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EFFECT OF SINTERING TIME ON THE TRANSPORT CURRENT DENSITY OF Ag-SHEATHED $(Bi_{1.6}Pb_{0.4})Sr_2Ca_2Cu_3O_y$ SUPERCONDUCTOR TAPES

A.Masrianis[1], S.A. Halim[1], R. Abd-Shukor[2], M.Hashim[1],
Z.A. Hassan[1] and M.M. Awang Kechik[1].

[1] *Superconductor and Thin Films Laboratory, Department of Physics,*
Faculty of Science & Environmental, University Putra Malaysia,
43400 Serdang, Selangor, Malaysia
[2]*School Of Applied Physics, Universiti Kebangsaan Malaysia,*
43600 Bangi, Selangor D.E

ABSTRACT

The effect of heat treatment on the critical current density, j_c was studied for monofilament tape samples without intermediate rolling. $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{7+\delta}$ polycrystalline ceramic powder was prepared by co-precipitation technique. The tape samples were prepared using standard powder-in-tube method and were sintered at 850°C at different sintering time. XRD result shows the peaks (*00l*) that belong to the 2223 phase which appear with the increase in sintering time. The electrical transport and magnetic properties of the tapes werewas characterized from 60 K to 77 K in zero magnetic fields and in magnetic field up to 0.6 T at 77 K. The transport critical current density, j_c for the tape sintered for 24 hour is 2400 A/cm^{-2} and is 6000 A/cm^{-2} for tape sintered for 100 hour. This showed that j_c increases with the increasing of the sintering time.

INTRODUCTION

High temperature superconductors are ceramic materials sandwiched between layers of copper and oxygen atoms. The brittle nature inherent in the ceramic possess one of the most serious difficulties which must be overcome before wider application of the material in a technological environment can be made possible (Kohno, 1988). One way to overcome this difficulty is by packing the ceramic materials or precursor powders into silver tube, which are extruded and drawn into wires or rolled to form a flat tape before being heat-treated. Several experiments have been carried out in order to elucidate the mechanisms which are responsible for the current transport in Ag-sheathed Bi (2223) tapes. It is nowadays accepted that the railway switch model first proposed by Hensel *et al*. (1995) and the freeway model by Riley *et al*. (1997) give the best interpretation of the transport measurements performed at liquid nitrogen temperature. Cimberle *et al*. (1998) identified a low temperature – low-field domain of the H-T plane where the intrinsic Bi (2223) grain current density is much higher than that of the grain boundaries; in this domain, the intergrain j_c decreases exponentially with increasing temperature, as expected. Conversely, the transport j_c presents history dependence, and it decreases linearly with increasing temperature. Cankurtaran and Saunders (1992) reported that the addition of Ag could stiffen the granular ceramic by filling the pores and intergranular spacing. Among various processing methods for superconducting oxide material, the powder-in-tube (PIT) methods appears to be the most promising for applications on the industrial scale. Various processing methods have been attempted to optimize the PIT (powder-in-tube) superconductor wires and tapes. For Bi-2223 / Ag tapes, thermomechanical treatments have been found to improve the phase purity and grain alignment. The Ag at the superconductor interface helps the superconducting phase formation and grain alignment (Kung, 1992 & Ren, 1995). The chemical routes such as oxalic acid co-precipitation, sol gel (Shich, 1991), and micro-emulsion-based techniques (Kumar, 1993) are given priority to fabricate Bi(Pb)SrCaCuO powder in order to get compositional homogeneity in such multi-component powder. Ismail *et al.* (2004) reported that the j_c is 13400 A/cm^2 for tape prepared by co-precipitation method heated at 72 hour (77 K; B=0) and scanning electron microscope result showed it is due to the longer plates where boundaries between plates are reduces. Lau et al. (2002) reported that the j_c for monofilament tape prepared by solid-state reaction heated at 24 hour is 1560 A/cm^2.

In this paper result of X-ray diffraction for monofilament tape samples and j_c of the tape due to different sintering time. The transport current measurements were made in zero and applied magnetic fields using the d.c four-point probe technique.

EXPERIMENTAL DETAILS

Superconductor powders with nominal composition of $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10}$ were prepared using the metal acetates of bismuth, strontium, lead, calcium and cooper (purity ≥ 99.99 %), oxalic acid, deionized water and 2-proponal. The co-precipitation technique has been described in a previous paper (Imad *et. al*, 2001). The powder precursor was heated up to 730 ˚C in air for 12 hours to remove the remaining volatile materials. The calcined powders were reground in a marble mortar for 30 minutes and heated up again at 845 ˚C in air for 24 hours followed by oven cooling at 2˚C/minute. Ag-sheathed tapes were fabricated by using the standard powder-in-tube-method. The powders were reground and then packed into a 2.6 mm long silver tube with outer diameter and inner diameter of 6.35 mm and 4.35 mm, respectively. Both ends of the tube were closed by Ag caps and successively drawn into wires with final outer diameter of 0.8 mm through a grooved roller. Ag-sheathed tape was made by rolling the wire from the initial diameter of 0.8 mm and decreased to 0.23 mm thickness. The tapes were cut into 2 cm long sections. The samples were heated at 850 ˚C and slowly cooled to room temperature at 6 ˚C per hours until 780 ˚C and continued at 60 ˚C per hours. The samples were heated at 850ºC for 24 hours (24-hr), 48 hours (48-hr) and 100 hours (100-hr) followed by cooling to room temperature at the same rate. The transition temperature was measured using the standard dc four-point probe technique. The transport critical current density (j_c) was determined by the dc four-point probe technique with the tapes immersed directly into liquid nitrogen. 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. A Shimadzu XRD 6000 diffractometer with CuK_α radiation was used to identify the phase in the tapes. The transport current measurements were made in zero and applied magnetic fields up to 0.6 T. Magnetic fields which are parallel ($H_\parallel$) or perpendicular ($H_\perp$) to the plane of the tapes were applied perpendicular to the direction of the current flow.

RESULTS AND DISCUSSION

Table 1 shows the result of critical current density, j_c in zero magnetic fields at 77 K for monofilament tape samples prepared by co-precipitation method heated at different sintering time. The critical current density in this work for the tape heated for 100-hour is 6000 A/cm^2. This j_c is very low compared to monofilament tape samples heated at 48 hour as reported by Ismail et al. (2004).

Tape sample	$T_{c\text{-zero}}$ (K)	Critical current density, j_c (A/cm^2)	$T_{c\text{-zero}}$ (K)	Critical current density, j_c (A/cm^2). Source: Ismail *et al*. (2004)
24-hr	100	2428	97	2400
48-hr	97	3000	101	6500
72-hr	-	-	102	13400
100-hr	96	6000	-	-

Table 1: Critical Current density, j_c in zero magnetic fields at 77 K for samples heated at different sintering time.

Figure 1 shows the X-ray diffraction patterns for monofilament tape samples sintered in air at 850 °C for 24 hour and 48 hour. The heat treatment time indicates that there was a transformation from the low-T_c phase (Bi-2212) to the high-T_c phase (2223) and from high-T_c phase (Bi-2223) to the low-T_c phase (2212) due to the re-crystallization during the heating time. 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-2212, Bi-2201, Ca_2PbO_4, Sr_2PbO_4, CuO cannot be avoided. For the samples sintered at 850 ºC for 24 and 48 hour, the peaks (002H), (004H), (00$\underline{10}$H), (00$\underline{12}$H), (00$\underline{14}$H), (026H), (02$\underline{12}$H), (222H), (00$\underline{20}$H) and (22$\underline{12}$H) belong to 2223 phase. With increasing sintering duration, some peaks belonging to the 2223 phase appear. Some shift of the high angle peak was observed and it can be concluded as due to Ag. The Ag (200) peak ($2\theta = 44°$) can be clearly seen in both spectra in Figure 1. Powders prepared by co-precipitation method are very useful in fabricating high current density superconductor tapes as compared to the powder prepared by solid-state reaction.

Figure 2 shows that the critical current density, j_c of the tapes increased linearly with the sintering time due to better alignment of the grains. Critical current density, j_c also increased at lower temperature possibly due to the increasing of cooper pairs.

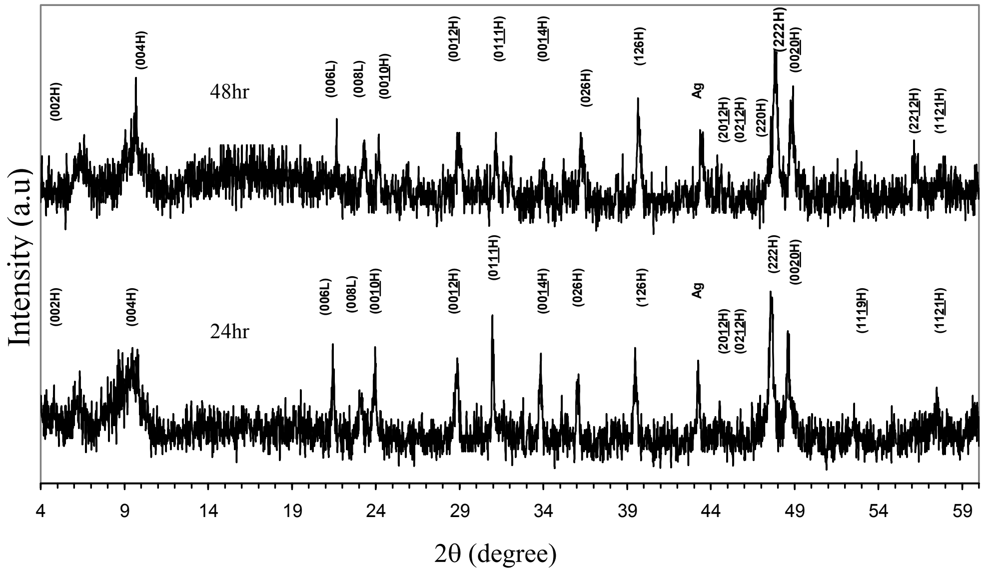

Figure.1: X-ray diffraction patterns for monofilament tape samples sintered in air at 850°C for 24 hour and 48 hour.

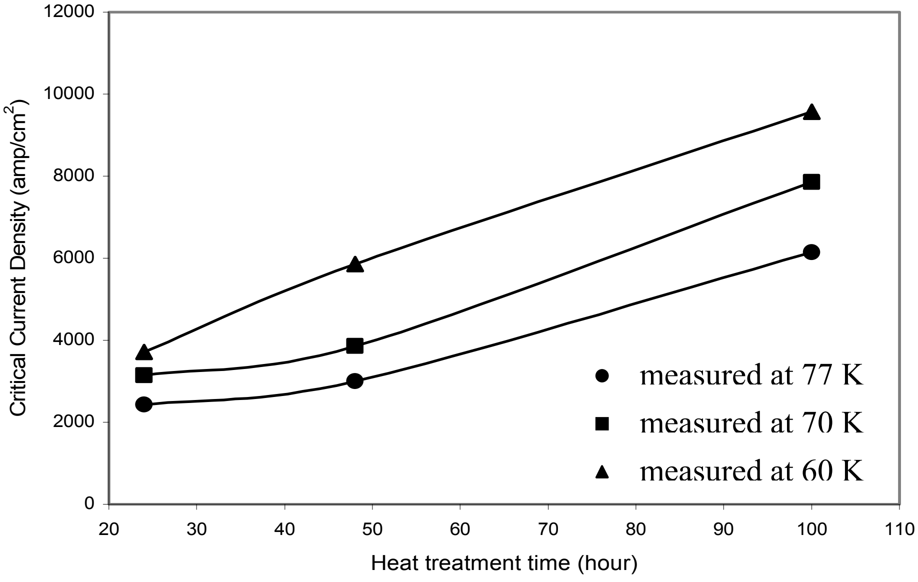

Figure 2: Critical current density versus heat treatment (sintering time) for monofilament tape at 77 (●), 70 K (■) and 60 K (▲).

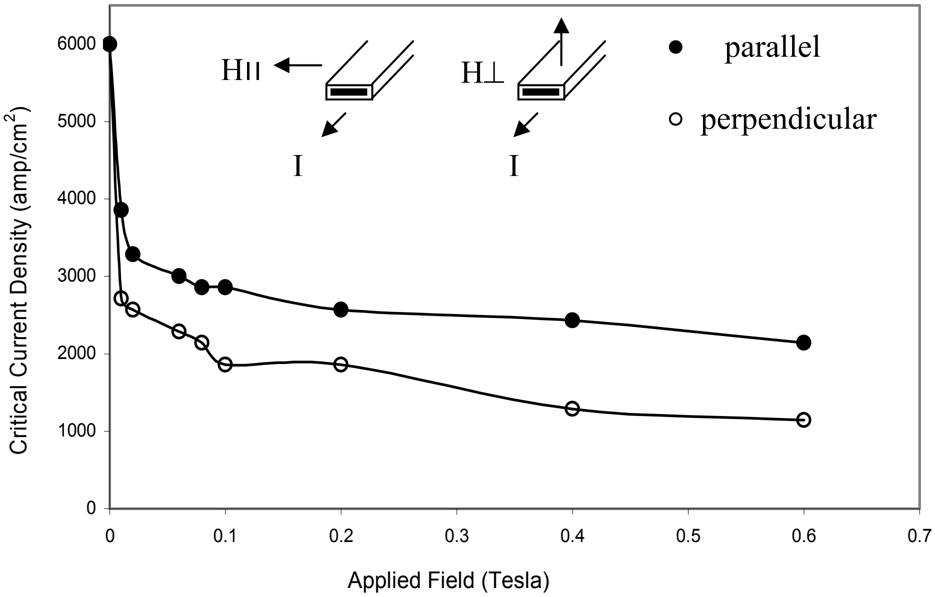

Figure 3: Critical current density versus magnetic field applied parallel and perpendicular to the tape's plane for tape sintered at 100 hours

Figure 3 shows that the critical current density, j_c versus applied field of the tapes decreased with the increase of magnetic fields. The main difference between the tapes is the links between the grains, which can divide 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 critical current density, j_c in low fields while the slow reduction (plateau) in j_c at higher fields is due to strong links (Ismail *et. al*, 2004). Previous paper present the following scheme for the current limiting mechanism in Ag-sheathed Bi(2223) tapes. Below 50K, the intergrain current limits the transport current density and an additional j_c improvement could be achieved by further reducing the misalignment angle in *c*-axis tilt grain boundaries. Conversely, above 50 K, the current-limiting mechanism is more related to the intrinsic Bi(2223) current density and an improvement of the pinning properties is essential for reaching higher currents and irreversibility fields (Cimberle *et. al*, 1998).

CONCLUSION

In conclusion, the effect of heat treatment on the transport critical current density of Ag-sheathed superconductor tapes employing powders of nominal composition $(Bi_{1.6}Pb_{0.4})Sr_2Ca_2Cu_3O_y$ is reported. The transport critical current density, j_c of the tapes increased with heating time and reached 6000A/cm^2 at 77K for 100 hours of heat treatment. The difference of critical current density, j_c value between the tapes is possibly due to the nature of the superconducting links, which may be in form of SNS (Superconducting-Normal-Superconducting) or SIS (Superconducting-Insulator-

Superconducting) between the grains. The transport current is reduced by the presence of voids and secondary phases that severely affect the number of parallel current paths through which the current flows.

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