rolling that yielded the tape geometry. After the groove-rolling, the cross-sectional dimensions were 2 x 2 mm, and after the flat-rolling, the tape had cross-sectional dimensions of ≈3 mm x 250 μm. Small pieces were cut from the as-rolled tape and weighed, and the core's cross-sectional area was measured to determine the packing efficiency of the powder.

C. Melt-Processing

The melt-processing step was performed in a high-gradient zone-melting furnace. Four-cm-long pieces of the as-rolled

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tape were cut, mounted on an alumina rod, inserted into the preheated furnace, held there for 30 min to bring the sample to thermal equilibrium, and then moved through the furnace at a controlled rate. The furnace gradient was determined to be 130-150°C/cm. Process variables such as oxygen partial pressure, pO_2 , processing temperature, T_z , and growth rate, v , were varied, and their effects were correlated to the electrical properties of the tape.

D. Annealing

The melt-processed tapes were subjected to an oxygen annealing to convert the tetragonal phase to the superconducting orthorhombic phase.

E. Characterization

Resistive critical temperature, T_c , and transport critical current, I_c , were measured by the four-point method. Transport I_c was measured at various temperatures from 77 K down to 4.2 K. AC susceptibility measurements were used to determine T_c . Electron probe microanalysis (EPMA) was used to study the phase assemblage of the melt-processed superconducting core, and SEM was used to examine the microstructure of the superconductors.

III. RESULTS AND DISCUSSION

A. Effect of pO_2

Three melt-processing atmospheres were studied, with pO_2 values of 0.21, 0.01, and 0.001 atm, respectively.

1. T_c Measurements

There was little change in the T_c when the pO_2 was varied. Fig. 1 shows magnetization behavior for a sample processed in 0.01 atm, while Fig. 2 shows a resistive plot for a sample processed in 0.001 atm. In both cases, $T_{c,onset}$ was ≈ 82 K. However, the broad transition in the magnetization curve indicates inhomogeneity in the sample due to the presence of regions with differing T_c values. On the other hand, in 0.001 atm, the transition is fairly sharp (within 2 K), showing a well-connected percolative path.

2. J_c Measurements

While the J_c values in the first two cases remained fairly low, significant increase in J_c was observed at a pO_2 of 0.001 atm. Fig. 3 shows a V-I plot for tapes that were melt-processed at 0.21 and 0.01 atm. Both samples carried very little current at 77 and 65 K. However, when the melt-processing pO_2 was reduced to 0.001 atm, the J_c at 65 K increased significantly (Fig. 4). At 4.2 K, sample A had an I_c of 52 A, which was equivalent to a J_c value of 20,000 A/cm².

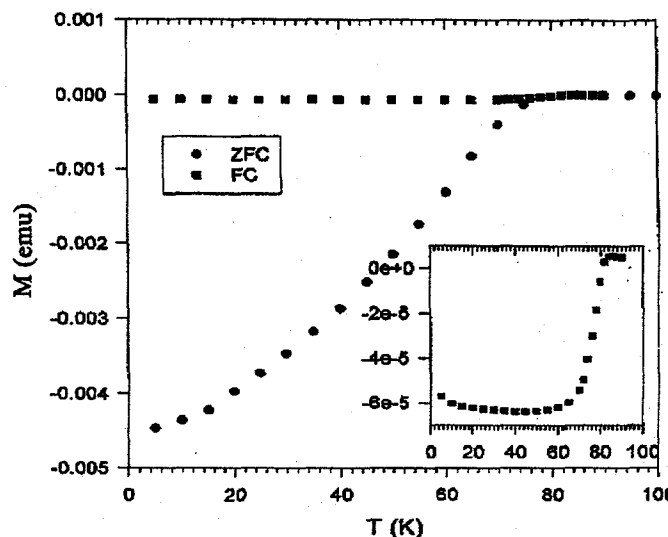


Fig. 1. AC susceptibility measurements ($pO_2 = 0.01$ atm).

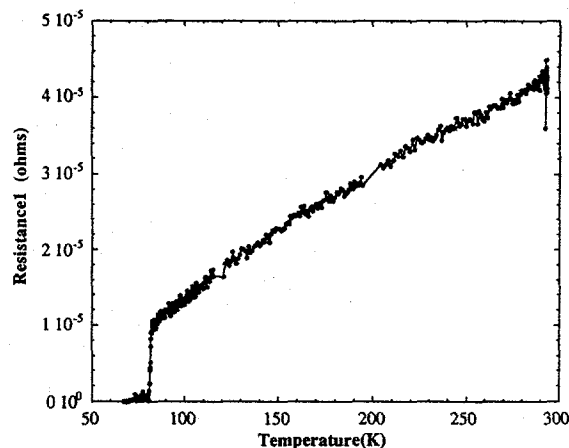


Fig. 2. Resistive plot ($pO_2 = 0.001$ atm).

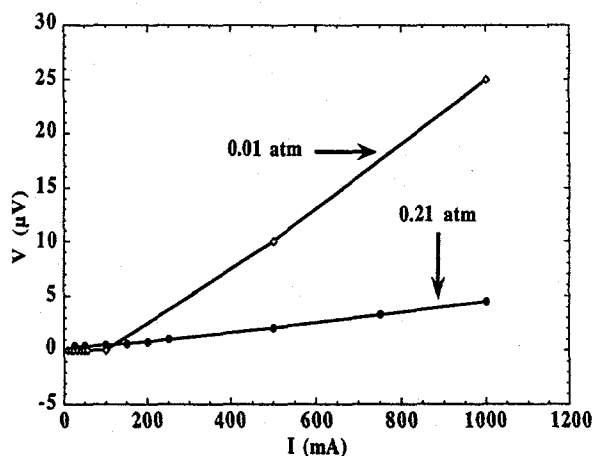


Fig. 3. V-I plot at 65 K ($pO_2 = 0.21$ and 0.01 atm).

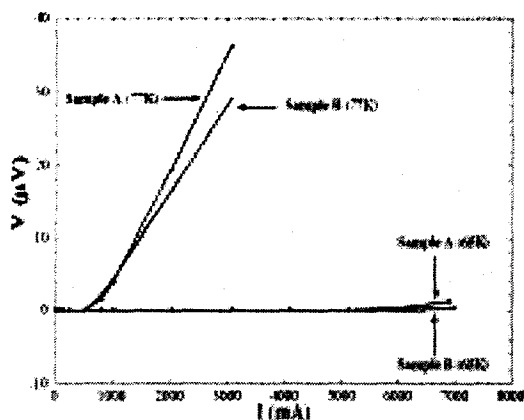


Fig. 4. V-I plot at 77 and 65 K ($p\text{O}_2 = 0.001$ atm).

3. SEM/EPMA

EPMA revealed the oxide core to be multiphasic, containing (apart from Yb-123) Yb-211, BaCuO_2 , and CuO (Fig. 5). Significant fractions of voids, pores, and cracks were also observed. Wave length dispersive spectroscopic analysis (WDS) shows the Yb-123 to be nonstoichiometric, with a slight Ba deficiency and some Yb and Cu excess.

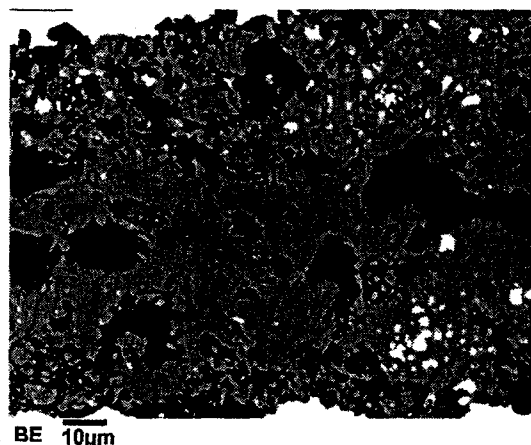


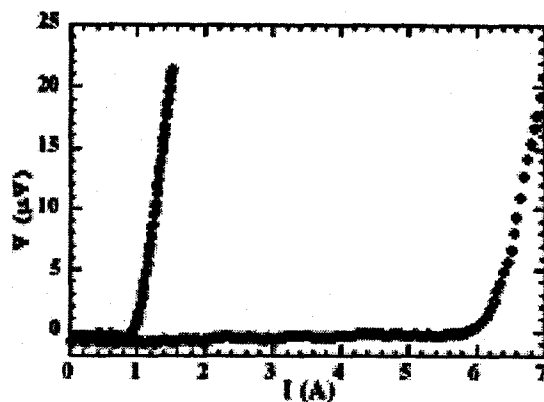
Fig. 5. Backscattered electron image (BEI) of tape processed in 0.001 atm. Gray regions: Yb-123; White: Yb-211; Black: CuO or pores.

B. Effect of T_z

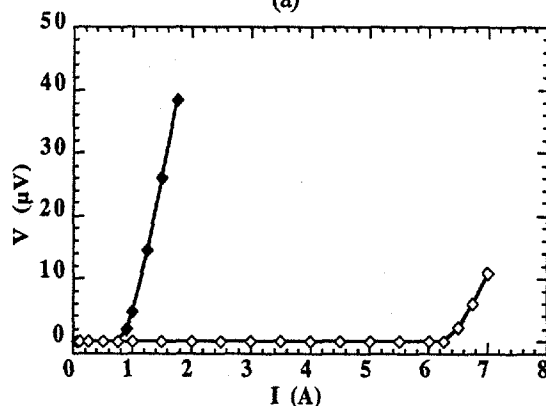
1. J_c Measurements

For a $p\text{O}_2$ of 0.001 atm and v of 1 mm/h during melt-processing, the I_c was optimized for different T_z values. The optimal T_z was found to be 965°C . Figs. 6a-e show V-I plots for T_z values of 920, 935, 950, 965, and 980°C . As can be seen from the figures, there is not much variation in I_c , although the highest I_c value was measured at 965°C to be 7 A, corresponding to a J_c of 3500 A/cm^2 at 65 K.

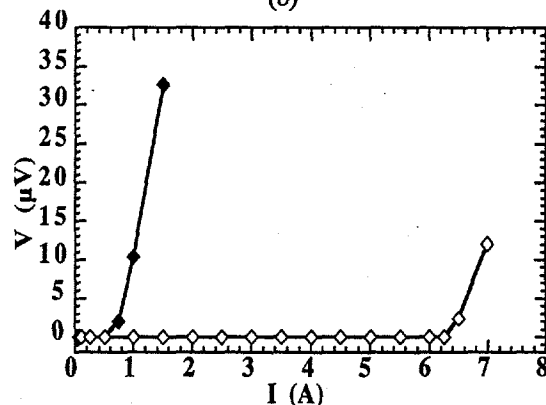
The tape melt-processed at a T_z of 965°C (referred to as tape A) was also characterized for I_c at 4.2 K and was found to carry an I_c of 52 A, corresponding to a J_c of $20,000 \text{ A/cm}^2$. Fig. 7 shows a V-I plot for the measurement at 4.2 K. J_c measurements at intermediate temperatures between 77 and 65 K were also done, and Fig. 8 shows a plot of J_c versus temperature, T , for the tape processed at 965°C , 1 mm/h, and $p\text{O}_2$ of 0.001 atm.



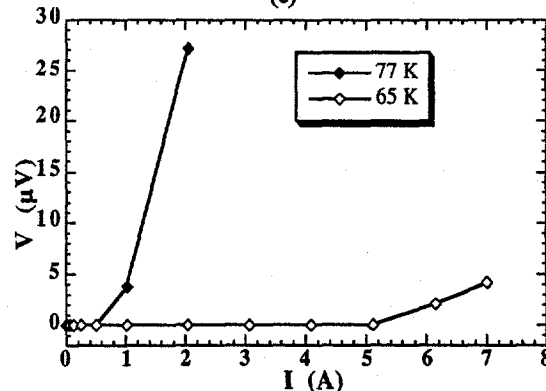
(a)



(b)



(c)



(d)

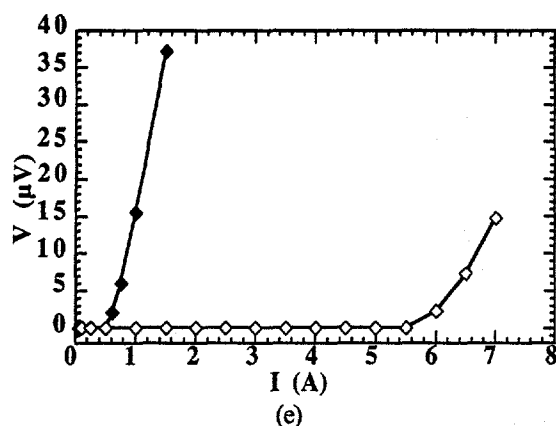


Fig. 6. V-I plots at 77 and 65 K for tapes melt-processed at T_z of (a) 920, (b) 935, (c) 950, (d) 965, and (e) 980°C, respectively.

The variation of J_c with T shows a dependence similar to the variation of J_c with magnetic field for weak-linked material, indicating the possible presence of similar weak links in the tape. It is therefore likely that textured domains in the sample are fairly small, with a large fraction of nonsuperconducting secondary phases present in the microstructure. This was confirmed by the photomicrograph in Fig. 5. Thus, the J_c enhancement upon reducing the pO_2 during melt-processing to 0.001 atm is probably due to densification and liquid-phase-assisted sintering.

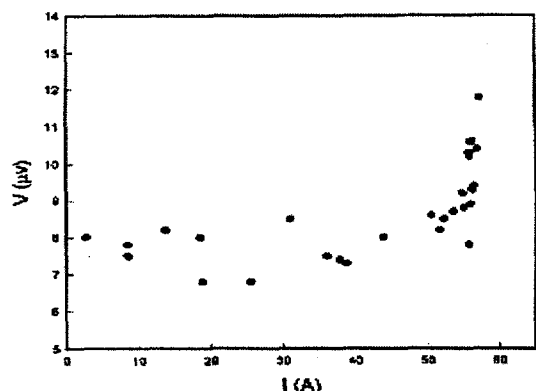


Fig. 7. Pulse V-I plot at 4.2 K for tape A melt-processed in pO_2 of 0.001 atm at T_z of 965°C and v of 1 mm/h.

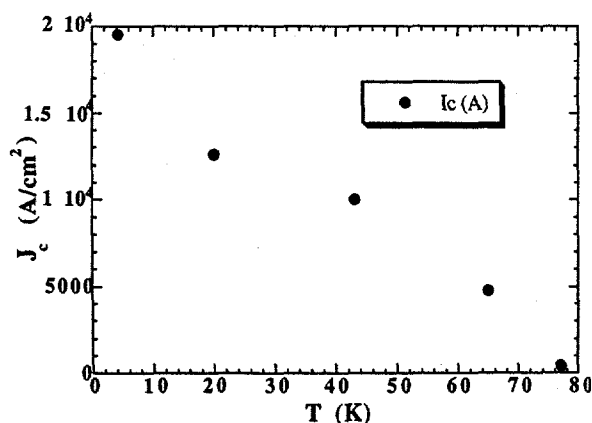


Fig. 8. J_c versus T plot for tape A.

IV. CONCLUSIONS

An alternative processing route for fabricating long-length Ag-clad Yb-123 conductors was explored by amalgamating the PIT process with the melt-texturing technique. It was found that pO_2 played a crucial role in the Yb-23 powder synthesis and in the melt-processing step. J_c values of $\approx 3,500$ A/cm² at 65 K were achieved in these tapes, and T_c values of ≈ 82 K were measured. The highest I_c achieved at 65 K was 11 A; at 4.2 K, it was 52 A. SEM imaging of the tape cross sections showed the presence of secondary phases such as Yb-211, BaCuO₂, and CuO, as well as voids and porosities. Large melt-textured domains typical of other R-123 compounds such as Y-123 and Nd-123 were not present, indicating lack of optimization of texturing process. Optimization of processing conditions to increase the textured volume in the superconducting core can lead to significant enhancement of J_c , lending promise to the development of this technique as a viable and economical alternative to first- and second-generation conductor technology.

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REFERENCES

- [1] K. Sato, N. Shibuta, H. Mukai, T. Hikita, M. Ueyama, and T. Kato, "Development of silver-sheathed bismuth superconducting wires and their application," *J. Appl. Phys.* vol. 70, pp. 6484-6488, 1991.
- [2] R. Flukiger, T. Graf, M. Decroux, C. Groth, and Y. Yamada, "Critical currents in silver-sheathed tapes of the 2223 phase in (Bi,Pb)-Sr-Ca-Cu-O," *IEEE Transactions on Magnetism*, vol. 27, pp. 1258-1263, 1991.
- [3] X.D. Wu, S.R. Foltyn, P. Arendt, J. Townsend, and C. Adams, "High current YBa₂Cu₃O_{7-x} thick films on flexible nickel substrates with textured buffer layers," *Applied Physics Letters*, vol. 65, pp. 961-963, 1994.
- [4] A. Goyal, D.P. Norton, J.D. Budai, M. Paranthaman, E.D. Specht, D.M. Kroeger, D.K. Christen, Q. He, B. Saffian, F.A. List, D.F. Lee, P.M. Martin, C.E. Clabunde, E. Hartfield, and V.K. Sikka, "High critical current density superconducting tapes by epitaxial deposition of YBa₂Cu₃O_x thick films on biaxially textured metals," *Applied Physics Letters*, vol. 69, pp. 1795-1797, 1996.
- [5] K. Salama, V. Selvamanickam, L. Gao, and K. Sun, "High current density in bulk YBa₂Cu₃O_x superconductor," *Applied Physics Letters*, vol. 54, pp. 2352-2354, 1989.
- [6] T. Mochida, M. Takahashi, S. Goshima, N. Sakai, S.I. Yoo, and M. Murakami, "Preparation of YbBa₂Cu₃O_{7-x} bulk superconductor by melt growth process," *Superlattices and Microstructures*, vol. 21, supplement A, pp. 37-39, 1997.
- [7] V. Badri and U.V. Varadaraju, "Structure and superconductivity studies on LnBa_{2-x}Sr_xCu₃O₇ (Ln = Yb and Lu, 0.0 ≤ x ≤ 0.5)," *Materials Research Bulletin*, vol. 27, pp. 591-602, 1992.
- [8] S.P. Athur, P. Putman, U. Balachandran, and K. Salama, "Phase formation and melt processing of Yb-123," *Journal of Superconductivity*, vol. 11, pp. 525-531, 1998.