The effects of starting precursor conditions on the phase formation and transport properties of Bi-2223/Ag tapes

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Abstract - The influence of starting precursor powders on the phase formation and transport properties of Bi-2223/Ag tapes has been studied. The experimental results show that the average particle size of precursors as fine as 1.64 ㎛ and 1.51 ㎛ can still increase the transport properties. The J_c-B behavior is also enhanced in tapes fabricated with powders in finer particle sizes. However, at higher magnetic fields, J_c of tape started from the powder wit고 the finest particles drops rapidly at the direction of H//*c*, which is possibly attributed to the small grain sizes and weak flux pinning ability that due to the short induction period at the initial stage of phase formation as the result of fine particles in precursor powder.

1. Introduction

Improving the J_c properties of Bi2223/Ag tapes by optimizing the manufacturing parameters is still a research focus of many groups. As a proceeding step of PIT process, starting precursor powder has been proven of vital importance to the J_c of Bi-2223/Ag tapes. Many works on the influence of phase assemblage, carbon content, lead variations, and particle size distributions of precursors have been studied [1-9]. Among these aspects, particle size distribution is thought of a sensitive factor for the microstructure evolution and transport properties of Bi-2223/Ag tapes. Fine and homogenously distributed particles in precursor powders have been proven of benefits for getting uniform microstructure and high transport critical current density (J_c). Jiang et al. [8] suggested that too fine particles would induce large non-superconducting secondary phases among the ceramic matrices of 110K phase, which was detrimental to the J_c and flux pinning properties. In this paper, the effects of average particles of precursor powders larger and smaller than 2 ㎛ on the J_c and J_c-B behaviors of Bi-2223/tapes have been studied. The results suggest that finer particles less than 2 ㎛ are still beneficial to the J_c improvement.

2. Experimental

Precursor powder with nominal composition of $(BiPb)_2Sr_{2.0}Ca_{2.2}Cu_3O_x$ was prepared by spray drying method. After multiple calcinations, the powder was divided into 3 batches, which were subjected to different ball milling durations, marked as P-A, P-B, and P-C, respectively. The particle size distributions were examined using the Helos particle analyzer (Dry method) combined with SEM observations. Thereafter, the above powders were made into 61 filaments Bi-2223/Ag tapes using the standard PIT method with repeat drawing and rolling steps. The resultant green tapes, which are of 4 mm wide and 350 um thick, are denoted as T-A, T-B and T-C, respectively, according to the starting powders. Multiple sintering and pressing circles were performed at 843°C and 2 Gpa respectively to get the final high T_c superconducting tapes. I_c was measured by the standard DC four-probe method with a criterion of 1μV/cm in both 0 T and external fields. The 2223 phase percentage was calculated on the intensities of $(0\ 0\ 8)_{2212}$ and $(0\ 0\ 10)_{2223}$ from the XRD measurement [10].

3. Results and discussions

The detail particle size distributions of various ball milled powders are listed in Table 1. With the increasing of grinding time, both the average particle size and particle size distribution width are decreased.

Table 1. Particle size distribution of various ball milled powders

Sample	P-A	P-B	P-C
Average particle size	2.34㎛	1.64㎛	1.51㎛

The variation of 2223 phase content with respect to the accumulative sintering time is present in Fig.1. Obviously, the formation rate of 2223 phase in T-C is more rapid at the initial stage of heat treatment. This means that finer and shallow distributed particle size in precursor can promote the phase formation. As suggested by other authors [7,8], fine particles are characterized by high surface energy and lower melting points, which were thought able to improve the powder reactivity, shorten the induction period of the nucleation, hence accelerate the formation of 2223 phase in tapes. The present result supports their suggestions.

The I_c values in different tapes after each sintering period is shown in Fig.2. With the elongation of grinding time, I_c in T-B and T-C increase steadily among all the sintering stages. The highest I_c, 62A, is achieved in T-C after 210 h sintering and twice pressing. Since only the grinding durations on precursors are different between these samples, the enhancements of transport critical currents in T-B and T-C are directly related to the finer and homogenously distributed particle sizes in starting precursors. That is to say, average particle sizes as fine as 1.64 ㎛ and 1.51 ㎛ in precursors can still improve the transport properties of Bi-2223/Ag tapes. It is not well consistent with the results of Jiang et al. [8] and Kim et al.[9]. In their studies, powders with average particle sizes less than 2 ㎛

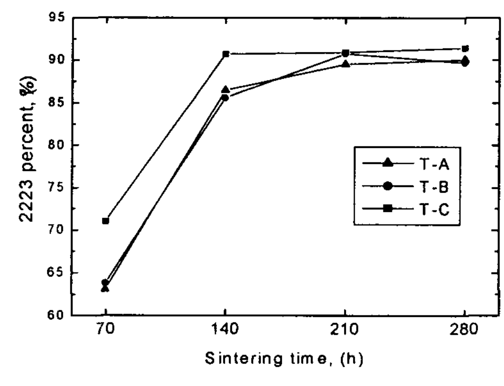

Fig. 1. Variation of 2223 phase percentage as a function of sintering time.

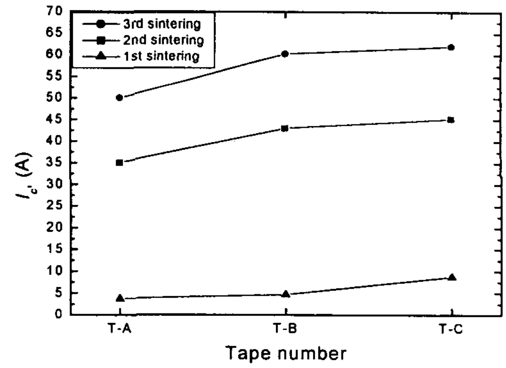

Fig. 2. I_c versus the tape number after each sintering step.

resulted in very low J_c values. It is probably due to the different preparing routes, starting materials and measuring methods of particle sizes between different groups. The powders prepared by spray drying method are high reactive comparing with the ones made from other routes.

Fig.3 plotted the normalized J_c variations for the fully processed tapes in different magnetic fields with H parallel to c-axis and ab plane. At low fields (<0.03~0.05T), J_c in T-B and T-C are enhanced in both directions. J_c is suggested be controlled by the weak links of superconducting grains at low fields [11,12]. That means the grain connectivity of T-B and T-C have been improved due to the finer particle sizes of precursor powders. J_c of T-C always shows the best performance along H//ab, but it decreases rapidly along H//c at B larger than 0.05T. At high fields, the J_c are

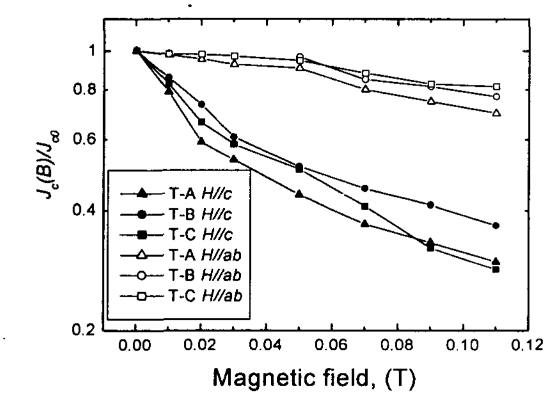

Fig. 3. The normalized Jc (***B***) curves for fully processed tapes T-A, T-B and T-C at 77K in the magnetic fields with H parallel to c-axis and ab plane

mainly governed by flux pinning for the direction of H//*c*, but by both flux pinning and grain connectivity for the direction of H//*ab* plane. Thus we can conclude that both the grain connectivity and flux pinning in T-C along H//*ab* have been improved, while the flux pinning ability along H//*c* is still need to be improved.

The SEM micrographs from polished and etched longitudinal cross-section of final tapes are illustrated in Fig.4. The microstructures of T-B and T-C are characterized by high core density and better grain alignment, which is directly responsible for the high J_c in T-B and T-C. However, the average grain sizes in T-C are obviously smaller than that of T-A and T-B. As suggested by Kim et al. [13], the very fast formation of 2223 phase at the initial stage of sintering would result in large amount of 2223 nuclei, which would impinge on each other impede the further growth of 2223 grains, thus result in small particle sizes in final tapes. Our results also show that short induction period of the nucleation of 2223 phase is unexpected for the grain growth.
Fig.4, SEM micrographs from polished and etched longitudinal cross-section of fully The weak flux pinning ability along H//*c* in T-C is also possibly caused by the large amount of grain boundaries.

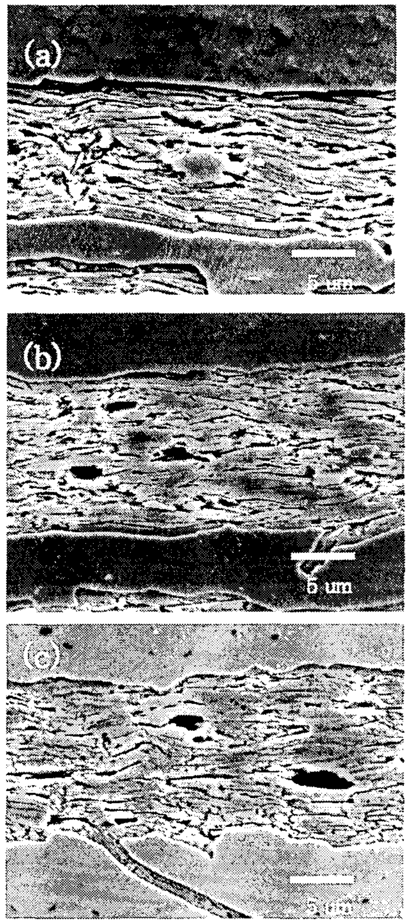

Fig. 4, SEM micrographs from polished and etched longitudinal cross-section of fully processed samples (a) T-A, (b) T-B, and (C)T-C.

4. Conclusion

In this study, the effect of starting precursor powders on the phase formation, microstructure and transport properties of Bi-2223/Ag tapes have been studied. Powders with fine and homogeneously distributed particles lead to dense ceramic core, better grain alignment and connectivity as well as high J_c values in Bi-2223/Ag tapes. Very fine particles results in a fast phase formation at the initial stage of heat treatment, but also results in small grains in final tapes, thus weaken the flux pinning ability.

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