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THE INFLUENCE OF COOLING RATE ON THE CRITICAL CURRENT DENSITY OF BSCCO-2223 Ag-SHEATHED SUPERCONDUCTOR TAPES USING POWDERS PREPARED BY CO-PRECIPITATION METHOD

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ABSTRACT

The influence of cooling rate after final reaction on the transport critical current density of Ag-sheathed $Bi_2Sr_2Ca_2Cu_3O_x$ superconductor tapes prepared from fine powders is reported. The tapes were fabricated using the powder-in-tube (PIT) method. The critical temperature, T_c and transport critical current density, J_c were measured at liquid nitrogen temperature by standard four-probe method in conjunction with a CTI cryogenics Model 21 closed cycle refrigerator. A Philips XL-30 scanning electron microscope was used to record the microstructure of the sample. It was found that J_c for these tapes increased by 40% when slow cooled at 1 ^oC/min. The 2223 phase generally align almost parallel to the current flow direction. SEM image also shows that larger grains size is visible in high critical current density tapes. We suggested that the decreased in J_c occur when cooled with slower rate than 1 ^oC/min is because of the appearance of the 2212 phase by the decomposition of the 2223 phase.

INTRODUCTION

Since time immemorial advances in technology depend greatly on human ability in understanding properties of materials [1]. The copper-oxide superconductor are expected to contribute to the development in electrical and electronic sectors in the future. In order to produce a good superconductor tape, many parameters must be controlled and optimized during the fabrication and heat treatment procedures. Fabrication process using PIT method may break the a-b plane alignment of the BSCCO grains and therefore carefull heat treatment must be followed to heal the crack. Because the preparation of a high fraction of Bi-2223 phase is a very elaborate procedure, improvement in the heat treatment procedure have been the subject of many studies. However, the optimum annealing conditions reported in the literature are quite different because they also depend on many other parameters, such as starting powders, particle size and annealing atmosphere.

One of the goals of research in Ag-sheathed BSCCO-2223 tapes is to improved the microstructures of the final superconducting tape [2]. Microstructures containing a large volume fraction of secondary phase particles are not desirable because these particles can disturb the alignment of the BSCCO-2223 grains [3] and decreased the J_c of the tapes. The size of superconductor powders is one of the parameters that must be considered in order to produce good quality superconducting tapes [4]. Enhancement in J_c by manipulation of the final cooling rate was reported by Parrell et al.[5]. Meanwhile, significant difference in the optimum cooling rate for achieving the best J_c was reported [5,6].

In this work, superconductor powders produced by the co-precipitation method with fine grain size are used as starting material for powder-in-tube tapes fabrication. We studied the first step of heat treatment because the major transformation of 2212 phase to 2223 phase occurs during this heat treatment. We report results, in which the tapes were heat treated at different cooling rate at 850 ^oC for 48 hours. The purpose of this method is to find a better way which could reduce the processing time.

METHODOLOGY

Powders synthesized by coprecipitation were prepared from a mixed acetate solution of $Bi(CH_3COO)_3.5H_2O$, $Sr(CH_3COO)_2$, $Pb(CH_3COO)_2Ca(CH_3COO)_2.H_2O$, and $Cu(CH_3COO)_2$, corresponding to the nominal composition $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$. The coprecipitation powder was prepared by pouring solution containing metal ions in another containing 0.5 M oxalic acid dissolved in water: isopropanol (1: 1.5). A uniform, stable, blue suspension was obtained. The slurry was filtered after 5 minutes of reaction followed by the drying stage in the temperature range of 80-85 ^oC for 8 – 12 hours. Blue precipitate powder, which is slightly aggregated with particle size of 0.1 – 0.6 µm was heated up to 730 ^oC in air for 12 hours to remove the remaining volatile materials. The calcined powder was reground in a marble mortar for 10 minutes and heated up again at 845 ^oC in air for 24 hours followed by oven cooling at 2 ^oC/minute.

Ag-sheathed Bi-2223 tapes with nominal composition of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_x$ were prepared using the standard powder-in-tube method. A silver tube with 6 mm outer diameter and 4.5 mm inner diameter was heated at 600 ^oC for 1 hour to eliminate organics elements prior to the filling stage. The powders were packed into the silver tube with packing density of about 2.3 g/cm^3. The tube was then rolled to form wire with 1.15 mm outer diameter which then rolled into tape with 0.25 mm thickness. The tapes for heat treatment were obtained by cutting the as-prepared tapes into short sections (~ 2 cm long). These samples were heated at 2 ^oC/min in air until 850 ^oC for 48 hours followed by cooling to room temperature at 2 ^oC/min, 1 ^oC/min and 0.5 ^oC/min. The T_c and J_c of the tapes were measured at liquid nitrogen temperature by the standard four-point probe method in conjunction with a CTI cryogenics Model 21 closed cycle refrigerator. A Philips XL-30 scanning electron microscope (SEM) was used to record the microstructure of the samples. Phase composition in the tapes was determined using X-ray diffraction (XRD) method.

RESULTS AND DISCUSSION

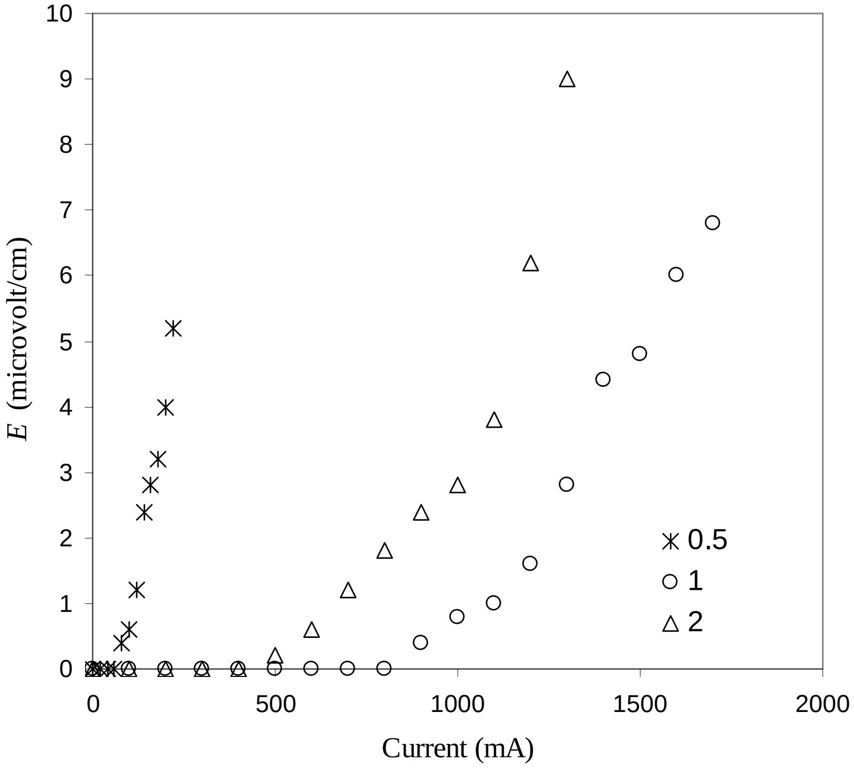


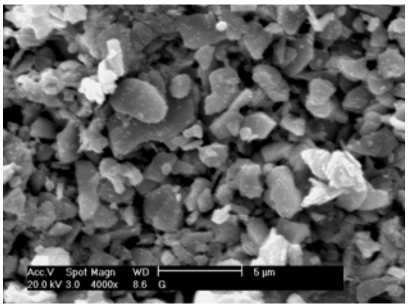
Figure 1. *E* vs current for tape with different cooling rate

Fig. 1 shows the electric field (*E* = *V*/distance between voltage contacts, *d*) *vs* current of the sample for different cooling rate. Table 1 shows that the critical current, I_c and critical current density, J_c of the tape reaches maximum for the tapes that have undergo heat treatment with cooling rate of 1 ^oC/min. I_c was obtained from Fig. 1 by using 1 μV/cm criterion. J_c was calculated by dividing I_c with cross-sectional area of the superconducting core.

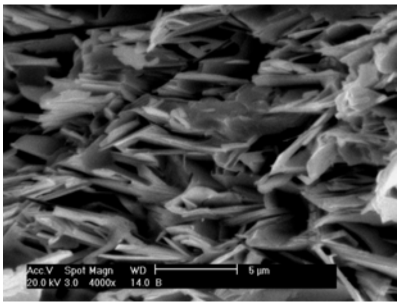
Table 1 : I_c and J_c of the tapes

Tapes sample	Critical current, I_c (mA)	Critical current density, J_c (A/cm^2)
2 ^oC/min	800	6000
1 ^oC/min	1100	9000
0.5 ^oC/min	150	1230

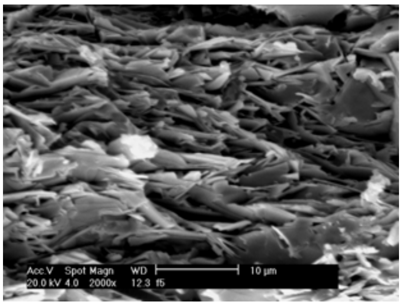
Previous studies of Bi-2223 tapes [2] shows that microstructure of the tape plays an important role in determining the superconducting properties. Longitudinal cross-section parallel to the rolling direction of each sample were imaged in a Philips-XL scanning electron microscope (SEM) as shown in Fig. 2.



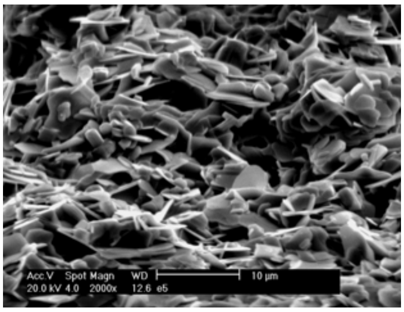
(a)



(b)



(c)



(d)

Figure 2 : SEM micrographs of the tapes (a) starting powder, (b)2 ^{o}C/min, (c) 1 ^{o}C/min and (d) 0.5 ^{o}C/min

The micrograph of the starting powders is shown in Fig. 2 (a) which is about 1-2 µm. SEM picture in Figure 2(b), (c) and (d) shows the longitudinal cross-sections of the tapes that have been heat treated at different cooling rate; i.e (b) 2 ^{o}C/min, (c) 1 ^{o}C/min and (c) 0.5 ^{o}C/min. All of the samples are *c*-axis oriented, with the *c*-axis of the grains perpendicular to the tape surface in which 2223 grains having a common c-axis separated by (001) twist boundaries. Since the 2223 grains tend to grow inward from the interface of silver and ceramic core, any roughness the interface tends to propogate into the unaligned oxide core. This sets up conditions for the formation of high angle grains boundaries between neighbouring 2223 grains. Therefore the mechanical process is one of the important factors that must be consider during fabricating of the superconductor tape.

From these micrographs, it appears that the average grain size increased by almost 100 %, when the cooling rate is reduced to 1 ^{o}C/min. The higher values for the critical current density observed in these tapes could be explained by the assumption that for tape with larger grain size, the total grain boundaries was reduced and this could lead to better transport current properties [7]. Grain boundary is one type of weak links which would resist the flow of current in the tapes. The larger contact area between the grain can also reduce resistance to the current flow in *c*-axis direction as proposed by brick-wall model [8]. SEM observation also shows the presence of small angle tilt boundaries in high critical current density tape. Microstructures of the tape shows that transport current properties could be explained by railway-switch model [9].

Of additional concern, in the production of high quality superconductor tapes is the presence of secondary phases, such as alkaline earth cuprates or Bi-2212. These secondary phases tend to reside on grain boundaries and even in small amounts, they could dominate the transport properties of the tapes [2]. In this work, although there is a higher 2223 phase content in the tape cooled with rate slower than 1 ^{o}C/min (i.e 0.5 ^{o}C/min), the appearance of 2212 phase at the 5.8^{o} with noticeable intensity could explain why the maximum J_c is obtained at 1 ^{o}C/min. Some authors [7] have reported that faster cooling freezes the optimum phase equilibrium region and yields a minimum amount of 2212, while slower rates enhance its presence. Parrell et al. [5] suggested that the 2212 formed by decomposition has a very different morphology than that of residual 2212. Some authors [2,6] reported that residual 2212 was found in almost all 2223 grains while the 2212 formed by decomposition of the 2223 phase is larger and seems to form discrete grains [5].

CONCLUSION

In order to improve the critical current density, J_c of Ag-sheathed Bi-Pb-Sr-Ca-Cu-O tapes the influence of cooling rate have been investigated. Slow cooling at 1^{o}C/min increased J_c by almost 40 % to 9000 A/cm^2 compared to the tape that was fast cooled at 2^{o}C/min. The improvement in the microstructural properties were also observed in which the grains size increased by almost 100%. However decomposition of 2223 phase to 2212 phase occurs at cooling rate lower than 1^{o}C/min which leads to decrease in J_c. We suggest that this behaviour is caused by the changes in the connectivity of the 2223 grains as the cooling condition is varied. Great improvement in J_c could be expected with better understanding of phase transformation and microstructure of Bi-2223 system, which would lead to a better heat treatment procedure.

ACKNOWLEDGEMENT

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