Transport current density of Ag-sheathed superconductor tapes using Bi-Sr-Ca-Cu-O powders prepared by the co-precipitation method

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Improvement in the transport critical current density (J_c) of the high temperature superconductor tapes and conductors is important in realizing their full commercial potential. J_c of Bi-Sr-Ca-Cu-O superconductor tapes for example, is mainly determined by the dominant phase and the high degree of *c*-axis texture. Heat treatment procedure to fabricate these tapes using the powder-in-tube method with high content of $Bi_2Sr_2Ca_2Cu_3O_x$ (Bi-2223) phase is complex and depends on many factors such as starting composition [1–4], processing temperatures and atmosphere [5–9], heating time [10] and cooling rates [11].

For the Bi-Sr-Ca-Cu-O superconductor tapes, a long heat treatment of up to several hundred hours is required to obtain a high fraction of Bi-2223 phase [12]. In order to reduce the heating time, improvements in the processing and heat treatment procedure have been the subject of many studies. The initial heat treatment is very important because during this stage most of the precursor powders are converted into the Bi-2223 phase. However the presence of secondary phases such as $Bi_2Sr_2CaCu_2O_x$, $Bi_2Sr_2CuO_x$, Ca_2PbO_4, Sr_2PbO_4, CuO, and $Ca_7Bi_{10}O_{22}$ cannot be avoided. The Bi-2223 phase is stable (not necessarily as a single phase) for $\sim 835\,^\circ C < T < 880\,^\circ C$ and for compositions approximately lying between the stoichiometric composition and a copper–calcium-rich composition close to $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_x$ [13].

The size of starting superconductor powders is one of the parameters that must be considered in order to produce high quality superconductor tapes [12]. 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 also present results of the effects of heating time and microstructure on the transport critical current density of the tape measured at liquid nitrogen temperature in magnetic field applied parallel and perpendicular to the tape's surface.

Superconductor powders with nominal composition of $Bi_{1.6}Pb_{0.4}Sr_2Ca_3Cu_4O_x$ were synthesized by the co-precipitation method from a mixed acetate solution of $Bi(CH_3COO)_3{\cdot}5H_2O$, $Sr(CH_3COO)_2$, $Pb(CH_3COO)_2Ca(CH_3COO)_2{\cdot}H_2O$, and $Cu(CH_3COO)_2$. The co-precipitation powders were prepared by pouring the solution containing the metal ions into another containing 0.5 M oxalic acid dissolved in water:isopropanol (1:1.5) and a uniform, stable, blue suspension was obtained. The slurry was filtered after 5 min of reaction time followed by a drying stage in the temperature range of 80–85 °C for 8–12 hr. The blue precipitate powders, which are slightly aggregated with particle size of 0.1–0.6 μm, were heated up to 730 °C in air for 12 hr to remove the remaining volatile materials. The calcined powders were reground and heated again at 845 °C in air for 24 hr followed by oven cooling at 2 °C/min.

Ag-sheathed tapes were fabricated by 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 °C for 1 hr to eliminate organic elements prior to the filling stage. The powders were packed into the silver tube with a packing density of about 2.3 g/cm^3. The tube was then rolled to form wire with 1.15 mm outer diameter which was then rolled into tapes with 0.25 mm thickness and cut into short sections about 2 cm in length. The samples were heated at 2 °C/min in air to 850 °C and held at that temperature for 24 hr (24-hr), 48 hr (48-hr), and 72 hr (72-hr) followed by cooling to room temperature at the same rate.

The transition temperature (T_c) was measured at liquid nitrogen temperature by the standard dc four-point probe method in conjunction with a CTI cryogenics Model 21 closed cycle refrigerator. The transport critical current density (J_c) was determined by dc four-probe technique with the tapes immersed directly into liquid nitrogen. J_c measurements were performed on several tapes of the same heat treatment to confirm the reproducibility of the critical current. The 1-μV/cm criterion was used to define J_c. In this criterion as the current is varied, the voltage (V) across the tape is measured and divided the distance d between the voltage probes. J_c is determined as the value of current where V/d is 1 μV/cm. The transport current measurements were made in zero and applied magnetic fields up to 0.30 T. Magnetic fields were applied perpendicular to

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TABLE I Zero-resistance transition temperature ($T_{c\,zero}$), onset transition temperature ($T_{c\,onset}$), and transport critical current density (J_c) for the tapes that were heated at 850 °C for 24 hr (24-hr), 48 hr (48-hr), and 72 hr (72-hr)

Tape sample	$T_{c\,zero}$ (K)	$T_{c\,onset}$ (K)	J_c (A/cm^2)
24-hr	97	102	2400 ± 60
48-hr	101	109	6500 ± 200
72-hr	102	109	13 400 ± 300

the direction of current flow, parallel ($H_{||}$) and perpendicular ($H_{\perp}$) to the plane of the tapes. A Philips XL-30 scanning electron microscope (SEM) was used to record the microstructure of the samples.

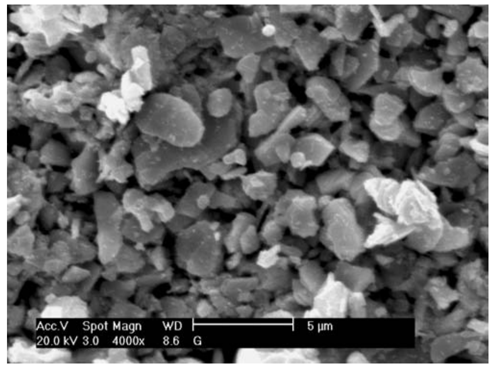
J_c of the tapes increased with heating time (Table I). Transition temperature also increased with heat treatment time which indicates that there was a transformation from the low-T_c phase (Bi-2212) to the high-T_c phase (Bi-2223).

Previous studies of Bi-2223 tapes revealed that their microstructure plays a significant role in determining the superconducting properties. In order to correlate the microstructure and the transport current observed, micrographs of these tapes have been recorded and are shown in Fig. 1. Fig. 1a shows the starting powders, which are about 1–2 μm in size. SEM micrographs in Fig. 1b, c and d show the longitudinal cross-sections of the tapes. All of the tapes except the 24-hr one are c-axis oriented. From these micrographs, it appears that the average plate size increased with time, i.e., 1 μm for 24 hr, 5 μm for 48 hr, and 10 μm for 72 hr of heat treatment, respectively.

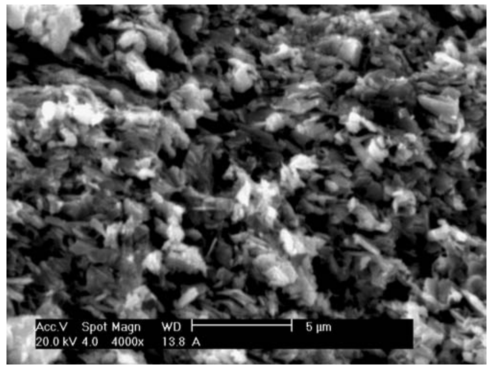
The higher values for the critical current density observed in tape 72-hr of 13 400 ± 300 A/cm^2 at 77 K is due to the longer plates where boundaries between plates are reduced, leading to better transport current properties. It is generally known that grain boundary is one type of weak link which would resist the flow of current in the tapes. The larger contact area between the grains can also reduce resistance to the current flow in the c-axis direction. SEM micrographs also show small angle tilt boundaries in the higher critical current density tape. Microstructures of the tape show that transport current properties could be explained by the railway-switch model [14].

J_c versus applied field of the tapes decreased drastically as soon as the field was applied (Fig. 2). This is especially true for the tapes that exhibit plate-like structure with high J_c (48-hr and 72-hr tapes). The higher the J_c, the more drastic is the drop. The main difference between the tapes is the links between the grains which can be divided into two groups, i.e., weak links (e.g., large angle tilt boundaries and twist boundaries) and strong links (e.g., small angle tilt boundaries). Weak links are responsible for the sudden decrease of J_c in low fields while the slow reduction (plateau) in J_c at higher fields is due to strong links.

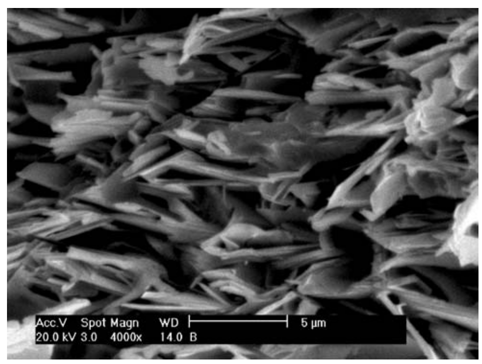
In conclusion, the effect of heat treatment on the transport critical current density of Ag-sheathed superconductor tapes employing powders prepared by the co-precipitation method with nominal starting composition $Bi_2Sr_2Ca_2Cu_3O_x$ is reported. The microstructure which consists of plate-like structure becomes more c-axis oriented as the heating time is increased. The transport critical current density, J_c, of the tapes increased



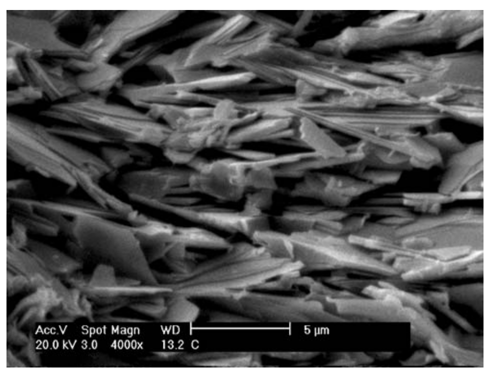
(a)



(b)



(c)



(d)

Figure 1 (a) SEM micrograph of the starting powders. (b) SEM micrograph of the longitudinal cross section of tape 24-hr. (c) SEM micrograph of the longitudinal cross section of tape 48-hr. (d) SEM micrograph of the longitudinal cross section of tape 72-hr.

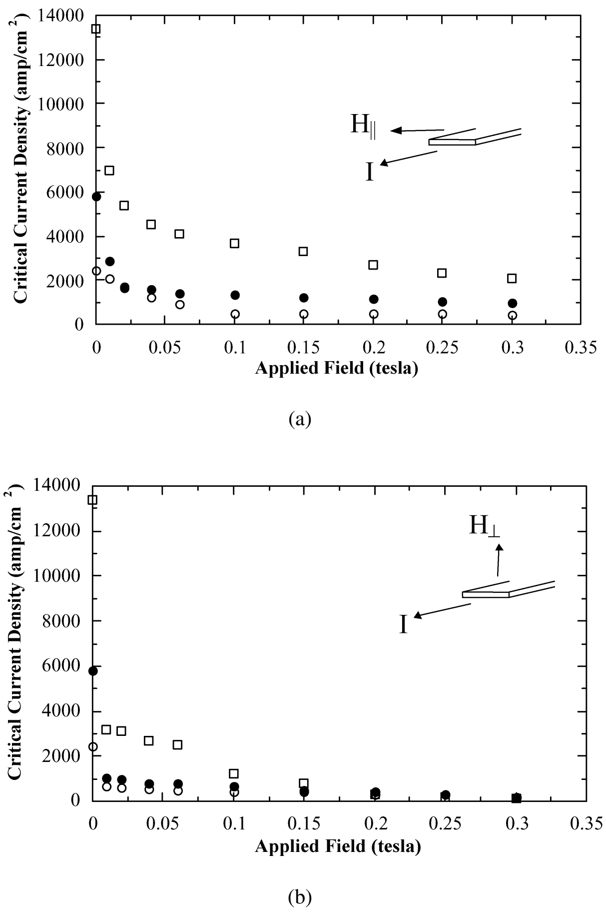


Figure 2 (a) Critical current density versus field applied parallel to the tape's plane: (○) 24-hr, (●) 48-hr, and (□) 72-hr. (b) Critical current density versus field applied perpendicular to the tape's plane: (○) 24-hr, (●) 48-hr, and (□) 72-hr.

with heating time and reached 13 400 ± 300 A/cm^2 at 77 K after 72 hr of heat treatment. This study shows that this route of using fine powders prepared by the co-precipitation method is very useful in fabricating high current density superconductor tapes. Great improvement of J_c can be expected with better understanding of phase transformation and microstructure of the Bi-2223 system, which would lead to a better heat treatment procedure.

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