The Effect of Relatively Low Lead Contents on the Phase Formation and Jc values of Bi-2223/Ag Tapes

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The effects of relatively low lead content (0.2~0.3) on the 2223 phase formation and transport property of Bi-2223/Ag tapes have been investigated. The results show that lead contents have great impact on the phase assemblage of precursors, subsequently, on the phase formation and transport property of silver sheathed BSCCO-2223 tapes. Powders containing Pb=0.25 and Pb=0.3 resulted in the nearly identical J_c values in fully processed tapes, but leaded to significant difference on the phase formation process. For the case of Pb=0.2, both low conversion fraction of 2212 to 2223 and low J_c value were obtained in final reacted tape, which was probably due to lack of enough liquid phase to facilitate the phase transformation.

Keywords: Lead content; Bi-2223/Ag tape; Phase formation; J_c

1. Introduction

A world-wide research efforts have been focused on the improvement of critical current density J_c of silver-sheathed Bi-2223 tapes since it is the key limiting factor on the way to practical applications of this miracle material [1,2]. Large and well *c*-axis oriented grains, better grain connectivity, together with as little as possible secondary phase in final reacted tapes were considered to be beneficial to carry large transport currents. Many factors related to the fabricating process of Bi-2223/Ag tapes, including chemical composition, powder calcination, sintering temperature and periods, and mechanical deformation have been optimized [3-6]. Among these, lead content is an important factor because it is directly related to the amount of transient liquid phase, which is of vital importance to stabilize the 2223 phase, accelerate the grain growth, heal cracks and improve core density [7,8]. The lead contents within the range of x=0.3~0.4 in $Bi_{2-x}Pb_xSr_2Ca_2Cu_3O_y$ have been reported to be favorable to the phase conversion and transport properties [9]. Zeimetz et al. [10] suggested that higher Bi/Pb ratio would facilitate the grain growth due to a relatively low phase formation rate. In this study, lead contents in a relatively low range (0.2~0.3) on the phase formation and transport current density of Bi-2223/Ag tapes have been investigated.

2. Experimental

Precursor powders with nominal composition of $Bi_{1.8}Pb_xSr_{2.0}Ca_{2.2}Cu_{3.0}O_z$ (x=0.2, 0.25, 0.3) were prepared by spray drying method. The phase assemblages of final calcined powders prior to filling into silver tubes were examined using X-ray diffraction. DTA patterns of precursor powders were determined by a Perkin-Elmer DTA1700 at a ramping rate of 10°C/min. Three kinds of 61 filaments Bi-2223/Ag tapes, different in lead contents in precursor powders, were fabricate by the well-established powder-in-tube method. The short pieces of tapes cut from the as-rolled long tapes were subjected to multiple sintering and pressing circles and single sintering followed by quenching in air after various dwelling time. Both the single and multiple sintering were performed at 843°C. The critical current was measured at 77 K and 0 T by the standard four-probe method with a criterion of 1μV/cm. XRD measurements were performed on longitudinally peeled samples. The 2223 phase variations were calculated on the basis of XRD intensity ratios of $(0\ 0\ 8)_{2212}$ and $(0\ 0\ 10)_{2223}$ peaks [11]. The cross sections of reacted tapes were observed

using SEM and EDX.

3. Results and discussion

3.1 Precursor powder characterizations

Fig.1 plots the XRD patterns of final calcined powders with varied lead contents. It is clear that the dominant phase in these powders is Bi-2212. However,

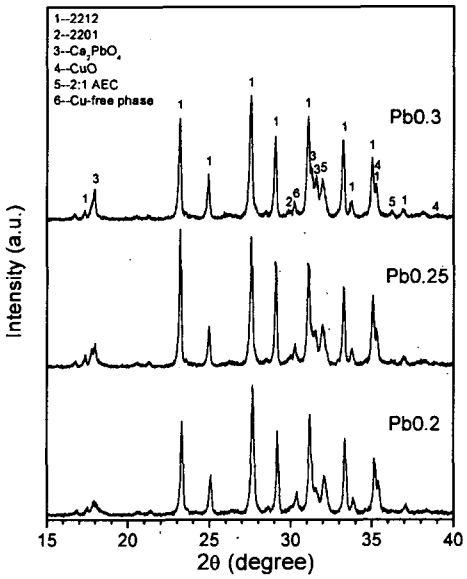

Fig.1. XRD patterns of final calcined precursor powders with varied lead contents.

with the increasing of lead contents, the characteristic peak for Ca_2PbO_4 phase around 2-theta=17.8° increases,

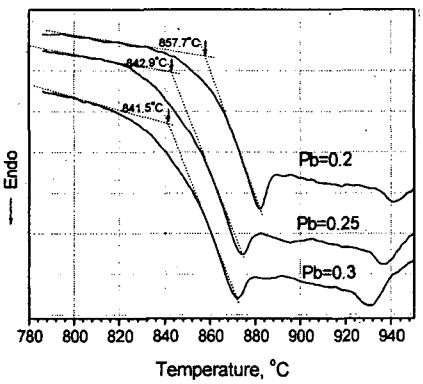

Fig.2. DTA curves of final powders containing varied Pb contents prior to filling into silver tubes.

which is quite well consist with our previous work [12].

DTA curves of the precursor powders were compared in Fig.2. The first endothermic peak, belonging to the melting of (Bi,Pb)-2212, is shifted to lower temperatures with the increase of lead contents.

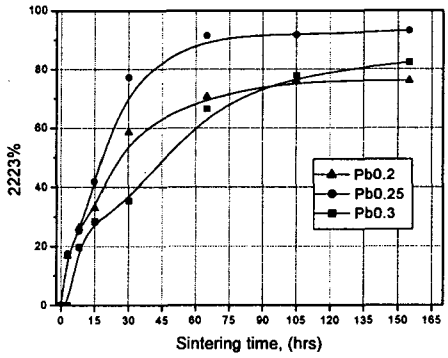

Fig.3. Volume fraction of transformed 2223 with respect to sintering time before quenching at 843°C.

The onset points in powders containing Pb=0.2, 0.25 and 0.3 are 857.7°C, 842.9°C and 841.5°C, respectively. It is worthy to note that the difference of the onset points between powders with Pb=0.25 and 0.3 is not as significant as that between Pb=0.2 and 0.25, which means the former two kinds of powders maybe have the similar quality.

3.2 Phase formations and I_c values

Fig.3 presents the 2223 percentages in different

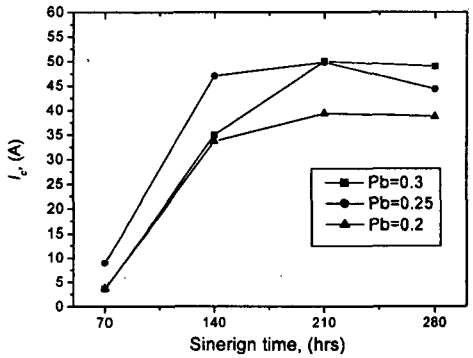

Fig.4. I_c at 77 K and 0T as a function of sintering time for tapes fabricated with different lead contents powders.

tapes versus dwelling time at the reaction temperature (843°C) before quenching. It shows that powder with

Pb=0.25 result in the fastest formation rate of 2223 phase at all the sintering process. Higher Pb content, i.e. Pb=0.3 in this study, does not correspond to high conversion rate from Bi-2212 to Bi-2223, which is even lower than the one made from Pb=0.2 at the first 65h sintering. Powder containing Pb=0.2 leads to the lowest 2223 content after 155h sintering, i.e. only 76%, which is probably due to the inadequate liquid phase formed in the later sintering for the phase transformation. The formation of 2223 phase is a complicated process that can be influenced by many factors, such as precursor phase assemblage, lead content, powder particle size, mechanical deformation and sintering temperature. The different phase formation can't be ascribed to the effect of lead content only while neglecting the fabrication history of powders and tapes.

The critical currents I_c after every single sintering in the sintering-pressing process were measured and compared in Fig.4. At the first two sintering stages, tape made from powder containing Pb=0.25 shows the fastest I_c increase comparing to the other two kinds of tapes, which is consistent with the fast phase formation as shown in Fig.3. However, the highest I_c of tapes made from Pb=0.25 and 0.3 are nearly same, which are 49.8 and 50A, respectively. While, for the tape made from powder containing Pb=0.2, the I_c still keeps slightly increasing after totally 280h sintering and thee times pressing, which indicates that the phase conversion is far from be completed due to lack of the assistant of enough liquid phase.

3.3 Microstructural observations

The SEM images taken from the polished and etched longitudinal cross section of above tapes after 210 h sintering and twice pressing were shown in Fig.5. It is clear that the large grains are well aligned in tapes made from powders of Pb=0.3 and 0.25, especially near the oxide/silver interface. While for the case of Pb=0.2, small grains together with large amount of black particles (AEC phase) laying on the 2223 matrices, which can explain the low I_c values. Thus, we can conclude that the low lead content (x=0.2 in this study) is unfavorable for the phase formation and grain growth although its Bi/Pb ratio is the highest one among these three samples.

4. Conclusion

The influence of relatively low lead contents on the powder phase assemblage, phase formation and Jc values in Bi-2223/Ag tapes have been studied. Powder containing Pb=0.25 results in a faster phase formation in tape than samples made from powders of Pb=0.3 and 0.2. The highest I_c of fully reacted tapes from powders with Pb=0.25 and 0.3 are nearly equivalent, however, it is obvious lower in tape made from powder containing

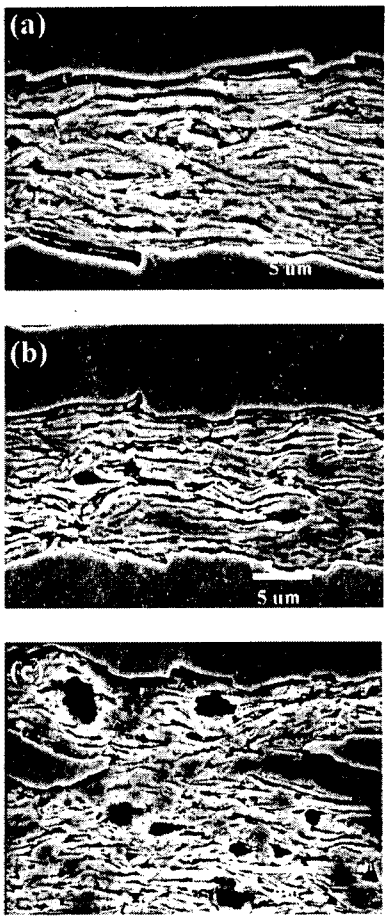

Fig.5. SEM images of polished and etched longitudinally cross section of fully processed tapes made from different lead contents (a) Pb=0.3, (b) Pb=0.25 and (c) Pb=0.2.

Pb=0.2. The microstructures of the former two fully reacted tapes are characterized with large and well-aligned grains, which is favorable to carry high transport currents. Large amount of secondary phase, (mainly AEC phase), exist in the final tape started from Pb=0.2, and the conversion of 2212 to 2223 is far from be completed, both of which are the possible reasons for the poor transport currents.

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