

## Controlling the initial conditions of precursor powders and its effects on the phase evolution and $J_c$ properties of Bi-2223/Ag tapes

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### Abstract

By varying fabricating process, precursor powders with different initial conditions were prepared. Subsequently, Bi-2223/Ag tapes were made through these powders. The effects of precursor powders on the phase evolution and  $J_c$  properties of Bi-2223/Ag tapes were studied along with several thermomechanical cycles. Our results showed that the initial conditions of precursor powders could strongly influence the phase formation rate and  $J_c$  value in final tapes. The factors of precursor powders that influence the phase formation and  $J_c$  of Bi-2223/Ag tapes must be studied and optimized in combination.

*Keywords:* Bi-2223/Ag tapes; precursor powder; phase evolution;  $J_c$

### I. Introduction

It has been well accepted that the powder process is the first and most important step in fabricating high  $J_c$  Bi-2223/Ag superconducting tapes by PIT technique [1,2]. To achieve Bi-2223/Ag tapes with better performance, the initial conditions of precursor powders must be optimized. Intensive research efforts on the chemical composition, particle size distribution, calcination procedures, carbon content and phase assemblage of precursor powders have been carried out to investigate their effects on the microstructure evolution and properties of Bi-2223/Ag tapes.

Nonaka et al. [3] suggested that precursor powders calcined at 760°C could result in faster phase

formation rate and higher  $J_c$  values in Bi-2223/Ag tapes. This is consistent with the result in ref. [4]. Syamaprasad et al. [5] suggested that precursor powders containing high volume fraction of 2212, low 2201 together with  $\text{Ca}_2\text{PbO}_4$ , CuO and some other secondary phases, without any 2223 phase, was a good choice for the fabrication of high  $J_c$  (Bi,Pb)-2223/Ag tapes.

Li et al. [6] showed that powders with small particle size were expected for fabricating high  $J_c$  tapes. The results of Jiang et al. [7] also showed that small particle size of precursor powders would promote the formation of Bi-2223 phase, but too fine a powder may cause the formation of large secondary phases, which are detrimental to the  $J_c$  properties. Vo et al. [8] and Su et al. [9] have also separately shown that small particle size of precursor powders could increase the phase formation rate and give an uniform microstructure, hence higher  $J_c$  value in Bi-2223/Ag

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tapes.

However, the factors of precursor powders that influence the microstructure and properties of Bi based tapes were usually investigated independently. It is essential to combine these factors together and study their effects at the same time.

In this paper, precursor powders with different initial conditions, such as particle size and phase assemblage, were fabricated and their effects on the phase evolution and  $J_c$  properties have been investigated.

## II. Experimental

Powders with a nominal composition of  $\text{Bi}_{1.8}\text{Pb}_{0.2}\text{Sr}_{2.0}\text{Ca}_{2.2}\text{Cu}_3\text{O}_x$  were prepared by spray-drying method. The spraying dried powders were treated by a series of calcination and grinding steps. The final powders prior to PIT were identified by P-1 and P-2 according to different low temperature post annealing time. The microstructures of the powders were observed by SEM. The phase assemblages of the precursor powders were examined by X-ray diffraction.

The 61 core multifilament Bi-2223/Ag tapes T-1 and T-2 were fabricated by the well established powder-in-tube (PIT) technique through powder P-1 and P-2. Then short green tapes about 4cm long, 350 $\mu\text{m}$  thick and 4mm width, which were cut from the as-rolled long tapes, were subjected to multi-step sintering-pressing circles. Every sintering was performed at 843°C for 70 hours according to our previous study. The  $I_c$  values were measured by the standard four-probe method at 77K and 0T with a criterion of 1 $\mu\text{V}/\text{cm}$ . XRD measurements were performed on longitudinally peeled samples. The Bi-2223 contents were calculated on the basis of the XRD intensity ratios of the (0 0 8)<sub>2212</sub> and (0 0 10)<sub>2223</sub> peaks [10].

## III. Results and discussion

Fig. 1 shows the XRD patterns of the two precursor powders. The main difference between P-1 and P-2 is the former includes less Bi-2201 and more  $\text{Ca}_2\text{PbO}_4$  phase. That means that longer time annealing will decompose the Bi-2212 into Bi-2201 and some other second phases. From our result and

ref. [5], we can see that the phase assemblage can vary much due to different annealing conditions on precursor powders.

The secondary electron micrographs of both powders are shown in Fig. 2. The powder that was annealed for longer time has relatively larger average particle size. With increasing annealing time, some large particles appeared. These large particles have been thought harmful to phase formation and phase purity [7].

$I_c$  and Bi-2223 content of both tapes are compared in Fig. 3 after each sintering period. From the first sintering period, the 2223 content of T-1 is obviously higher than that of T-2, and the  $I_c$  value of T-1 is also higher than T-2 in all sintering periods. After the first 70 hours sintering,  $I_c$  of T-1 is 2.1A, while it is only 0.2A in T-2. The highest  $I_c$  value of T-1 is 46.8A after three times sintering and two intermediate pressings, while in T-2 it is only 29.7A even after 280 hours sintering and three time pressings.  $I_c$  of T-1 decreased after the fourth sintering, while it increased monotonously in T-2 along all the sintering periods. This result is well consistent with the one of Su et al. [9]

From Fig. 2, we can see that P-1 has finer particle size and more homogenous particle size distribution. The fast phase formation rate in T-1 must have intimate relation with the fine and homogenous particle sizes. The smaller particles can shorten the induction period at the beginning of Bi-2223

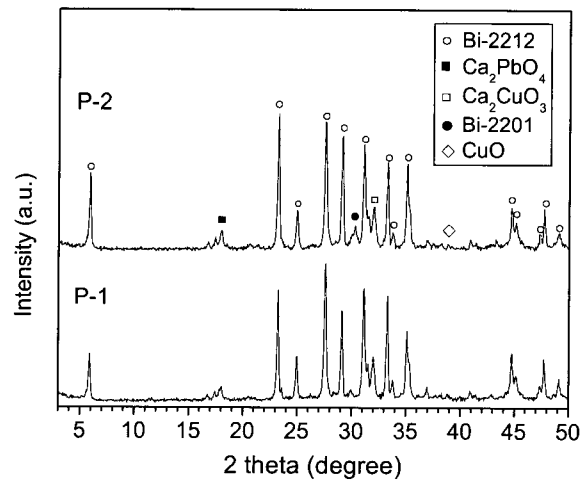


Fig. 1. XRD pattern of the precursor powders P-1 and P-2.



Fig. 2. Secondary electron images of precursor powders (a) P-1 and (b) P-2.

formation, hence accelerate the formation of 2223 phase. The large particles in P-2 would enlarge the diffusion distance during the phase transformation, hence hinder the 2223 phase formation and phase purity.

In ref. [5], the authors suggested that a precursor that containing more 2212, less 2201, together with CuO and  $\text{Ca}_2\text{PbO}_4$  was an expected choice for fabricating high  $J_c$  Bi-2223/Ag tapes. Our results showed the same tendency. For T-2, due to long time low temperature annealing, 2212 decomposed into 2201 and some other secondary phases. So it would inescapably take time to recover the 2201 to 2212 in the heat treatment, as a result, delay the formation of 2223. The Pb-rich liquid phase has been proven of vital importance to the 2223 phase nucleation and grain growth. It is also important for healing cracks

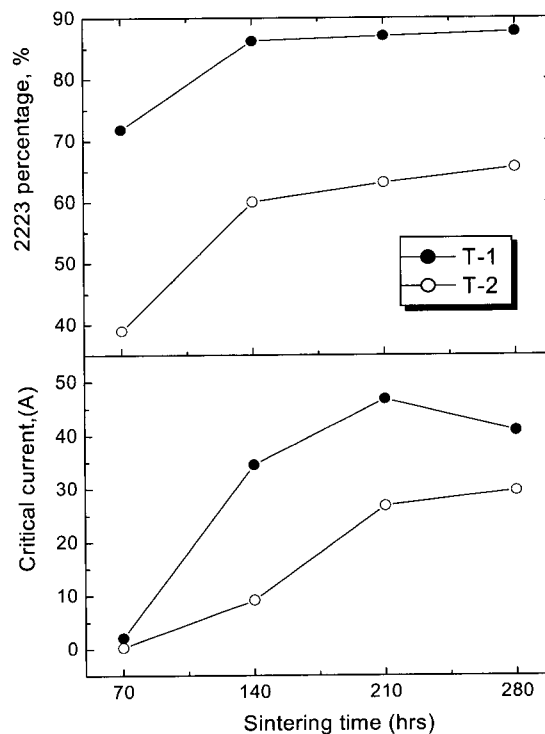


Fig. 3. Transport critical current  $I_c$  and Bi-2223 content vs sintering time for tapes T-1 and T-2.

that were introduced by rolling or pressing. It was suggested that Pb-rich Bi-2212 was formed firstly from the reaction of Pb-free Bi-2212 and  $\text{Ca}_2\text{PbO}_4$  or 3221, and then it was converted into Bi-2223 phase during the phase transformation [11]. In our case, P-1 containing less 2201 and  $\text{Ca}_2\text{PbO}_4$ , this means that most part of Pb-rich 2212 has been formed in the precursor powders. So it is not difficult to understand the fast phase formation rate in T-1.

Fig. 4 Shows the XRD spectra of tape T-1 (after third sintering) and T-2 (after fourth sintering). We can see that in tape T-2 there are still many high density 2212 peaks even after 280 hours sintering. This also illustrates that P-1 is more reactive than P-2 due to the different characters of particle size and phase assemblage.

#### IV. Conclusion

In this paper, we studied the influence of initial conditions of precursor powders on the phase

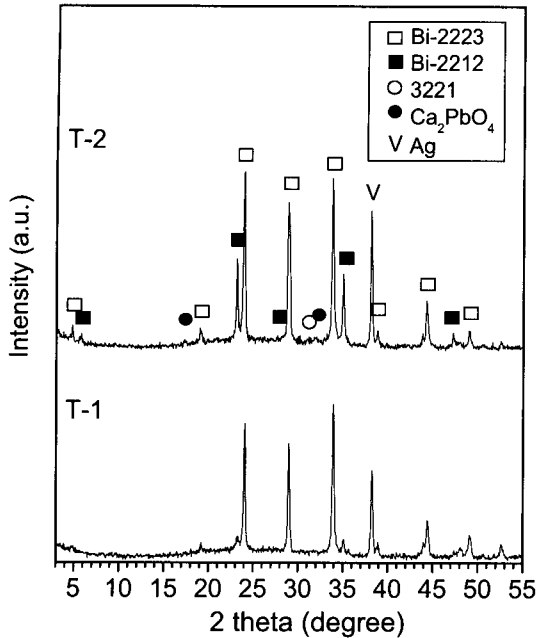


Fig. 4. XRD patterns of reacted tapes T-1 (after 210hrs sintering) and T-2 (after 280hrs sintering).

formation and  $J_c$  properties of Bi-2223/Ag tapes. Our results showed that a precursor powder with fine and homogeneous particle size and proper phase assemblage could result in fast phase formation rate and higher  $I_c$  values. Although the two factors affect the phase formation by different ways, they are both important for producing high performance tapes. So in order to fabricate long length and commercial use tapes, both the particle size and phase assemblage of precursor powders should be optimized in combination, while not independently.

## Reference

- [1] L. Hua, J.M. Yoo, J.W. Ko, H.D. Kim, H.S. Chung, G.W. Qiao, "Effect of precursor powders on phase evolution and electromagnetic properties of controlled melting processed Bi-2223 Ag-sheathed tapes", *Physica C*, 308, 105-114 (1998).
- [2] J. -C. Grivel, R. Flukiger, "Kinetic study of the growth of the  $(\text{Bi, Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$  phase from calcined precursor powders", *J. Alloys. Comp.* 235, 53-58 (1996).
- [3] S. Nonaka, K. Osamura, "Influence of calcining temperature on critical current density of Ag/Bi2223 tapes", *Physica C*, 281, 205-210 (1997).
- [4] Y. T. Huang, D.S. Shy, L.J. Chen, "Effects of powder calcination on the properties of Bi-2223 tape", *Physica C*, 254, 159-166 (1995).
- [5] U. Syamaprasad, M.S. Sarma, P. Guruswamy, V. Prakash Kumar, R. Ragini, K.G.K. warrier, A.D. Damodaran, "Effect of precursor phase assemblage on 2223 phase formation and  $J_c$  in Ag/(Bi,Pb)-2223 tapes", *Physica C*, 297, 85-90 (1998).
- [6] Q. Li, K. Brodersen, H.A. Hjuler, T. Freltoft, "Critical current density enhancement in Ag-sheathed Bi-2223 superconducting tapes", *Physica C*, 217, 360-366 (1993).
- [7] J. Jiang, J.S. Abell, "Effects of precursor powder particle size on the critical current density and microstructure of Bi-2223/Ag tapes", *Physica C*, 296, 13-20 (1999).
- [8] N.V. Vo, J.O. Willis, D.E. Peterson, H.K. Liu, S.X. Dou, "Optimization on processing parameters for Bi(Pb)-2223 superconducting tapes", *Physica C*, 299, 315-326 (1998).
- [9] X.D. Su, J.M. Yoo, J.W. Ko, H.D. Kim, H.S. Chung, Z.Q. Yang, G.W. Qiao, "Influence of precursor powders on the phase evolution, grain size and transport current properties of BCSSO 2223 Ag-sheathed tapes", *Physica C*, 331, 285-291 (2000).
- [10] Q.Y. Hu, H.D. Liu, S.X. Dou, "Formation mechanism of high- $T_c$  phase and critical current in  $(\text{Bi, Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ /Ag tapes", *Physica C*, 250, 7-14 (1995).
- [11] B. Lehdorff, P. Hardenbicker, M. Hortig, H. Piel, "Evidence for enhanced grain connectivity due to second phase reduction of Bi-2223/Ag tapes", *Physica C*, 312, 105-111 (1999).