

## Fabrication of Bi-2212/SrSO<sub>4</sub> Composite Superconductors by Melting Powder Mixtures

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### Abstract

*We fabricated Bi-2212/SrSO<sub>4</sub> composite superconductors and evaluated the effects of the powder mixing method and melting temperature on their microstructure and superconducting properties. The Bi-2212 powders were mixed with SrSO<sub>4</sub> by hand-mixing (HM) and planetary ball milling (PBM) and then the powder mixtures were melted at 1100 °C~1200 °C, solidified, and annealed. We found that the powder mixture prepared by PBM was finer and more homogeneously mixed than that prepared by HM, resulting in more homogeneous microstructure and smaller SrSO<sub>4</sub> and second phases after annealing.*

**Keywords :** Bi-2212, SrSO<sub>4</sub>, planetary ball milling, transition temperature, critical current

### 1. Introduction

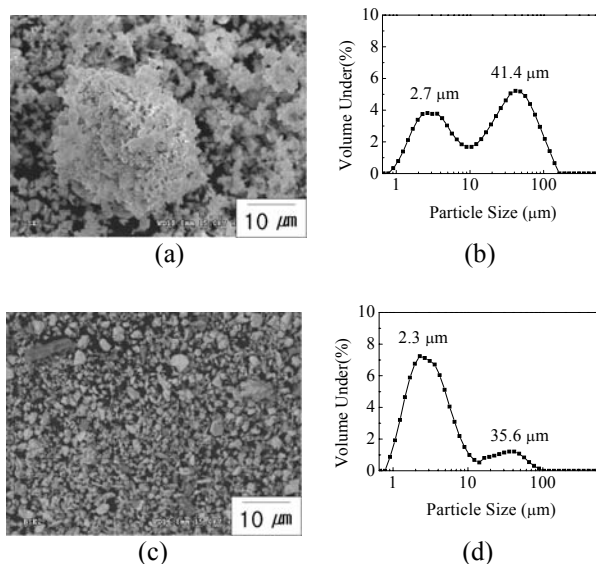
The melt-casting process (MCP) has been widely used to obtain 2212-SrSO<sub>4</sub> composite superconductors [1]. In the fabrication of 2212-SrSO<sub>4</sub>, the nonuniform microstructure and segregation of SrSO<sub>4</sub> phases can occur due to the difference in the density, melting point, and powder conditions between 2212 and SrSO<sub>4</sub>. To improve the homogeneity of the melt, it is necessary to optimize the powder conditions together with the melting temperature, in order to allow a uniform microstructure and excellent critical properties to be obtained.

### 2. Experimental and Results

Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>1</sub>Cu<sub>2</sub>O<sub>8+δ</sub> (2212) and 10wt.% SrSO<sub>4</sub> powders were mixed by HM and PBM. In the HM process, the two powders were put into a jar and stirred for 20 minutes. In the PBM process, the powders were loaded into a zirconia jar along with zirconia balls and isopropyl alcohol and planetary ball-milled for 4 hours. The mixtures were heated at two different temperatures, viz. 1100 and 1200 °C, for 10 min in an alumina crucible. Subsequently, the melt was poured into a quartz-tube mold and cooled to room temperature. The ingot was heat treated at 800 °C for 120 hr in an oxygen atmosphere to form the 2212 phase.

Figures 1 shows the SEM micrographs of the HM- and PBM-powders and their size distributions. The morphology of the HM-powder seemed to be inhomogeneous and large agglomerates were present. The PBM process reduced the

powder size slightly, moreover, it effectively eliminated/reduced the agglomerates. It is suggested that the finer and more homogenous powder mixtures produced by PBM enhances their interaction and leads a more uniform microstructure, as compared to those produced by HM.



**Fig. 1. The SEM micrographs of the (a) HM-powder and (c) PBM-powder and their size distributions (b) and (d)**

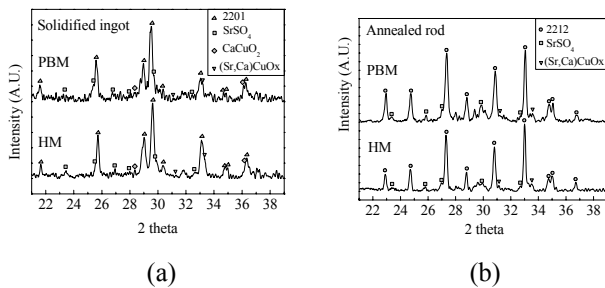


Fig. 2. The XRD patterns of the (a) ingots and (b) annealed rods prepared by melting at 1200 °C

Figure 2 shows the X-ray diffraction (XRD) patterns of the ingot and annealed rod prepared by melting at 1200 °C. Both the HM- and PBM-ingots had 2201 as the major phase and SrSO<sub>4</sub>, (Sr,Ca)CuO<sub>x</sub>, and CaCuO<sub>2</sub> as minor phases. After annealing, the 2201 phase disappeared and the 2212 phase became the major one, suggesting that the 2201 reacted with the Ca- and Cu-rich phases to form the 2212 phase during the heat treatment in an oxygen atmosphere [2]. It is to be noted that the XRD patterns did not show any phase difference between the specimens prepared by the HM and PBM methods.

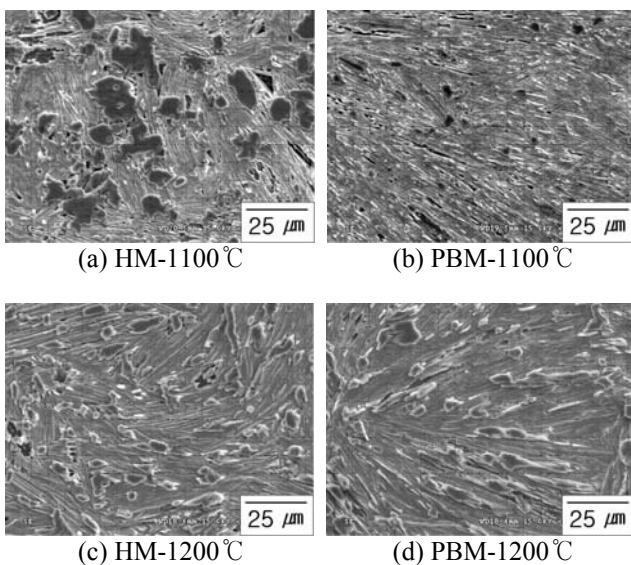


Fig. 3. The SEM micrographs of the rods by (a) HM and (b) PBM melted at 1100 °C and (c) HM, (d) PBM melted at 1200 °C.

According to the EPMA analysis, the black and dark gray particles in figure 3 are Ca-rich second phase and SrSO<sub>4</sub> precipitates, respectively, and the plate-like light gray grains are 2212 phase. For the HM-rod melted at 1100 °C (Fig. 3(a)), the 2212 grains were less textured, and larger SrSO<sub>4</sub> and other second phases were present, which were much more segregated than those in the PBM-rod (Fig 3(b)).

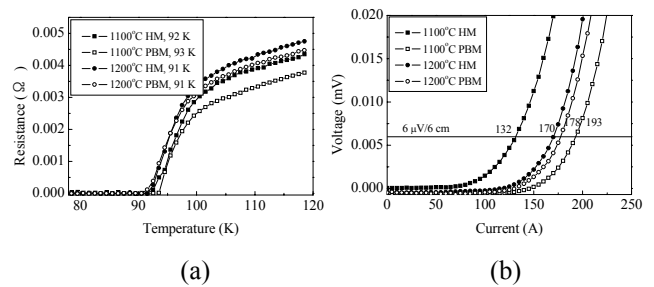


Fig. 4. Dependence of (a)  $T_c$  and (b)  $I_c$  of the rods on mixing methods and melting temperatures.

Figure 4 shows the  $T_c$  and  $I_c$  of the annealed rods. The  $T_c$  was measured to be in the range of 92–93 K for both rods prepared at the melting temperature of 1100 °C. At the melting temperature of 1200 °C, the  $T_c$  was slightly decreased to 91 K for both of the rods. According to the DTA/TG analysis, both powders began to volatilize at approximately 1200 °C and the weight loss increased drastically at ~1224 °C for the PBM-powder and at ~1261 °C for the HM-powder. On the other hand, the  $I_c$  was significantly dependent on both the mixing method and melting temperature. The improvement in the  $I_c$  value of the HM-rod melted at 1200 °C seems to be due to its more homogeneous microstructure, as shown in Fig. 3.

### 3. Summary

It was observed that the morphology of the powder mixture varied significantly with the mixing method. The PBM method allowed a more homogeneous mixture of the powders to be obtained, and thus PBM-rod had a more homogeneous microstructure and smaller SrSO<sub>4</sub> and second phases, which is probably due to its powder conditions. The  $I_c$  of the PBM-rod was higher than that of the HM-rod melted at the same temperature and this improvement in the  $I_c$  value of the PBM-rod is considered to be due to its more uniform microstructure.

### 4. References

1. J. Bock, H. Bestgen, S. Elchner and E. Preisler: IEEE Trans. Appl. Supercond. Vol. 3 (1993), p. 1659.
2. J. Bock and E. Preisler: Solid State Commun. Vol. 72 (1989), p. 453.