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The influence of process variables of precursor powders on the microstructure evolution and transport current properties of Bi-2223/Ag tapes

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Abstract

The effect of Pb content and particle size of precursor powders has been studied to improve the grain growth and texture of Bi-2223/Ag tapes. The Pb content and particle size of precursor powders dominate the amount of liquid phases, and thus affect the value of J_c with a variation of heat treatment condition. The best values of J_c reached 53 kA/cm² at 77 K and 0 T, after 210 h heat treatment at 839 °C and two times intermediate pressing. It suggests that the improvement of J_c characterized from high fraction of Bi-2223 with enlarged crystallites is attainable by optimizing the processing variables of precursor powders.

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Keywords: Bi-2223; Bi-2212; Pb content; Particle size; BSCCO wires and tapes

1. Introduction

Recently, interest in the long length silver sheathed Bi-2223 superconducting wires has been increasing along with increase of practical demand for the prototype power application [1,2]. The stringent requirements for Bi-2223 superconductor applicable for practical use strongly need to improve the critical current density J_c , which conse-

quently demands to optimize process variables of precursor powder for microstructural control.

Among the many process variables, Pb content and particle size of precursor powders are regarded as the most important factors for dominating the microstructure evolution and J_c in the Bi-2223 wire. These variables closely correlate with each other and influence J_c by either accelerating or counterbalancing the amounts of liquid phase. Our previous work shows a tendency that tapes made from a precursor powder with lower Pb content and smaller particle size have a higher J_c [3]. For a systematic study, we are mainly concerned with the precursor powder with lower Pb

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content ($x = 0.2, 0.3$) and smaller particle size (2 and 4 μm) in the present work.

2. Experimental

Various precursor powders were fabricated with different Pb content of $\text{Bi}_{1.8}\text{Pb}_x\text{Sr}_2\text{Ca}_{2.2}\text{Cu}_3\text{O}_y$ ($x = 0.2, 0.25, 0.3, 0.33, 0.36, 0.38$ and 0.4) by the spray-drying method. The powders were sequentially calcined at 800 °C for 2 h, and then 830 °C for 15 h two times. Powders were milled to have an average particle size of 2 and 4 μm . The final carbon content of precursor powders was less than 50 ppm. The phases in the precursor powders were analyzed by using XRD. Differential thermal analysis (DTA) for the precursor powders was carried out. The well-established powder-in-tube technique was used to fabricate the 61 filament superconducting tapes. The green tapes fabricated from the precursor powders were about 3.0 mm wide and 280 μm thick or so, and the fill factor of superconductor was around 12%. The J_c was measured at 77 K and zero fields by the standard four-probe technique with a criterion of 1 $\mu\text{V}/\text{cm}$. Correlation between transport current property and microstructure for the tape samples was investigated by using XRD.

3. Result and discussion

Fig. 1 shows the X-ray diffraction patterns for final calcined powders with different Pb content. All powders consist of Bi-2212 as the major phase and some minor phases such as Bi-2201 and Ca_2PbO_4 . The main difference between these powders is the Ca_2PbO_4 content. Comparing these patterns with each other, it is obvious that the peak intensity of Ca_2PbO_4 ($2\theta = 17.8^\circ$ and 32°) for Pb = 0.4 is relatively higher than for Pb = 0.2. The DTA patterns of the precursor powders with the different Pb content (Pb = 0.2 and 0.3) are shown in Fig. 2. It is apparent that two endothermic peaks are partially overlapped in the range of 850–900 °C. The broad peak is attributed to the melting of the Bi-2212 phase, while the small one is attributed to the decomposition of the Ca_2PbO_4 [4,5]. The

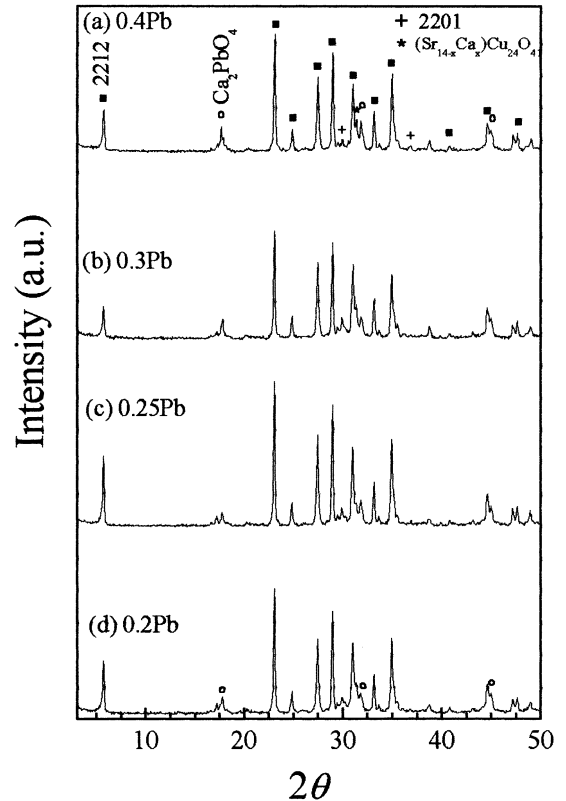


Fig. 1. XRD patterns for the final calcined powder of (a) Pb = 0.4, (b) Pb = 0.3, (c) Pb = 0.25, (d) Pb = 0.20.

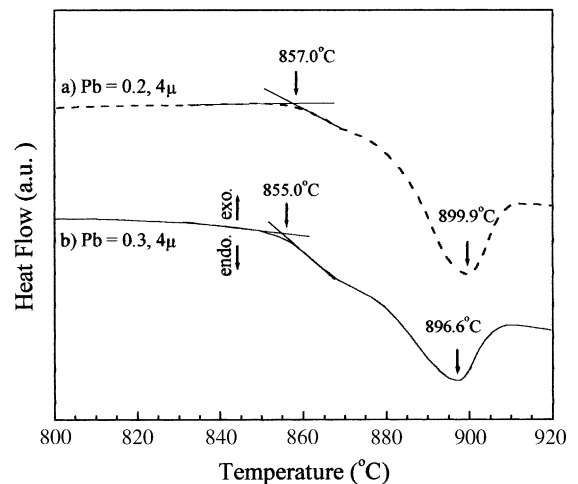


Fig. 2. DTA curves for the different starting precursor powders with (a) Pb = 0.2, (b) Pb = 0.3.

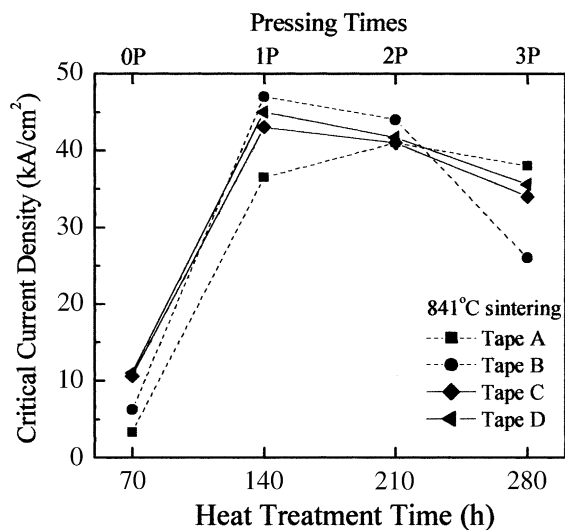


Fig. 3. Variation of critical current density with respect to heat treatment at 841 °C and multiple pressing. Tape A, B, C, D were fabricated from the powder with (a) Pb = 0.2, 2 μm (b) Pb = 0.2, 4 μm , (c) Pb = 0.3, 2 μm , (d) Pb = 0.3, 4 μm .

powder sample with higher Pb content shows the lower onset temperature (T_{onset}) and melting temperature (T_{m}). On the other hand, little difference was observed between the powders with the same composition but different particle size.

Fig. 3 shows the variation of J_{c} with respect to multiple pressing and heat treatment at 841 °C for the tapes. The sintering temperature was chosen consistent with the previous work [3]. The comparison between the tapes heated 140 h with one intermediate pressing step shows that for the same Pb content, the larger particle size has a little higher J_{c} . This may be related with the amount of liquid phase formed in the early stage of the heat treatment. It has been suggested that too fine a powder may result in deteriorated J_{c} due to the formation of large second particles [6]. The J_{c} in the Fig. 3 almost reaches a maximum after heat treatments for a total time of 140 h with one intermediate pressing step, and then, begins to drop with further pressing and heat treatment. The decrease in J_{c} is attributed to unhealed cracks introduced by mechanical deformation [7].

Sintering at 839 °C was carried out for the tape made for the powder with Pb = 0.3, 4 μm (tape D).

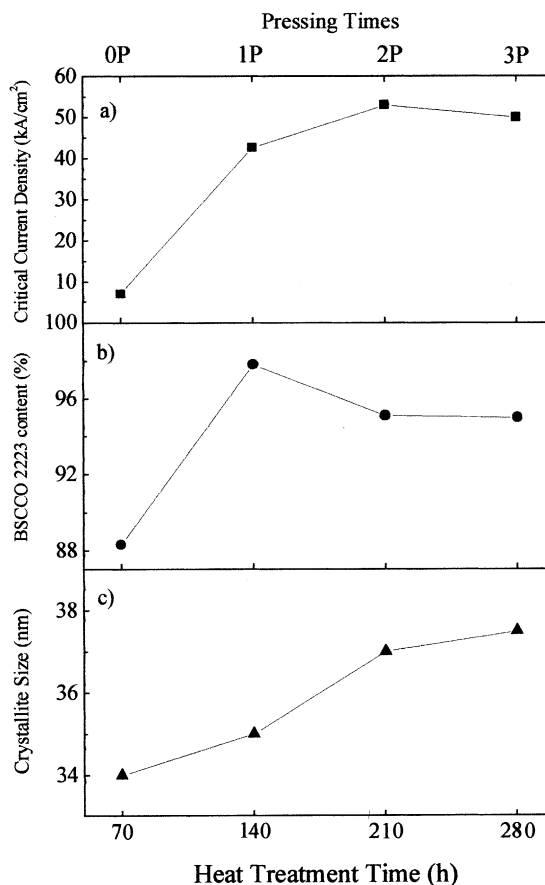


Fig. 4. (a) Critical current density, (b) Bi-2223 conversion rate and (c) crystallite size of tape D with respect to heat treatment at 839 °C and multiple pressing.

Tape D is chosen because it shows better J_{c} over the whole range of heat treatment time as sintered at 841 °C. Fig. 4(a) shows the variation of J_{c} of tape D with respect to sintering at 839 °C and multiple pressing. J_{c} does not reach its maximum of 53 kA/cm² until total time of 210 h and with two intermediate pressings. Besides, the maximum value of J_{c} was definitely improved as compared with those of 841 °C shown in Fig. 3. Fig. 4(b) shows that the Bi-2223 fraction calculated on the basis of the XRD intensity ratio of the (008)₂₂₁₂ and (0010)₂₂₂₃ peaks [8]. The Bi-2223 fraction already reached saturated value, above 95% after only 140 h heat treatment and one time pressing. Fig. 4(c) shows crystallite size determined from the

peak for the (0 0 1 4) reflection of 2223 by using the Scherrer formula [9]. The estimated crystallite size increases with increasing sintering time consistent with the observed increase in J_c . These results suggest that J_c improves as a result of higher fraction of Bi-2223 with enlarged crystallite size.

4. Conclusion

DTA and XRD results indicate that Pb content of the powder influences the amount of Ca_2PbO_4 , and then consequently J_c . For the same Pb content, the tape with the larger particle size has the higher J_c . The best value of J_c , 53 kA/cm² was obtained at 77 K and 0 T, after 210 h heat treatment at 839 °C and two times intermediate pressing. It suggests that the great improvement of J_c characterized from the high fraction of Bi-2223

with enlarged crystallites is attainable by optimizing the processing variables of precursor powders.

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