MELT PROCESSING OF YB-123 TAPES*

S. P. Athur, U. Balachandran,** and K. Salama
Texas Center for Superconductivity
University of Houston
Houston, TX 77204-4792

**Energy Technology Division
Argonne National Laboratory
Argonne, IL 60439

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Melt-processing of Yb-123 tapes

S.P. Athur, U. Balachandran, and K. Salama

Texas Center for Superconductivity, University of Houston, Houston, TX, USA 77204-4792

Energy Technology Division, Argonne National Laboratory, Argonne, IL, USA 60439

The innovation of a simple, scalable process for manufacturing long-length conductors of HTS is essential to potential commercial applications such as power cables, magnets, and transformers. In this paper we demonstrate that melt processing of Yb-123 tapes made by the PIT route is an alternative to the coated conductor and Bi-2223 PIT tape fabrication techniques. Ag-clad Yb-123 tapes were fabricated by groove rolling and subsequently, melt processed in different oxygen partial pressures in a zone-melting furnace with a gradient of 140 {degree}C/cm. The transition temperatures measured were found to be around 81 K under most processing conditions. EPMA of the tapes processed under different conditions show the 123 phase to be Ba deficient and Cu and Yb rich. Critical current was measured at various temperatures from 77 K to 4.2 K. The $I_c$ increased with decrease in $pO_2$. The highest $I_c$ obtained was 52 A at 4.2 K.

1. INTRODUCTION

The current thrust of HTS research is to devise economically feasible, commercially scalable processes for delivering long length conductors which are competitive in terms of cost and performance to the copper cable industry. For most of the past decade, Bi-2223 oxide powder-in-tube (OPIT) process has been the workhorse of the HTS industry, but it has been limited thus far to use in prototypes [1]. Efforts are currently being made to develop second generation wire technology using novel processing techniques such as IBAD and RABiTS [2,3]. These techniques have shown good promise in terms of performance, yielding high $I_c$ values, but their feasibility in terms of cost competitiveness to the OPIT process is still being explored. In the case of RBaCu$_2$O$_x$ (R-123) system, melt-texturing has been shown to be a successful technique for achieving high $I_c$ values in disks and bars [4,5]. However, this technique has not yet been successfully adapted to conductor fabrication for several reasons. Firstly, the high temperatures associated with the heat treatment, and the presence of a high volume fraction of liquid phase, necessitates the use of a flexible metallic substrate, or cladding material. The only material benign to R-123 is Ag, however, its use is curtailed in view of the melt-processing temperatures being higher than its melting point in most R-123 systems. Among the R-123 systems, YbBa$_2$Cu$_3$O$_x$ (Yb-123) has been reported to have the lowest peritectic decomposition temperature, and hence, the lowest expected processing temperatures [6]. In order to use Ag as cladding in melt-texturing processes, one is therefore, limited to Yb-123 as the choice of HTS material. The OPIT process is a very well established and standard method for fabricating long length tapes, and serves as an ideal candidate for making Yb-123 conductors.

Very little work has been done on Yb-123 thus far, primarily in view of the apparent difficulty in forming the Yb-123 phase. Athur et al. [7] have
managed to synthesize nearly phase-pure Yb-123 powder by a simple solid state sintering process, and subsequently, fabricated Ag-clad Yb-123 superconducting tapes, reporting $T_c$'s above 77 K on the melt-processed tapes [8]. This paper reports on the effect of the processing atmosphere on the superconducting properties

2. EXPERIMENT

The processing of the Yb-123 tapes involved essentially 4 steps, namely, powder synthesis, tape fabrication, melt-processing, and annealing.

2.1. Powder synthesis

The powder synthesis was performed through a solid state sintering route using precursor Yb$_2$O$_3$, CuO and BaCO$_3$. The sintering atmosphere was Ar + 1% O$_2$ flowing gas, and the sintering temperature was 825 °C. Details about the phase formation characteristics are listed elsewhere [7].

2.2. Tape fabrication

A silver billet of dimensions 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 $\approx$ 35%, and closed. This silver billet was then subjected to a mechanical deformation which consisted of two stages, a groove-rolling during which the billet was reduced in cross-sectional area, and a final flat-rolling stage to form the tape. After the groove-rolling, the cross-sectional dimensions of the billet were 2 mm x 2 mm and that of the as-rolled tape were 3.3 mm x 0.25 mm. Small pieces were cut from the as-rolled tape, weighed and cross-sectional area measurements performed in order to determine the packing efficiency of the powder.

2.3. Melt-processing

The melt-processing step was performed in a high gradient zone-melting furnace. Four cm long pieces were cut from the as-rolled tape, mounted on an alumina rod, inserted into the preheated furnace, held there for a half-hour to bring the sample to thermal equilibrium, and then moved through the furnace at a controlled rate. The gradient in the processing zone was determined to be 130 - 150 °C/cm. The travel length in the furnace was 50 mm. The processing atmosphere was controlled so as to study the effect of the oxygen partial pressure, pO$_2$, on the superconducting properties of the Ag-clad Yb-123 tapes. The maximum temperature in the hot zone was set at 945 °C.

2.4. Annealing

The melt-processed tapes were subjected to an oxygen anneal to convert the tetragonal phase into the superconducting orthorhombic phase. The annealing profile used is shown in Figure 1. Extended annealing times in 100% O$_2$ were used in order to isolate the effect of this step.

2.5. Characterization

Resistive $T_c$ and transport $J_c$ measurements were performed using the four-point method. Transport $J_c$ measurements were performed at different temperatures from 77 K down to 4.2 K. AC susceptibility measurements were used to determine $T_c$ by the magnetization method. EPMA was used to study the phase assemblage of the melt-processed superconducting core, and scanning electron microscopy was used to look at porosities, voids, and cracks.

3. RESULTS & DISCUSSION

3.1. Packing density of tapes

Cross-sectional area measurements made on different sections of the tape
showed minimal variation. From the weight of the tape, its dimensions, and the cross-sectional area measurements of the core, the packing density was determined to be 85%, with a standard deviation of 6%.

3.2. Effect of $p_{O_2}$

Three processing atmospheres were studied with $p_{O_2}$ values of 0.21, 0.01 and 0.001 atm respectively.

3.2.1 $T_c$ measurements

There was not much change in the $T_c$ when the processing atmosphere was varied. Figure 2 shows the magnetization behavior for a sample processed in 0.01 atm while Figure 3 shows a resistive plot for a sample processed in 0.001 atm. In both cases, the $T_{cons}$ was around 82 K. The broad transition in the magnetization curve shows inhomogeneity in the sample, indicating presence of regions with differing $T_c$ values. On the other hand, in 0.001 atm, the transition is fairly sharp, within two degrees, showing a well connected percolative path.

3.2.2 $J_c$ measurements

While the $J_c$ values in the first two cases remained fairly low, significant changes in the $J_c$ values were observed at a $p_{O_2}$ of 0.001 atm. Figure 4 shows a V-I plot for samples processed at a $p_{O_2}$ of 0.21 atm and 0.01 atm. Both samples carried very little current at 77 K and 65 K. However, when the $p_{O_2}$ was reduced to 0.001 atm, the $J_c$ at 65 K increased significantly (Figure 5).

At 4.2 K, sample A had an $I_c$ of 52 A, which was approximately equal to a $J_c$ of 20,000 A/cm².

3.2.3 SEM/EPMA

EPMA revealed the core to be multiphasic, having apart from Yb-123, Yb-211, BaCuO2 and CuO (Figure 6). A significant fraction of voids, pores and
cracks were also observed. WDS shows the Yb-123 to be nonstoichiometric, with a slight Ba deficiency, and some Yb and Cu excess.

4. CONCLUSIONS
The processing atmosphere is very crucial to determining the $I_c$ of the Yb-123 melt-processed tapes. The $T_c$ is however, fairly independent of $pO_2$. The highest $I_c$ value obtained was 52 A at 4.2 K. WDS reveals possible solid solution formation in the Yb-123 system.

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