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Correlation of the 2223 percentage before the first intermediate pressing and the transport property of the fully processed Bi-2223/Ag tapes

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Abstract

Two kinds of multifilament Bi-2223/Ag tapes, which are different in the precursor calcination temperatures, were heat treated for different time (12, 20, 30, 50, 70, or 100 h) firstly to obtain varied 2223 contents, and then followed by the same pressing and sintering cycles. The relation of the 2223 phase contents after the first sintering and the transport property of the fully processed tapes was studied. The results show that 75-80% 2223 phase formed in tapes before the first cold pressing is beneficial to get a high I_c in the final tapes. Compensating the total heat treatment time of the tapes first sintered for 20 h to the same length as that first sintered for 50 h in the subsequent sintering stages, different I_c enhancements were observed in these two tapes. No improvement on I_c was found in the tape made from the powder calcined at higher temperature, whereas for the tape prepared with the lower temperature calcined powder, the I_c was increased to the same level as that first sintered for 50 h. The 2223 contents before the intermediate mechanical work is related to the residual reactants, especially to the liquid phase, which is of vital importance to the phase conversion and healing microcracks, meanwhile, to the size and distribution of the non-superconducting secondary phases. The lower temperature calcined powder resulted in slow fomation of 2223 phase, but also provided more reactants and liquid phase for the further phase conversion, as a consequence, for the improvement of I_c.

Keywords : Bi-2223/Ag tapes, Sintering time, Phase formation, Critical current density

1. Introduction

Bi-2223/Ag tapes prepared by the powder in tube (PIT) method are still the most promising superconductor for applications [1,2]. The PIT process includes a number of complex parameters, such as powder process, mechanical deformation and heat treatments. Among these variables, the first sintering and the subsequent intermediate deformation have been proven of vital importance to the core density, grain connectivity and texture, phase assembly, and critical current density of fully processed tapes [3,4]. The 2223 phase matrix and main phase assembly have formed after the first sintering. The aim of the intermediate deformation is to break up the sintered structure and bring the reactants into more intimate contact, hence improve the core density and facilitate a complete conversion from precursor powder to 2223 [5]. However, the optimal sintering stage (2223 phase content) at which the intermediate deformation should be applied has not yet been well established. Jiang et al. [6] suggested that a low conversion rate of 2212 to 2223 before intermediate deformation was necessary to get high J_c values. Kovac et al. [3] reported that rolling

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the tape with 50% 2223 phase resulted in the highest J_c. In contrary, as much as 2223 content before pressing was recommended by Xia et al. [7]. In this study, in order to find out the optimal 2223 phase content before deformation and especially to get a high purity 2223 phase and a high degree *c*-axis texture in the final processed tapes, a set of sintering time were performed during the first heat treatment. The effect of the 2223 phase content before the intermediate deformation and the total sintering time on the transport property of the fully reacted tapes was studied.

2. Experimental details

Two differently processed precursor powders, i.e., calcined at a higher temperature, named as powder P-H, and calcined at a lower temperature, named as powder P-L, were used to fabricate 61 filaments Bi-2223/Ag tapes using the well established powder in tube (PIT) method, and referred as tapes T-H and T-L, respectively. Both precursor powders have the same nominal composition of $Bi_{1.8}Pb_{0.25}Sr_{2.0}Ca_{2.2}Cu_{3.0}O_z$. Both green tapes have the same dimension of 350 m thick and 4 mm wide. A set of dwell time, i.e., 12, 20, 30, 50, 70, and 100 h, were used to perform the first heat treatment at 843℃ in air. The other sintering periods were fixed to 70 h. The intermediate uniaxial pressing was done under a pressure of 2 GPa between every two sintering periods. The detailed sintering schedule was listed in Table I. To investigate the influence of the total sintering time, a 100 h heat treatment was performed in the second or third sintering stage for the tapes first sintered for 20 h, hence maintained the same total sintering time as that first sintered for 50 h. After every heat treatment, the transport critical current I_c was measured using the standard DC four-probe method with a criterion of 1 μV/cm at 77 K and 0 T. The 2223 phase contents were calculated on the basis of the XRD intensity ratios of the $(0\ 0\ 8)_{2212}$ and the $(0\ 0\ 10)_{2223}$ peaks. The longitudinal cross sections of reacted tapes were examined using SEM and EDX.

3. Results and discussion

XRD patterns of precursor powders P-H and P-L are shown in Fig.1. Due to the different calcination conditions, these two precursors show varied phase assemblages. In powder P-H, most of the precursor has been converted to (Bi,Pb)-2212 phase, whereas in powder P-L, large amount of secondary phases are present, including Bi-2201, Ca_2PbO_4 and CuO. From our experience, the phase assemblage in powder P-L is one intermediate step in fabricating powder P-H.

Table I gives the detailed sintering schedule in this study. Number HT2 and number HT3 were designed to maintain the total sintering time of the samples first sintered for 20 h to 190 h, as same as that first sintered for 50 h.

The transformed 2223 phase fractions in both tapes after being heat treated for different durations at the first sintering period are plotted in Fig.2. It is clear

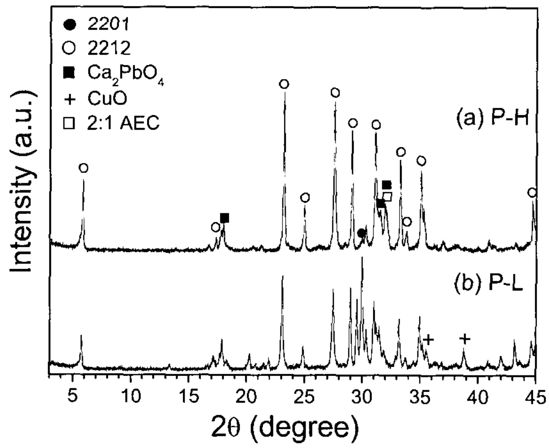

Fig. 1. XRD patterns of the precursor powders (a) P-H, and (b) P-L.

Table I. The detailed illustration of sintering schedule

Number	Sintering schedule
HT1	(12, 20, 30, 50, 70, 100) h /P/ 70 h /P/ 70 h
HT2	20 h /P/ 70 h /P/ 100 h
HT3	20 h /P/ 100 h /P/ 70 h

"P" presents the intermediate pressing

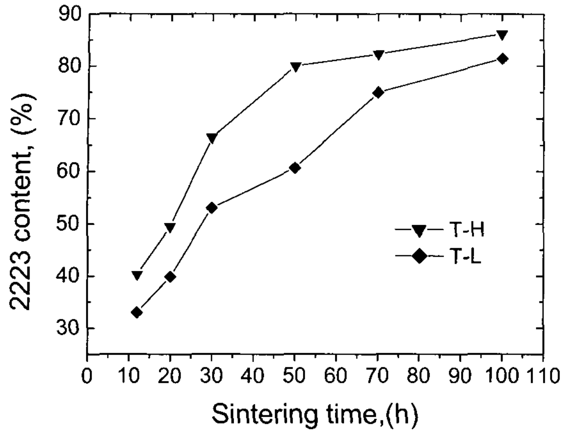

Fig. 2. The transformed Bi-2223 phase fractions in tapes T-H and T-L with respect to the sintering time during the first heat treatment.

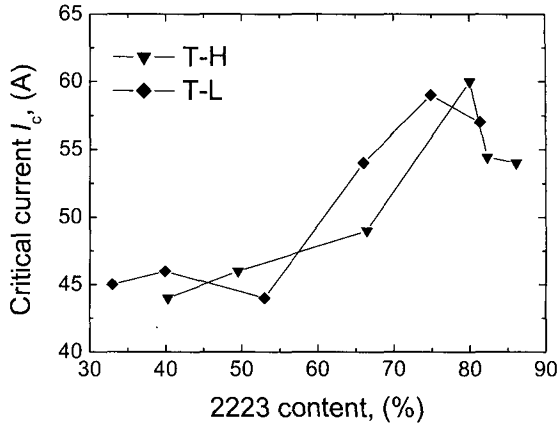

Fig. 3. The critical current I_c in fully processed tapes against the 2223 phase contents after the first heat treatment.

that the 2223 phase formed much faster in tape T-H than in tape T-L. After 50 h sintering for tape T-H and 70 h sintering for tape T-L, the increase of 2223 contents become moderate. It indicates that a higher calcination temperature resulted in a more reactive precursor powder, especially at the initial stage of heat treatment. In contrary, the powder contains more secondary phases needs long reaction time to reach the same 2223 phase content due to the slow formation rate. After reacted for a certain time, the phase formation rate is slowed down in both tapes. It is suggested that the phase formation at this stage is dominated by the two-dimensional diffusion controlled growth mechanism. With the consumption of liquid phase, the ions are difficult to diffuse to the grain growth front, so retards the 2223 phase formation.

The transport I_c values in the fully processed tapes against the 2223 percentages after the first sintering were compared in Fig.3. The highest I_c for tape T-H, 60 A, appears in the sample first sintered for 50 h, and for tape T-L, 59 A, appears in the sample first sintered for 70 h. In other word, the optimal stage for the first intermediate pressing was at 75-80% 2223 phase, being dependent on the starting precursor powders. The tapes with higher initial 2223 contents (sintered for 100h) did not lead to the highest I_c. It is not well consistent with the reports in [3, 6, 7]. This discrepancy most possibly comes from the varied precursor powders used by different authors.

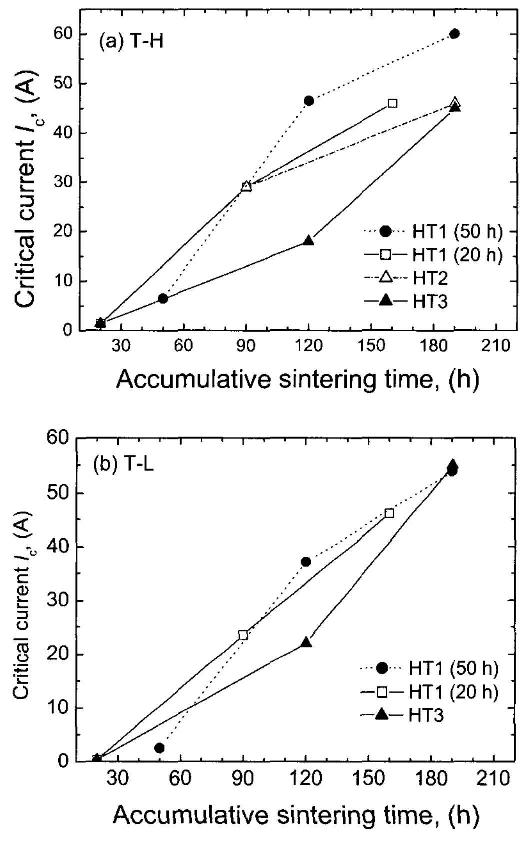

Fig. 4. The transport I_c in (a) tape T-H and (b) tape T-L with respect to various sintering-pressing cycles.

In order to make sure if the total sintering time has an impact on the transport I_c, we extended the dwell time of the second or third sintering stage to 100 h for the tapes first sintered for 20 h, thus maintaining a totally 190 h heat treatment as the sample first sintered for 50 h. Different I_c behaviors were observed in these two tapes. No improvement on I_c for tape T-H is found no matter at which stage the extended sintering period was performed, as shown in Fig.4 (a). However, for tape T-L, see Fig.4 (b), the I_c is improved to the same level as the tape first sintered for 50 h.

The heat treatment after an intermediate deformation usually has two effects: first to convert the remaining precursor phase into 2223, and second to use the residual eutectic liquid to heal deformation damages [5]. The 2223 phase formation is usually accompanied by a decrease of core density. So if the 2223 phase formation take places mainly during the first heat treatment, the subsequent sintering after intermediate deformation will not decrease the core density much anymore, hence improve the core density. However, there should also remain enough precursor phases to form sufficient liquid to heal the cracks. Our results show that maintaining the 2223 phase at about 75-80% after the first sintering fulfills both demands, i.e., improving core density and forming enough liquid to heal microcracks, which in turn leads to the enhancement of I_c property.

The dominant phase in powder P-H is (Bi, Pb)-2212, which leads to a rapid formation of 2223 phase at the beginning of sintering. Fig.5 shows the microstructure of both tapes after 20 h sintering. It is seen that the 2223 networks in tape T-H (Fig.5 (a)) has formed at this stage. However, the 2223 grains in tape T-L are still small and appear as isolate particles (Fig. 5 (b)). It is believed that the pre-formed 2223 framework in tape T-H has determined the main phase assembly and structure in final tape. The extended sintering did not affect much of the phase formation and grain growth. However, most of the reactants in tape T-L are still the precursor phases after 20 h sintering, which leaves much room and yields adequate liquid for the phase formation. So extending the sintering time allows a further nucleation and growth of 2223 phase, hence improves the critical current. Our results prove that the 2223 framework formed in the first heat treatment is very important for the subsequent grain growth, phase conversion, and as well as transport property in Bi-2223/Ag tapes.

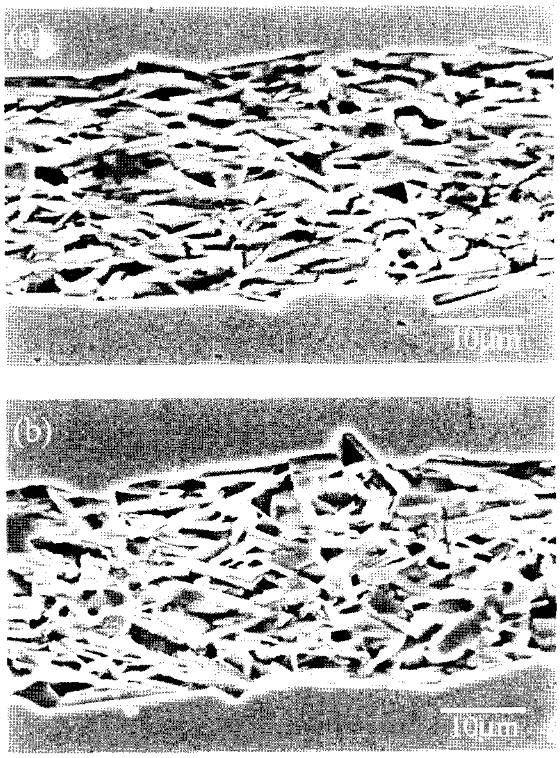

Fig. 5. The microstructures of (a) tape T-H and (b) tape T-L after 20 h sintering.

4. Conclusion

Monitoring the 2223 phase content to around 75-80% in the first sintering led to the best I_c value in fully processed Bi-2223/Ag tapes. The pre-formed 2223 framework after the first heat treatment has large impacts on the further phase formation, grain growth, as well as the transport property. A better control of the first heat treatment according to different precursor powders should be addressed before the following thermomechanical treatments.

Acknowledgment

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