Effect of MgO and Ag$_2$O on the microstructure and superconducting properties of the (Bi,Pb)-2223 phase in the partial-melting and sintering process

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Abstract

The effect of MgO and Ag$_2$O on the microstructure and superconducting properties of the (Bi,Pb)-2223 phase during sintering after partial melting has been investigated. In the Ag/oxide interface layer, a well-aligned Bi-2223 phase was preferentially formed. Both the degree of texturing and the volume fraction of the Bi-2223 phase decreased by increasing the distance from the Ag interface. Whereas, in the MgO/oxide interface layer, although well-aligned Bi-2223 grains existed at the MgO interface, a larger volume fraction of the Bi-2223 phase appeared in the center rather than at the MgO interface. The flux pinning was not clearly improved by the addition of MgO, Ag$_2$O or PtO$_2$ particles. However, MgO addition did not affect the formation rate of (Bi,Pb)-2223 phase, and could suppress the growth of Bi-free non-superconducting secondary phases. Ag$_2$O or PtO$_2$ addition resulted in coarse secondary phases, and slowed the formation rate of the Bi-2223 phase.

Author Keywords: Bi-2223; Texture; Flux pinning; Partial-melting; Microstructure

1. Introduction

Among all the high-temperature superconductors discovered to date, Bi-2223 oxide superconductor is the most promising material for tapes and wires for high-current applications. However, critical current density (J_c) is strongly influenced by the presence of pinning defects and their possible matching with the cores of the vortex lines [1]. The defect structure is essential for flux pinning enhancement, which is in particular, the most important problem for the Bi-based superconductors [2 and 3]. In order to increase flux pinning, the Bi-2212 phase was doped by Al, Pb or rare earth elements to produce point defects [3 and 4], while in the Bi-2223 phase, Sr was partially substituted by Ba [5] or Sn [6] for the same purpose.

High T_c oxide superconductors, as with all ceramics, are very brittle and difficult to shape and handle. Since Ag does not have a significant detrimental effect on the superconducting properties of Bi-based superconductors [7], it has been successfully employed to fabricate Ag/superconductor composites, to compensate for the ceramic's brittleness. However, the influence of Ag on texture and formation of Bi-2223 phase during sintering after partial melting has not yet been investigated well. In this paper, we report results on the effect of MgO, Ag_2O, PtO_2 additions and Ag, MgO substrates on the microstructure and superconducting properties of the (Bi,Pb)-2223 oxide superconductor in the partial-melting and sintering process.

2. Experiment details

High purity Bi_2O, PbO, $SrCO_3$, $CaCO_3$ and CuO powders (99.99% in purity) were weighed and mixed in the atomic ratio Bi:Pb:Sr:Ca:Cu=1.8:0.4:1.9:2.1:3.5, which is the optimum

composition for the preparation of (Bi,Pb)–2223 in the partial–melting and sintering process in our previous experiment [8]. Then, 5 wt.% MgO, Ag_2O or PtO_2 particles with an average size of 0.5 μm were added to this optimum composition, respectively. After being calcined at 800° C for 12 h and pulverized, samples were made by cold pressing a pellet to the size of $2 \times 3 \times 15$ mm^3. These bulk samples, which were set on a Ag or MgO substrate, were partially melted in air at 875° C for 1 h, and then furnace–cooled or air–cooled to room temperature, and finally sintered at 840° C for 240 h. In this work, a specific method for the preparation of the Ag–sheathed tapes was used. First, the bulk samples were cut into $1 \times 1 \times 15$ mm^3 sizes, and then wrapped with six rotations of 0.1–mm–thick Ag sheets. The wrapped samples were rolled to tapes with an overall thickness of 0.2 mm and a width of 3 mm. Finally, the resultant tapes were sintered at 840° C for 240 h. The microstructure and pinning properties of Ag–sheathed (Bi,Pb)–2223 tapes were studied by means of XRD, SEM, optical microscope, AC susceptibility and J_c measurements. The degree of texturing of Bi–2223 grains in this study is quantified by an alignment factor f defined as

$$f=(P-P_o)/(1-P_o) \tag{1}$$

$$Q=H(0010)/(H(0010)+H(1115)), \tag{2}$$

where P is a parameter as stated above in the textured sample and P_o is an equivalent parameter for random samples. The volume fraction (Q) of the Bi–2223 phase is defined as

$$Q=H(0010)/(H(0010)+L(008)), \tag{3}$$

where H(0010) and (115) are the integrals of the (0010) and (115) peaks of the Bi–2223 phase, respectively, and L(008) is the integral of the (008) peak of the Bi–2212 phase.

3. Results and discussion

Fig. 1 shows the dependence of the alignment factor of the Bi−2223 phase on the distance from both the Ag and MgO interfaces. A well−aligned Bi−2223 phase exists at the Ag and MgO interfaces, and their alignment factors decrease with increasing distance from the interfaces
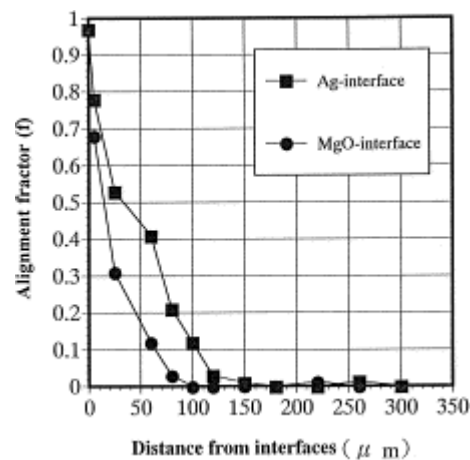

Fig. 1. Dependence of the alignment factor of the Bi−2223 phase on the distance from both the Ag and MgO interfaces.

Fig. 2 shows the volume fraction of the Bi−2223 dependence on the distance from both the Ag and MgO interfaces. It is clear that the highest volume fraction of the Bi−2223 phase exists in the Ag interface layer, and the volume fraction decreases with increasing distance from the Ag interface. In the MgO interface layer, although well−aligned Bi−2223 grains exist at the MgO interface, a larger volume fraction of the Bi−2223 phase appears in the center rather than at MgO interface. These results indicate that the Bi−2223 phase in the partial−melting and sintering process has preferentially formed along the Ag substrate.
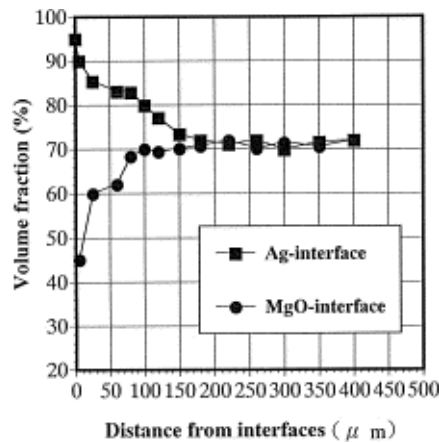
Fig. 2. Dependence of the volume fraction of the Bi−2223 phase on the distance from both the Ag and MgO interfaces.

Fig. 3 shows the critical current densities (J_c) of tapes at 77 K and zero field. It can be seen that the J_c of the undoped tape is the highest among all the tapes made from the air−cooled bulk samples. Whereas, the J_c of the MgO−doped tape is the highest among all the tapes made from the furnace−cooled bulk samples. The tapes with Ag_2O or PtO_2 additions have quite low J_c values.
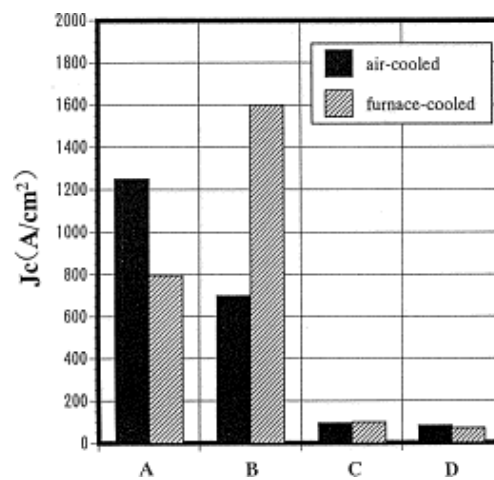

Fig. 3. The critical current densities of tapes at 77 K and zero field, A: undoped, B: MgO−doped, C: Ag_2O−doped, D: PtO_2−doped.

The applied magnetic field dependence of the normalized critical current density at 77 K for the MgO−doped and undoped tapes made from the furnace−cooled bulk samples is plotted in Fig. 4. The J_c−magnetic field characteristic at 77 K is not clearly improved by the MgO addition. The J_c measurements at 4.2 K show a similar result as stated above.
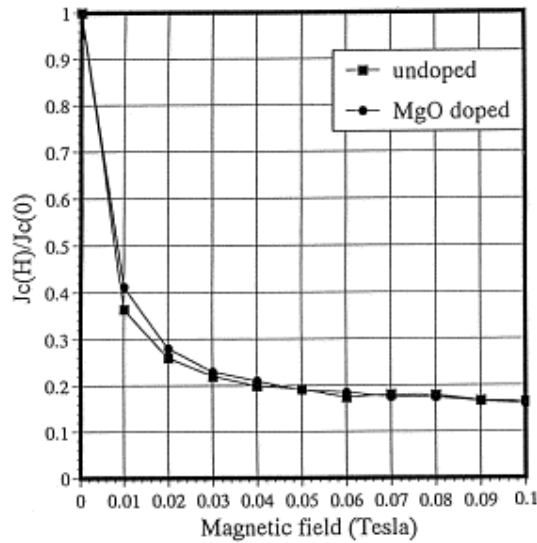

Fig. 4. Normalized J_c versus magnetic field curves at 77 K for tapes with and without MgO addition.

Fig. 5 shows the AC susceptibility plots for bulk samples sintered at 840° C for 240 h after being partially melted at 875° C for 1 h. Although the susceptibility changes at 108 K due to the diamagnetism of the Bi−2223 phase are observed in all samples, the diamagnetism of the Bi−2223 phase at the MgO−doped and undoped samples is much larger than those at the Ag_2O− and PtO_2−doped samples. This indicates that MgO addition does not affect the formation rate of the Bi−2223 phase; whereas, Ag_2O and PtO_2 additions slow the formation rate of the Bi−2223 phase.
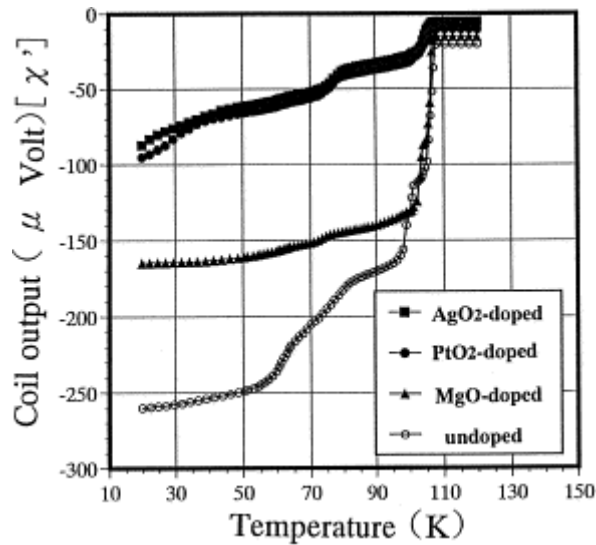
Fig. 5. The AC susceptibility plots for bulk samples sintered at 840° C for 240 h after partial melting at 875° C for 1 h.

In order to clarify the reason why the formation rate of the Bi–2223 phase was affected by the MgO, Ag_2O and PtO_2 additions, we observed the microstructure of the air–cooled samples before sintering at 840° C, as shown in Fig. 6. The microstructures of the undoped and MgO–doped samples mainly consist of the Bi–2201 phase, $(Sr,Ca)_2CuO_3$ and $(Sr,Ca)CuO_2$. The relative amount of $(Sr,Ca)_2CuO_3$ is nearly equal to that of $(Sr,Ca)CuO_2$. Whereas, the microstructures of the Ag_2O– and PtO_2–doped samples have large $(Sr,Ca)_2CuO_3$ and $(Sr,Ca)CuO_2$ crystals. The relative amount of $(Sr,Ca)CuO_2$ is much larger than that of $(Sr,Ca)_2CuO_3$. In our earlier research [8], we proved a phase content with equal amounts of $(Sr,Ca)_2CuO_3$ and $(Sr,Ca)CuO_2$ before sintering is favorable for the formation of the Bi–2223 phase. Therefore, it may be concluded that the Ag_2O and PtO_2 additions result in unfavorable variations of $(Sr,Ca)_2CuO_3$ and $(Sr,Ca)CuO_2$ before sintering, thus slow the formation rate of the Bi–2223 phase. Furthermore, it is more difficult to produce the Bi–2223 phase from the microstructures with large $(Sr,Ca)_2CuO_3$ and $(Sr,Ca)CuO_2$ crystals in the Ag_2O– and PtO_2–doped samples before sintering.
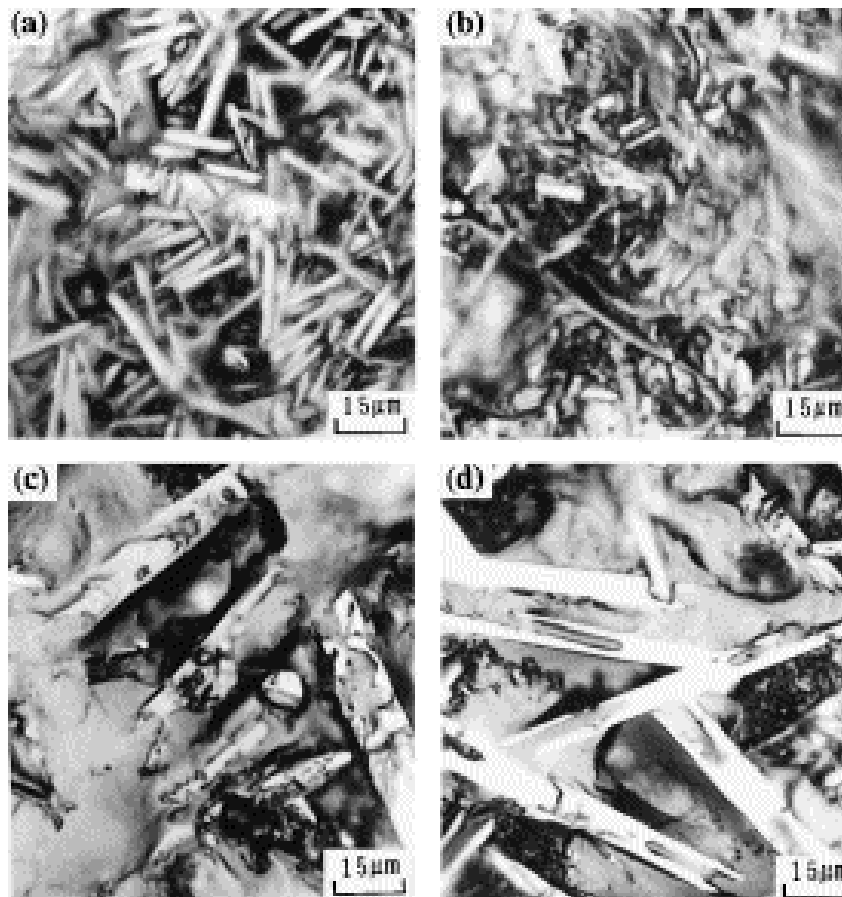
Fig. 6. Optical micrographs of free surface of samples heated at 875° C for 20 min and then air-cooled to room temperature: (a) undoped, (b) MgO-doped, (c) Ag$_2$O-doped, (d) PtO$_2$-doped.

Fig. 7 shows SEM micrographs for the transverse cross-sections of tape samples. By comparison with the undoped sample (Fig. 7a), the MgO-doped sample (Fig. 7b) has small secondary phases; whereas, the Ag$_2$O- and PtO$_2$-doped samples (Fig. 7c and 7d) have large Bi-free non-superconducting secondary phases. It is suggested that MgO addition can suppress the growth of Bi-free non-superconducting secondary phases, but Ag$_2$O and PtO$_2$ additions result in coarse secondary phases.
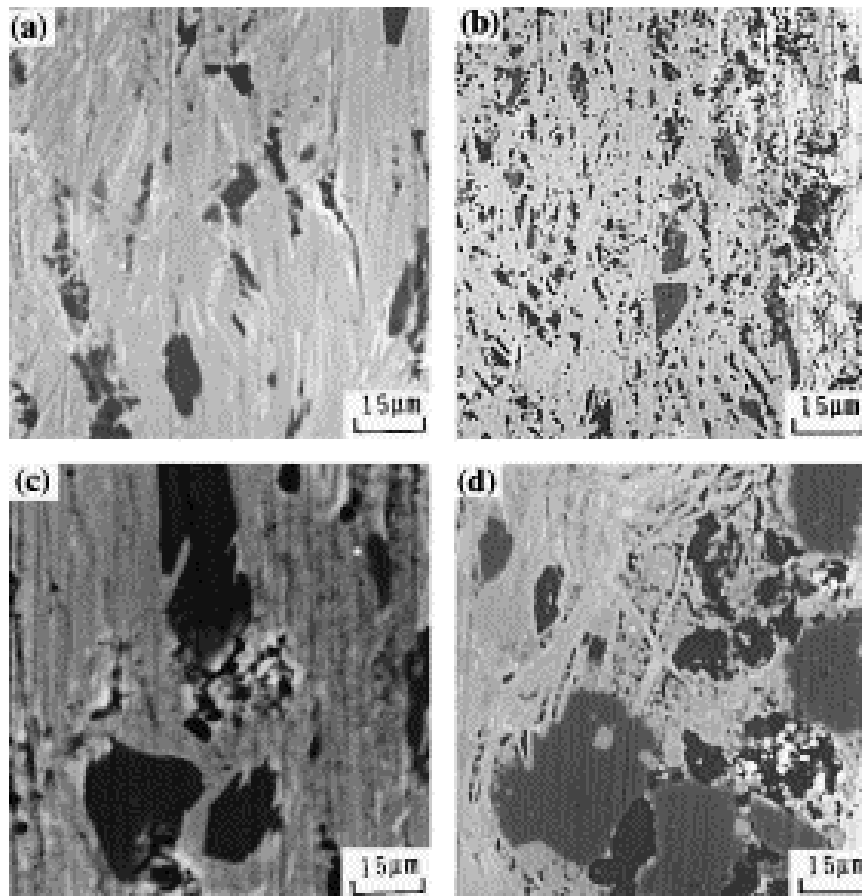
Fig. 7. SEM micrographs for the transvere cross-sections of tape samples: (a) undoped, (b) MgO-doped, (c) Ag$_2$O-doped,(d) PtO$_2$-doped.

4. Conclusions

The effect of MgO, Ag$_2$O, and PtO$_2$ particles, and Ag and MgO substrates on the microstructure and superconducting properties of the (Bi,Pb)-2223 phase during sintering after partial melting was studied. In the Ag/oxide interface layer, a well-aligned Bi-2223 phase was preferentially formed. Both the degree of texturing and the volume fraction of the Bi-2223 phase decreased by increasing the distance from the Ag interface. Whereas, in

the MgO/oxide interface layer, although well-aligned Bi-2223 grains existed at the MgO interface, a larger volume fraction of the Bi-2223 phase appeared in the center rather than at the MgO interface. The flux pinning was not clearly improved by the addition of MgO, Ag_2O or PtO_2 particles. However, MgO addition did not affect the formation rate of the (Bi,Pb)-2223 phase, and could suppress the growth of Bi-free non-superconducting secondary phases. Ag_2O or PtO_2 addition resulted in coarse secondary phases, and slowed the formation rate of the Bi-2223 phase.

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