

FABRICATION AND CHARACTERIZATION OF Ag-CLAD Bi-2223 TAPES*

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ABSTRACT

The powder-in-tube (PIT) technique was used to fabricate multifilament $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ (Bi-2223) superconducting tapes. Transport current properties of these tapes were enhanced by increasing the packing density of the precursor powder and improving the mechanical deformation condition. A critical current (I_c) of > 35 A in long lengths (> 200 m) tapes has been achieved. In measuring the dependence of critical current density on magnetic field and temperature for the optimally processed tapes, we found a J_c of $> 10^4$ A/cm² at 20 K in magnetic fields up to 3 T and parallel to the c-axis, which is of interest for use in refrigerator-cooled magnets. I_c declined exponentially when an external field was applied perpendicular to the tape surface at 77 K. Mechanical stability was tested for tapes sheathed with pure Ag and Ag-Mg alloy. Tapes made with pure Ag sheathing can withstand a tensile stress of ≈ 20 MPa with no detrimental effect on I_c values. Mechanical performance was improved by using Ag-Mg alloy sheathing: values of transport critical current began to decrease at the tensile stress of ≈ 100 MPa. Transport current measurements on tapes wound on a mandrel of 3.81 cm (1.5 in.) diameter at 30° to the longitudinal axis, showed a reduction of $\approx 10\%$ in I_c values for pure Ag-sheathed tapes and 5% reduction in I_c values for Ag-Mg sheathed tapes, compared with the I_c values of as-coiled tapes.

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INTRODUCTION

High critical current density (J_c) in superconducting wires and tapes is essential for many practical applications. Material processing remains the key to realizing the application potential of high-temperature superconductors. The powder-in-tube (PIT) process, which yields a highly textured $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ (Bi-2223) superconductor with its c-axis aligned parallel to the tape surface, is an industrially scalable technique for fabricating long-length superconductors [1-3]. Significant progress has been made over the past several years in improving J_c values in wires and tapes to acceptable levels for certain commercial applications. Two major issues are the focus of the ongoing research: transport current performance in applied magnetic field, and mechanical properties.

Recent progress in growing single crystals of Bi-2223 provided an opportunity to study its irreversibility line (IRL) [4]. A sharp irreversibility field (H^*) drop was observed between 20 and 40 K. Furthermore, H^* was only ≈ 0.3 T at 75 K. This observation showed very weak intrinsic pinning in Bi-2223 single crystals.

In zero applied magnetic field, the critical current of superconducting tape $I_c(0)$ is controlled by the transfer of current between grains. Grain boundaries act as barriers to the transfer of transport current between grains. The crystallographic anisotropy of Bi-2223, which exhibits a platelike morphology, allows large contact areas, alignment of grains with their c-axes perpendicular to the rolling direction of the tape, easy transfer of current across grain boundaries, and high J_c values [5-8]. In applied magnetic field, the critical current of superconducting tape $I_c(H_{\text{app}})$ is controlled by both "intragranular" and "intergranular" effects of flux pinning [9]. With a magnetic field H_{app} parallel to the c-axis of anisotropic Bi-2223 grains, a stacking of 2-D pancake vortices forms in CuO_2 layers and these layers are weakly coupled to each other [10]. The interlayer coupling between 2-D pancakes becomes weaker with increasing magnetic field. At certain fields well below the upper critical field, the motion of vortices is strong enough to bring the J_c value to zero. J_c remains relatively field-independent in fields applied along the CuO_2 planes.

The alignment of grains and their junctions has been discussed in terms of current transport across the grain boundaries by two models: the brick-wall and railway-switch [11,12]. The brick-wall model describes the low-temperature J_c - H_{app} behavior well. Transport critical current along the c-axis is seen as the bottleneck for current flow in the tape. In the railway-switch model, the small-angle tilt boundaries are assumed to be the strongest links and therefore

responsible for the current transfer. Significant currents can move across the tilt boundaries. The microstructural observations seem to support both models [13,14].

During fabrication and service, the conductors are subjected to axial and bending stresses. In operation, the material is subjected to additional stresses by temperature gradients and magnetic fields. In large and/or high-field magnets, electromagnetic hoop stresses could even reach the ultimate strength of the material. These stresses can cause microstructural damage in the conductors and thereby degrade current transport properties. Although Ag is widely used as a sheath material, its mechanical properties are not adequate to withstand the stresses developed during fabrication and service. Therefore, techniques such as adding Ag to the superconductor powder, using alloy sheath material as an alternative to Ag, and fabricating multifilament conductors have been developed to improve the strain tolerance characteristics of the conductors [15].

The use of Ag-2 at.% Mg alloy as an outer sheath in multifilament tapes provided excellent mechanical properties [16]. The AgMg outer tube combined two advantages: having a pure Ag sheath in contact with Bi-2223 filaments and thus avoiding possible chemical reaction, and a ductile, deformable outer tube of AgMg that becomes stronger through the MgO dispersions formed during heat treatment of the tape. However, the critical current reached only 80% of the value for the reference tape with a pure Ag sheath. The indication was that the oxygen exchange between filaments was modified, which affected the formation of 2223 phase and therefore the transport current values.

Several multifilament Bi-2223 tapes were made with pure Ag and Ag-alloy (AgMg or AgMn) sheaths [17]. The slope n of the V-I curves in a double logarithmic plot was taken as an indication of the tape quality. Microscopic defects such as microcracks in the filaments should force the current to flow locally through the Ag sheath, reducing the n value [18]. The n value in tapes made using Ag alloy was 14-15, compared with an n value of 20-22 for tapes with pure Ag [17]. These transport measurement results strongly suggest that the J_c reduction in Bi-2223 multifilament tapes with Ag-alloy sheaths occurs due to the local microscopic defects in individual filaments. To evaluate the strain tolerance characteristics of the Bi-2223 tapes, in-situ bending tests were conducted [19,20]. The bending characteristics of mono- and multifilament conductors showed that irreversibility strain (ϵ_{irr}) for the monofilament conductor increased with a decrease in superconductor fill factor. In multifilament tapes, the added Ag increased the strain tolerance of the tape, which is consistent with reported I_c values for bending strains [19,20]. The improvement in mechanical tolerance for bending in multifilament tapes is possibly due in part to the better grain alignment of Bi-2223 grains.

EXPERIMENTAL PROCEDURE

To fabricate long lengths of superconducting tape with high critical current densities, it is necessary to optimize conductor uniformity. This broad category includes parameters such as initial powder properties, deformation processing, and thermomechanical conditions (indicated in Table I). Recently, we have varied the packing density of the precursor powder, improved the mechanical processing, and modified the heat treating schedule; the results are described in this paper.

Table I Fabrication and characterization parameters for PIT Bi-2223 tapes

PARAMETERS	VARIABLES
Starting powder	1) Nominal Composition 2) Phase Composition 3) Powder vs. Pressed Rods (particle size and packing density)
Tube material	4) Pure Ag vs Ag-alloys
Drawing and rolling	5) Monofilament vs. multifilament 6) Reduction ratio per pass 7) Annealing 8) Rolling vs. "semicontinuous" uniaxial pressing
1 st Heat treatment	9) Temperature and atmosphere 10) Time 11) Heating and cooling rates
Intermediate deformation step	12) Reduction ratio 13) Rolling vs "semi-continuous" uniaxial pressing
2 nd Heat Treatment	14) Temperature and atmosphere 15) Time 16) Heating and cooling rates
Characterization	17) Transport current properties (temperature, magnetic field, AC losses) 18) Mechanical properties (tensile and bending stress/strain)

As in our previous study [21], multifilament Ag-clad Bi-2223 tapes were made by the PIT technique with precursor powder having the overall stoichiometry of Bi-2223. The precursor powder contained Pb-added 2212,

Ca₂PbO₄, alkaline-earth cuprate, and CuO phases. Packing density in the Ag tubes was varied by using precursor powder, including that prepressed into billets. The precursor powder was packed into the Ag tubes at a density of $\approx 2.3 \text{ g/cm}^3$, while the precursor billets were of two densities: 3.5 g/cm^3 (low packing density) and 4.5 g/cm^3 (high packing density). The powder and prepressed billet Ag tubes were swaged, drawn through a series of dies, and then rolled to a final thickness of $\approx 200 \text{ }\mu\text{m}$. The standard mechanical processing consisting of $>10\%$ reduction per pass was used in fabricating these tapes. Tape samples containing 37-filaments and measuring 1.5 m in length were cut from these three tapes and heat treated in 8% oxygen atmosphere. In another set of experiments, we varied the mechanical deformation schedule. The Ag tubes were drawn and rolled according to various reduction ratios per pass. Load cells were mounted on the dies, and pressure exerted on the wires being drawn was monitored. Reduction ratios per pass were optimized on the basis of the die pressure measurements. Ag-Mg alloy was also used to fabricate 37-filament tapes by PIT.

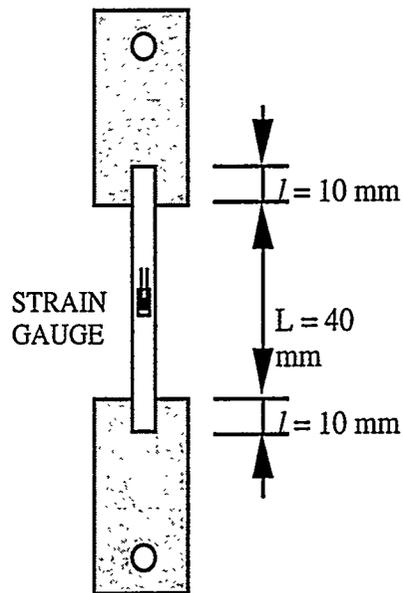


Figure 1 Schematic arrangement showing sample of Bi-2223 tape for tensile testing

The heat treatments were carried out in an 8% oxygen/balance nitrogen atmosphere according to the following schedule:

- 1) heat to 830°C at a rate of $2^\circ\text{C}/\text{min}$;
- 2) hold at 830°C for 50 h;

3) cool at 2°C /h to 800°C, at 10°C /h to 700°C and at 60°C /h to 30°C.

Dependence of the critical current density on magnetic field and temperature for the optimally processed tapes was measured. The transport critical current were measured at 77 K with a 1 $\mu\text{V}/\text{cm}$ criterion in self-field and applied magnetic field. Magnetic hysteresis measurements were made in a SQUID magnetometer over a temperature range of 20-77 K in magnetic fields up to 5 T. The magnetic field was aligned normal to the tape surface, which was the direction of the c-axis for the textured tapes. A 3-cm scan was used and the field was swept in a no-overshoot mode. The Bean model was used to derive J_c values from the magnetic hysteresis loops [22].

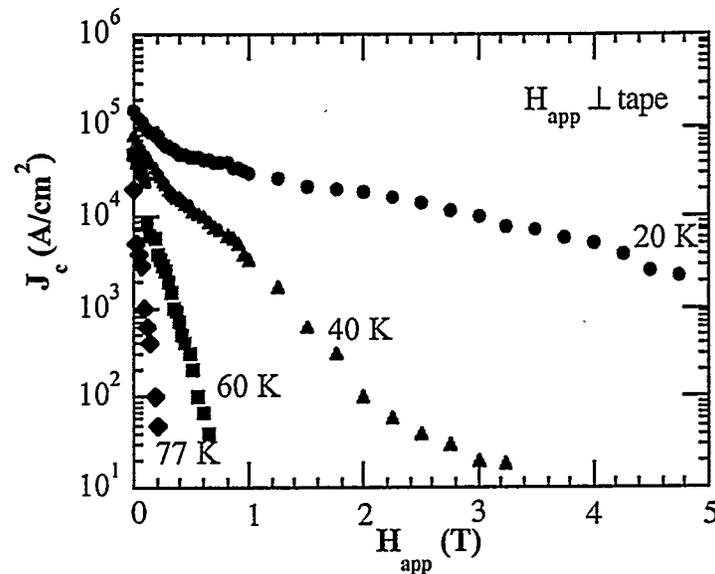


Fig. 2. Magnetization J_c vs. magnetic field applied parallel to c-axis at various temperatures (SQUID data). $J_c > 10^4 \text{ A}/\text{cm}^2$ at 20 K in 3 T field.

For the stress-strain measurements, 6-cm-long tapes were used. Figure 1 shows the experimental arrangement for tensile testing. Several samples from each spool of tape were tested under tensile load. Strain gauges were mounted on those samples in order to obtain stress-strain curves. Second, to correlate the effect of mechanical deformation on transport properties, tapes were subjected to different levels of load. The load was removed when desired value was reached. Transport critical currents were measured at 77 K with a 1 $\mu\text{V}/\text{cm}$ criterion in self-field and with applied magnetic field, before and after loading.

The effect of bending strain on I_c values was investigated by winding tapes on a G-10 mandrel of 3.81 cm (1.5 in.) diameter at 30° to the longitudinal axis and then measuring transport current. The transport critical currents were measured at 77 K with a $1 \mu\text{V}/\text{cm}$ criterion in self-field. Measurements were done on 2 or 3 samples from each spool, at first half pitch, second half, over one pitch length and over two pitch lengths.

RESULTS

Improved mechanical processing of the high-density billet showed a pronounced effect on the uniformity of the Ag/superconductor interface. The smoothness of the interface was improved. This effect is important for this processing method because the interface plays an important role in controlling the grain morphology and texture of 2223 grains. The more coherent Bi-2223/Ag interface for the light-reduction specimens resulted in higher I_c values.

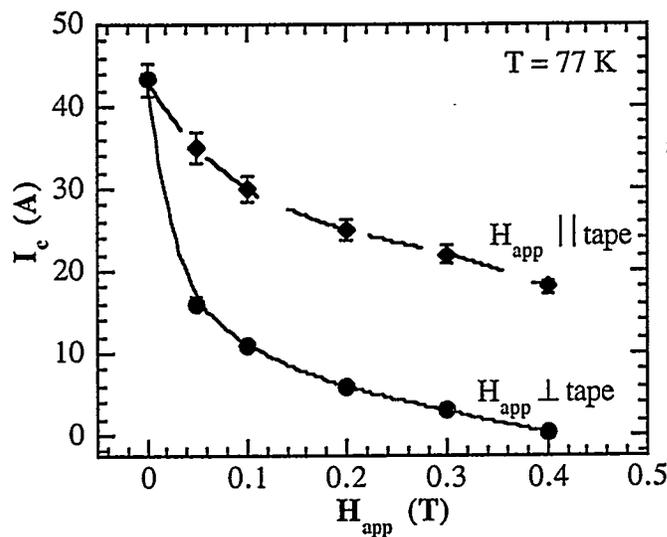


Figure 3 Characteristic magnetic field dependence of transport critical current for multifilament Ag/Bi-2223 tape at 77 K.

Dependence of critical current density on magnetic field and temperature for the optimally processed tapes was measured. Figure 2 shows the magnetization J_c (H_{app}) at several temperatures. J_c was $>10^4 \text{ A}/\text{cm}^2$ at 20 K in magnetic fields up

to 3 T and parallel to the c-axis, which is of interest for practical application in refrigerator-cooled magnets. At the higher temperatures, the sharp initial decline in J_c is followed by exponential decline with increasing magnetic field. The anisotropy of Bi-2223 with increasing temperature and field is pronounced at temperatures above 40 K and fields above 1 T.

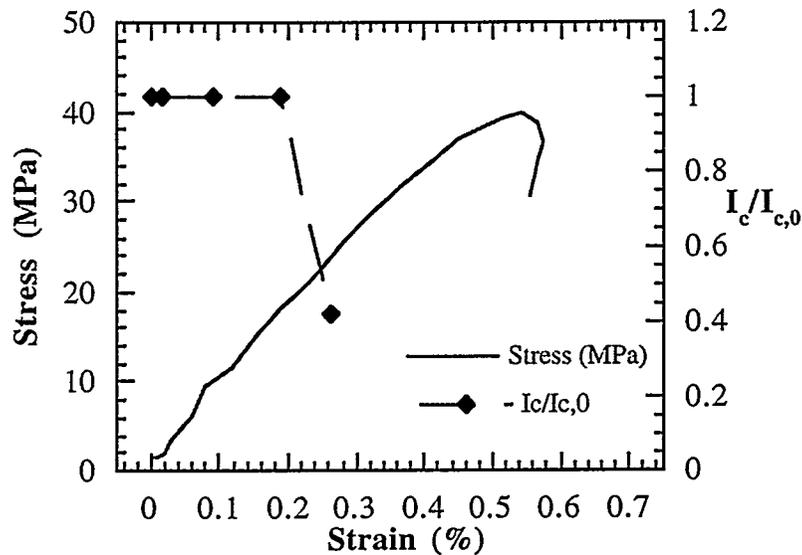


Fig. 4. Tensile stress and normalized transport critical current as functions of tensile strain for multifilament tape with pure Ag sheath.

An I_c of > 35 A at 77 K was obtained in a 200-m-long tape and zero applied magnetic field. Figure 3 shows the I_c - H_{app} characteristics of the superconducting tape that carried 42 A in a zero applied magnetic field. Magnetic fields up to 0.4 T were applied perpendicular and parallel to the tape width. The magnetic field dependence at 77 K showed pronounced anisotropy for fields parallel and perpendicular to the tape width. A perpendicular field of ≈ 0.2 T (2000 Oe) lowers the I_c value from 42 A to 4 A and changes the slope around the I_c value [30]. These results strongly suggest that ≈ 0.2 T is the "irreversibility field" (H^*) at 77 K. Figure 3 shows exponential decline of I_c with perpendicular H_{app} at 77 K.

The application of HTS tapes in electric power systems depends on their mechanical performance. Figure 4 shows the dependence of tensile stress and I_c on strain for Bi-2223/Ag tape. Mechanical properties were measured at 300 K, and transport current properties were measured at 77 K. Up to a critical stress of ≈ 20 MPa (strain of $\approx 0.2\%$) there was no degradation in I_c value. The yield strength for Bi-2223/Ag tape was ≈ 40 MPa. The strain of $\approx 0.2\%$ caused significant degradation in critical transport current, indicating that microcracks were induced in most of the filaments of the ceramic superconductor [23]. The 0.2% was the critical value of strain for a sample in tension. The corresponding load was ≈ 16 N (or 3.6 lb). If it is assumed that load is uniformly distributed between filaments in pure tension, then for each filament that carried ≈ 1 A before deformation, the amount of current was reduced to 0.4 A after 0.2% strain.

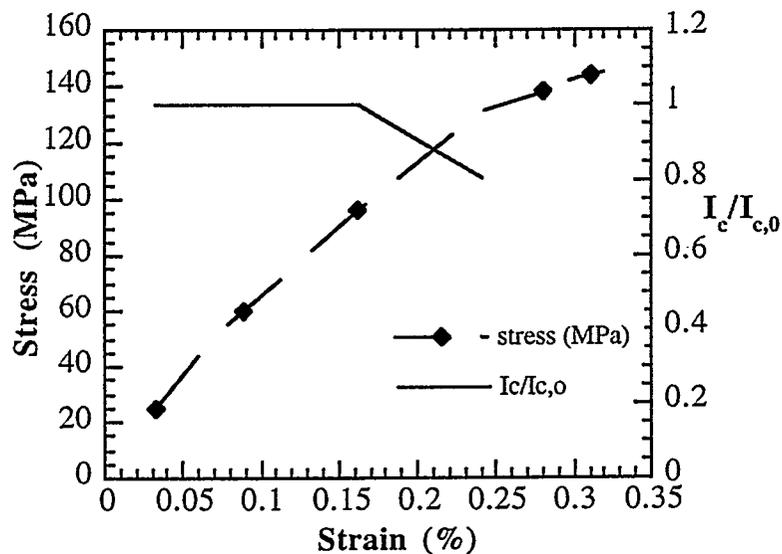


Fig. 5. Tensile stress and normalized transport critical current as functions of tensile strain for multifilament tape with Ag-Mg alloy sheath. Mechanical properties were measured at 300 K, and transport current properties were measured at 77 K.

Improvement in the mechanical properties was achieved by using Ag-Mg alloy as sheathing material. Figure 5 shows the dependence of tensile stress and I_c on strain for Bi-2223/AgMg-alloy tape. Up to a critical stress of ≈ 100 MPa (strain of $\approx 0.15\%$) there was no degradation in I_c value. Yield strength for the Bi-2223/AgMg-alloy tape was ≈ 140 MPa. The strain of $\approx 0.2\%$ caused

degradation in critical transport current. However, the decrease in I_c values was not as sharp as in the case of pure-Ag-sheathed tapes. This indicates that microcracks were induced from outside filaments to the center of the ceramic superconductor [24]. Alloy dispersion-hardened material limit the prestressing effect in the ceramic cores [24].

The effect of bending strain on I_c values was investigated by winding tapes on a mandrel of 3.81 cm (1.5 in.) diameter at 30° to the longitudinal axis, and measuring the transport current. Measurements showed that decrease in I_c as a function of bending strain can be affected by changing the sheathing material. Pure Ag-sheathed tapes showed $\approx 10\%$ reduction in I_c values, while Ag-Mg alloy sheathed tapes showed $\approx 5\%$ reduction in I_c values.

CONCLUSIONS

Transport current properties in multifilament Ag-clad Bi-2223 superconducting tapes were improved by varying the mechanical and thermal parameters during tape processing. Packing density of the precursor powder, improved mechanical deformation, and cooling rate all had a pronounced effect on the critical current of the superconducting tapes. The dependence of critical current density on magnetic field and temperature for the optimally processed tapes was measured. J_c was $>10^4$ A/cm² at 20 K in magnetic fields up to 3 T and parallel to the c-axis, which is of interest for use in refrigerator-cooled magnets. In the case of 77 K applications, an $I_c > 35$ A was obtained for a 200-m-long tape and zero applied magnetic field. I_c declined exponentially when an external field was applied perpendicular to the tape surface. Mechanical stability was tested for tapes sheathed with pure Ag and Ag-Mg alloy. Tapes made with pure Ag sheath can withstand a tensile stress of ≈ 20 MPa with no detrimental effect on I_c values. Mechanical performance was improved by using Ag-Mg alloy sheath: values of transport critical current began to decrease at the tensile stress of ≈ 100 MPa. Transport current measurements on tapes wound on a mandrel of 3.81 cm (1.5 in.) diameter at 30° to the longitudinal axis, and using a 1 μ V criterion, showed a reduction of $\approx 10\%$ in I_c values for pure Ag-sheathed tapes and 5% in I_c values for Ag-Mg sheathed tapes, as compared with the values of as-coiled tapes.

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