Transport Critical Current Density of Bi$_{1.6}$Pb$_{0.4}$Sr$_{2}$Ca$_{2}$Cu$_{3}$O$_{10}$/Ag Superconductor Tapes with Addition of 50 and 70 nm MgO

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Received: 7 February 2016 / Accepted: 14 March 2016 / Published: 1 April 2016

MgO nanoparticles with average particle size 50 and 70 nm were added into Bi$_{1.6}$Pb$_{0.4}$Sr$_{2}$Ca$_{2}$Cu$_{3}$O$_{10}$ (MgO)$_{x}$/Ag superconductor tapes and sintered at 845 ºC for 50 and 100 h. The effect of sintering time on the transport critical current density ($J_c$) was investigated. $J_c$ of the tapes were measured in zero-field between 30 and 77 K and under magnetic fields at 77 K. The phase and microstructure of the tapes were examined by X-ray diffraction method and scanning electron microscopy, respectively. All tapes showed plate-like grains and a larger plate-like grain was observed in the tapes sintered for 100 h. $J_c$ of the MgO added tapes was higher than those of the non-added tapes. $J_c$ of the non-added tape sintered for 100 h was 10820 A/cm$^2$ at 30 K and 1262 A/cm$^2$ at 77 K. $J_c$ of the tapes sintered for 100 h was higher than the tapes sintered for 50 h. The 50 nm MgO added tapes showed a higher $J_c$ than the 70 nm MgO added tapes. $J_c$ for the 100 h sintered MgO (50 nm) added tape was 22710 and 3070 A/cm$^2$ at 30 and 77 K, respectively which are higher than that of the 100 h sintered MgO (70 nm) added tape. The improved $J_c$ was attributed to the increase in flux pinning potential and improvement in the microstructure due to the addition of nano-sized MgO. This work showed the importance of the size of the added nanoparticles to the transport current properties of these superconductors.

Keywords: nano MgO; size effect; transport critical current density; flux pinning

1. INTRODUCTION

Inter-grain weak links and the weak pinning of magnetic flux lines suppress the transport critical current density ($J_c$) in Bi$_{1.6}$Pb$_{0.4}$Sr$_{2}$Ca$_{2}$Cu$_{3}$O$_{10}$ 9 ((Bi,Pb)-2223) superconductors [1-3]. The doi: 10.20964/110447
weak link is also observed in silver-sheathed (Bi,Pb)-2223 tape prepared by powder-in-tube (PIT) technique [4,5]. Addition of nano-sized particles into (Bi,Pb)-2223 superconductors in bulk or tape form can enhance magnetic flux pinning potentials, resulting in an increase in $J_c$ [6-9]. Several studies have focused on the addition of different nano-sized particles into (Bi,Pb)-2223 [10-12].

The effect MgO with particle size 20 and 40 nm added into (Bi,Pb)-2223 in bulk [10] and tape form [11] has been studied. These results indicated that the flux pinning capability in the 20 nm MgO added samples was better than in the 40 nm MgO added samples. MgO is chemically inert with (Bi,Pb)-2223 and the transition temperature $T_c$ did not change considerably with MgO content [7].

When the pinning center is larger than the coherence length $\xi$, but smaller than penetration depth $\lambda$, the $J_c$ will increase [13]. A strong interaction between magnetic flux line network and the (Bi,Pb)-2223 matrix can be expected if $\xi < L < \lambda$, where $L$ is the nanoparticle size [14]. However, it has been suggested that the optimum size for flux pinning centers should be comparable to $\lambda$ rather than $\xi$ [15,16]. For (Bi,Pb)-2223, $\xi$ is 2.9 nm [17] and $\lambda$ is 60-1000 nm.

In this work we investigated the effect of MgO addition with particle size 50 and 70 nm on the $J_c$ of the (Bi,Pb)-2223 Ag sheathed tapes. At 30 and 77 K, our initial study on MgO (50 and 70 nm) added (Bi,Pb)-2223(MgO)$_x$ polycrystalline (pellet) samples (with $x = 0$ - 0.15 wt. %) exhibited the highest $J_c$ at $x = 0.15$ wt. % for 50 nm MgO and $x = 0.01$ wt. % for 70 nm MgO. These optimal amounts were used to fabricate 50 nm and 70 nm MgO added (Bi,Pb)-2223/Ag tapes. The tapes were sintered for 50 and 100 h at 845 ºC. The effect of sintering time and particle size on the transport current density is discussed.

2. EXPERIMENTAL DETAILS

Powders of Bi$_{1.6}$Pb$_{0.4}$Sr$_2$Ca$_2$Cu$_3$O$_{10}$ were prepared by the acetate co-precipitation technique. Nano-sized MgO with average size 50 and 70 nm (US-nano, 99+% purity) were added into (Bi,Pb)-2223(MgO)$_x$ with $x = 0$ - 0.15 wt. %. The optimal amounts of added pellets for the highest $J_c$ were found in the $x = 0.15$ wt. % pellet for 50 nm MgO and 0.01 wt. % pellet for 70 nm MgO additions. These amounts were chosen to fabricate (Bi,Pb)-2223(MgO)$_x$/Ag tapes by the powder-in-tube (PIT) method. The powders were filled into a 6.35 mm outer diameter and 4.35 mm inner diameter silver tube (99.9 % metals basis, Alfa Aesar). The tube was drawn into a 1 mm wire by the extrusion process and then pressed into tape of 0.30 mm thickness and 1.53 mm width. The tapes were then sintered at 845 ºC for 50 h and cut into 3 cm sections for $J_c$ measurements. After measurements were made for the first sintering, the tapes were re-sintered for another 50 h at 845 ºC (for a total of 100 h) and $J_c$ was measured again. Non-added tapes were fabricated for comparison.

The phase of the tapes was examined by X-ray powder diffraction using a Siemens D 5000 diffractometer with CuK$_\alpha$ radiation. The size of the MgO was confirmed by using a Philips transmission electron microscope (TEM) model CM12. The microstructure of the tapes was examined by a Philips XL 30 scanning electron microscope (SEM). The distribution of Mg in the tapes was determined by using a Philips energy dispersive X-ray analyzer (EDX) model PV99. The $J_c$ was measured by the DC four-probe method using the 1 $\mu$V/cm criterion. The $J_c$ of tapes were measured
between 30 and 77 K in self-field and under magnetic field (0 to 0.75 T) at 77 K. The dependence of $J_c$ on magnetic fields applied parallel and perpendicular to the surface of tape was investigated.

3. RESULTS AND DISCUSSION

Figure 1. TEM micrographs of nano MgO showing 70 nm average particle size

Figures 1 shows the TEM micrographs of MgO with average size 70 nm. Figure 2 shows the $J_c$ of the pellet where the optimal addition were for samples with $x = 0.15$ (50 nm) and 0.01 wt. % (70 nm). The $J_c$ of the added pellets showed a higher value (at 30 and 77 K) compared with the non-added pellet. These amounts were used to fabricate (Bi,Pb)-2223(MgO)$_x$/Ag tapes. Figure 3 shows the XRD patterns of the non-added and MgO added (Bi,Pb)-2223(MgO)$_x$ tapes sintered for 50 h ($x = 0$, 0.15 wt. % of 50 nm MgO, and 0.01 wt. % of 70 nm MgO). Most of the X-ray peaks belong to the high-$T_c$ phase (Bi,Pb-2223) along with a small amount of the low-$T_c$ phase (Bi,Pb-2212). The peaks belonging to Ag and Ca$_2$PbO$_4$ were also observed in the tapes.

Figure 4(a) shows the SEM micrograph of the non-added tape sample. SEM micrographs of the cross section of the 50 nm MgO added (Bi,Pb)-2223(MgO)$_{0.15}$/Ag tapes sintered for 50 and 100 h are shown in Figures 4(b) and 4(c), respectively. Figures 4(d) and 4(e) show the SEM micrographs of the 70 nm MgO added (Bi,Pb)-2223(MgO)$_{0.01}$/Ag tapes sintered for 50 h and 100 h, respectively. All tapes consisted of plate-like grains. Figures 4(b) and 4(d) show the distribution of Mg in the tapes (white dots). In both tapes sintered for 100 h, larger plate-like grains are observed. This shows the effect of different sintering time on the morphology of the tapes. The plate-like grains of the 70 nm MgO added tape sintered for 100 h are larger and well-connected to each other. However, the plate-like grains did not fill the space completely.
Figure 2. $J_c$ values of nano-sized MgO (50 and 70 nm) added (Bi,Pb)-2223(MgO)$_x$ pellets ($x = 0$-0.15 wt.%) as a function of MgO content at 30 and 77 K
Figure 3. XRD patterns of (Bi,Pb)-2223(MgO)$_x$/Ag tapes for $x = 0$ wt. %, 0.15 wt. % of 50 nm MgO, and $x = 0.01$ wt. % of 70 nm MgO heated for 50 h
Figure 4. SEM micrographs of (Bi,Pb)-2223(MgO)/Ag tapes (a) $x = 0$ wt. %, (b) $x = 0.15$ wt. % of 50 nm MgO sintered for 50 h, (c) 100 h, and (d) $x = 0.01$ wt. % of 70 nm MgO sintered for 50 h and (e) 100 h

Table 1. $J_c$ of $x = 0$ wt. %, 0.15 wt. % of 50 nm MgO and $x = 0.01$ wt. % of 70 nm MgO added pellets and tapes sintered for 50 h and 100 h at 30 and 77 K in zero fields.

<table>
<thead>
<tr>
<th>$x$ (wt. %)</th>
<th>$J_c$ (30 K) Pellet (A/cm²)</th>
<th>$J_c$ (77 K) Pellet (A/cm²)</th>
<th>$J_c$ (30 K) Tape (50 h) (A/cm²)</th>
<th>$J_c$ (77 K) Tape (50 h) (A/cm²)</th>
<th>$J_c$ (30 K) Tape (100 h) (A/cm²)</th>
<th>$J_c$ (77 K) Tape (100 h) (A/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0.65</td>
<td>0.31</td>
<td>8104</td>
<td>949</td>
<td>10820</td>
<td>1262</td>
</tr>
<tr>
<td>0.15</td>
<td>4.43</td>
<td>2.29</td>
<td>20460</td>
<td>2660</td>
<td>22710</td>
<td>3070</td>
</tr>
<tr>
<td>(50 nm)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0.01</td>
<td>4.49</td>
<td>2.87</td>
<td>10000</td>
<td>1330</td>
<td>15000</td>
<td>2670</td>
</tr>
<tr>
<td>(70 nm)</td>
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</table>
Table 1 shows the $J_c$ of $x = 0$, 0.15 wt. % of 50 nm MgO, and 0.01 wt. % of 70 nm MgO added pellet, and tapes sintered for 50 and 100 h at 30 and 77 K. The $J_c$ in pellet form is limited by the weak links of grain boundaries. The weak link in the tapes was greatly strengthen [4,5] and thus a higher $J_c$ was achieved.

![Graph](image_url)

**Figure 5.** $J_c$ of $x = 0$ wt. %, $x = 0.15$ wt. % of 50 nm MgO, and $x = 0.01$ wt. % of 70 nm MgO added tapes sintered for (a) 100 h and (b) 50 h as a function of the temperature
The $J_c$ of the non-added, 50 nm MgO, and 70 nm MgO added tapes sintered for 50 and 100 h as a function of temperature are shown in Figure 5. The enhanced $J_c$ of added tapes could be attributed to the increase in flux pinning strength due to the addition of nano-sized MgO [18]. The tapes sintered for 100 h exhibited a higher $J_c$ than the tapes sintered for 50 h due to the improvement in grains connectivity and also due the improvement in the grain alignment (Figure 4) when the sintering time was increased.

![Figure 6. $J_c$ of $x = 0$ wt. %, $x = 0.15$ wt. % of 50 nm MgO, and $x = 0.01$ wt. % of 70 nm MgO added tapes sintered for 50 and 100 h under magnetic field parallel (para.) and perpendicular (perp.) to the surface of the tapes at 77 K](image-url)
$J_c$ values of the tapes sintered for 50 h and 100 h under magnetic field applied parallel and perpendicular to the tape surface are shown in Figure 6. As expected, the $J_c$ in all tapes decreased with increasing magnetic fields. However, additions of nano-sized MgO enhanced $J_c$ of the added tapes under magnetic field compared with the non-added tapes. The tapes sintered for 100 h exhibited a higher $J_c$ than the tapes sintered for 50 h in magnetic field. $J_c$ of the tapes when the fields were applied parallel to the surface of the tape was higher than when the field was applied perpendicular to the surface. This was due to the better flux pinning strength along the flat surface of the tapes [19]. $J_c$ values of the 50 nm MgO added tapes showed a lower rate of decrease with increasing field compared with the 70 nm MgO added tapes. This may be due to the fact that smaller particle size and a higher amount of MgO (50 nm) can pin the flux in the (Bi,Pb)-2223(MgO)/Ag tapes better than the 70 nm MgO particles. Generally the $J_c$ of the 50 nm MgO added tapes were higher than the 20 and 40 nm MgO added tapes [11]. This work together with the results from [11] showed that $J_c$ can be optimized if the particle size is either comparable to the penetration depth [15,16] or closer to the coherence length.

4. CONCLUSION

Additions of nano-sized MgO (50 and 70 nm) into (Bi,Pb)-2223/Ag tapes enhanced the transport critical current density. The optimal sintering time for the highest $J_c$ was 100 h. MgO with size 50 nm was more effective in increasing $J_c$ (22710 and 3070 A/cm$^2$ at 30 and 77 K, respectively) compared to the 70 nm particles (15000 and 2670 A/cm$^2$ at 30 and 77 K, respectively). $J_c$ of the non-added tape sintered for 100 h was 10820 A/cm$^2$ at 30 K and 1262 A/cm$^2$ at 77 K. A combination of improved microstructure and nano MgO addition that acts as effective pinning centers enhanced the $J_c$ in this system. The $J_c$ can be increased by a more refined heating and processing method. Nevertheless, this work showed the importance of the size of the nanoparticle and heat treatment on the transport critical current density of the BSCCO tapes.

ACKNOWLEDGEMENT
This work has been supported by the Ministry of Education, Malaysia under grant no. FRGS/2/2013/SG02/UKM/01/1 and Universiti Kebangsaan Malaysia under grant no. AP-2015-006 and UKM-DPP-2013-052.

Conflict of Interest
The authors declare that there is no conflict of interests regarding the publication of this article

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