Proceedings of the Korean Superconductivity Society Meeting 1999
(KSS 99), YongPyung, Kangwondo, Korea, Aug. 18-20, 1999

The effect of precursor powder on the properties of BSCCO 2223 Ag-sheathed tapes

Xiao-dong Su, Jai-moo Yoo, Jae-woong Ko, Sung-Chang Park, Hai-doo Kim,
Hyung-sik Chung, Gui-wen Qiao*

66 Sangnam-dong, Changwon, Kyungnam, Korea 641-010,
Korea Institute of Machinery & Materials
**Institute of Metal Research, Chinese Academy Sciences, Shenyang 110015, China*

The BSCCO 2223 Ag-sheathed tapes were prepared with spray dried precursor powders, which are of different starting condition. The J_c in the short tapes varied from 24 kA/cm^2 to 47 kA/cm^2 at 77K and 0T. The largely improvement of J_c in certain tape, which characterized with the large textured BSCCO 2223 grains, homogeneity of reactant and fewer tracks of second phases in final superconducting matrixes, could be due to optimization in the precursor powder.

1. Introduction

It is well known that the characterization of precursor powder, such as stoichiometry, the phase assemblage, particle size and so on, are of vital importance to the final properties of Bi-2223 tapes [1-5]. The precursor powder prepared by spray drying method has the merits of good chemical homogeneity, high activity and low carbon content. In general, J_c values of 10-30 kA/cm^2 can be obtained in the tapes with spry dried powder; this range of current density is only 1-5% of the best thin film values [6].. Hence, one can expect a significant improvement in the transport properties of the tapes by optimizing the manufacturing procedures. In fact, the particle size of spray dried powder is still in a wider range, such as from 0 - 50 ㎛, furthermore grinding or ball milling and sieving is effective to reduce large sized particles in the precursor powder. Vo. et al.[7] suggested that the fine and homogeneous powder can increase its reactivity, which would expedited the phase transformation process and to give a more uniform microstructure.

The purpose of this work is to study the effects of different starting condition of precursor powder on the phase evolution, microstructure and current transport properties of Bi-2223 Ag-sheathed tapes prepared by spry dried precursor powder.

2. Experimental

First, we obtained the mixed powder with an initial stoichiometry of $Bi_{1.8}Pb_{0.3}Sr_2Ca_{2.2}Cu_3O_y$ by spray drying method. The powders were sequentially calcinated at 800℃ for 2 hours, then 840℃ for 15 hours twice with intermediate grinding steps. Calcinated powder was separated into two pitches, which then were milled with planetary ball mill for 45 minutes and 120 minutes, and marked as P-A and P-B, respectively. All powders were degassed at 600℃ for 1 hour in flow oxygen atmosphere to reduce the carbon contaminant. DTA patterns for two powders were finished by using Perkin-elmer DTA1700 Differentail Thermal Analyzer.

Second, then well-established powder-in-tube technique (PIT) was used to fabricate the 61 cores superconducting tapes, and the details of PIT processing can be found elsewhere [9, 10]. The as-rolled tapes are about 4mm width and 350μm thickness. Preliminary experiments were carried out to determine optical sintering temperature for both tapes is 841℃ in air. Then short pieces (4cm in length) was cut from each tape and subjected to a thermomechanical process. Each sintering period was performed at 841℃ for 70 hours in air, and internal uniaxial pressure is 2Gpa holding for 1 minute.

After each step of sintering, Ic was measured as a function of self field or applied magnetic field B by the standard four-probe technique at 77K with a criterion of 1μV/cm. XRD measurements were performed on longitudinally peeled samples of tapes. The content of Bi-2212 and Bi-2223 was calculated on the basis of the XRD intensity ratios of the $(008)_{2212}$ and $(0010)_{2223}$ peaks[11]. Cross sections of final samples were examined by SEM and EDX.

3. Results and discussion

3.1 Precursor Powder Characterization

The precursor powders of P-A and P-B have same original composition. The XRD indicated that precursor powders were consisting of dominant Bi-2212 phase and small amount of second phases. The SEM images of precursor powder are shown in Fig. 1. The average size of precursor powder is lower than 10μm for both samples of P-A and P-B, but it is evidential the particles in P-B is more fine than that in P-A. EDX detected that large and plate-liked phase is major Bi-2212 phase.

DTA patterns performed on two precursor powders are shown in Fig. 2. The large endotherm present on two DTA curves is presumed to the melting of the Bi-2212 phase. The melting point of Bi-2212 in P-B is ahead of that in P-A, and it can be attributed to fine particles in former sample, which is high reactivity of precursor powder. However, the melting onset temperature is almost same in two samples.

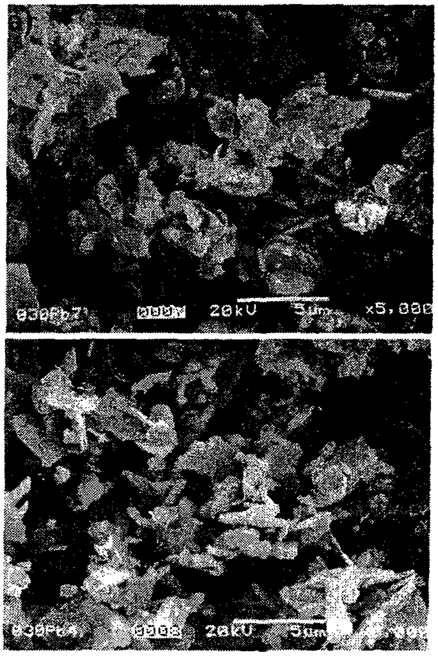

Fig. 1 Secondary electron micrographs of precursor powder: a) P-A and b) P-B

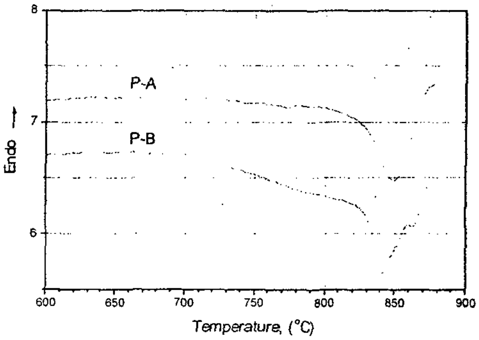

Fig. 2 Differential thermal analysis (DTA) patterns for precursor powder.

3.2 Critical Current(I_c) and I_c dependence on magnetic field.

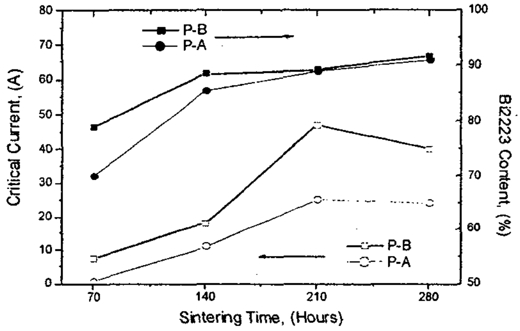

Fig. 3 Transport critical current I_c(77K, 0T) and Bi2223 content vs. Sintering time for two tapes made from different precursor powder.

The I_c and Bi-2223 content of two tapes, which made from different starting powders P-A and P-B, are compared in Fig. 3 after every period sintering. In the first period of sintering, the I_c and Bi-2223 content of tape P-B reached to 7.5A and 79%, which is very high compared with 0.7A and 60% in the tape of P-A (here, we still use P-A and P-B to mark two tapes). It means that in the beginning of sintering the Bi-2212 phase is easy to covert into Bi2223 phase in the tape P-B than that in the tape of P-A. Above results are consistent with others work [12], they found that the particle size will influence the induction period in the beginning of sintering processing. More coarser of precursor powder, more long induction period, which should be prevented, because the large sized particles contained in precursor powder in tape is difficult to react with other particles due to its relatively low activation energy. Furthermore, these particles will inhibit the transformation from Bi-2212 into Bi-2223, and causes the existence of residual phases, distorting the grain alignment and connectivity of the Bi-2223 grains in the matrixes.

In the second and third periods of sintering, the I_c and Bi-2223 content in both of tapes increase meanwhile. The best I_c is gotten after pressing twice and total sintering time is 210 hours at 841℃, and its values were 25A and 47A for tape P-A and P-B, respectively. The I_c in the P-B tape is almost double of that in the P-A tape, although Bi-2223 content almost reached to maximum 91% in each tape. In this case, first we think the improvement of I_c in two tapes is mainly due to the formation of large textured Bi-2223 grains and the almost elimination of second phases in the superconducting matrixes.

After the fourth period of sintering, the Bi-2223 content still have slight increasing but the I_c begin to decrease in both of samples. It should be due to the lack of enough of liquid phase Ca_2PbO_4 in the matrixes, then the cracks formed in the tapes after immediate pressing cannot be healed, and it results in the decresing of I_c in both P-A and P-B.

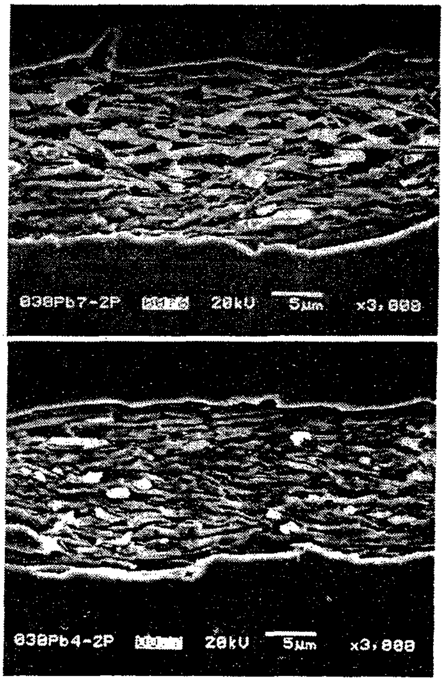

Fig. 4 Secondary electron micrographs of two tapes after 210 hours sintering and two times pressure: a) P-A and b) P-B

However, why the I_c in the two tapes performs so different? Fig. 1 has shown us that the particles in P-B are finer than that in P-A. After first period sintering and intermediate uniaxial pressure, many large impurity phases still remained in the tape of P-A, and it maybe decrease it size and quantity in followed sintering periods but it is difficult to remove it from matrixes completely. Fig. 4 shows the SEM images of two tapes, which have applied 210 hours sintering and pressing twice. From the observation, we can find that the superconduting matrixes(gray Bi-2223 grains) is highly densified and well-aligned, but there still have some white phases dispersed in both of tapes. Unfortunately, these white phases are predominantly located at Bi2223 grain boundaries, and results in greatly detrimental to current transport properties. It is obvious that the amount of white phases in P-B is less and small than that in P-A; The average size of Bi-2223 grains, which are 5μm width and 15 μm length typically in P-B, is also large than that in P-A. Since that, the Ic values in P-B get large improvement compared with P-A. So, the starting condition of precursor powder, is very important to the phase evolution, morphology and I_c properties in the final tapes.

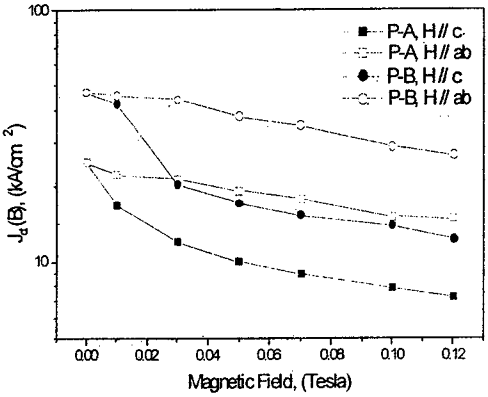

Fig. 5 The $J_{ct}(B)$ curves at 77K for the fully processed tapes with the applied field parallel to the c-axis and ab plane.

On the other hand, the starting condition of precursor powders maybe influences the connection and flux pinning properties of the each tape. The J_c values in magnetic fields were illustrated in Fig. 5. With the increasing of applied field, the Jc in P-A drops faster than that in P-B at both direction of H // c and H // ab in the low field regime (less than 0.03 Tesla), then J_c keep slow decreasing in both samples in the high field regime. In low field regime, J_c is controlled by the weak links between superconducting grains. At high fields, J_c behavior is governed mainly by flux pinning in H // c direction, but by both flux pinning and grain alignment in H // ab direction [13, 14]. The tape of P-B demonstrate better $J_{ct}(B)$ behavior than the tape of P-A throughout the entire measured magnetic field range. Undoubtedly, above results suggested that the P-B has better connection between superconducting grains and possibly more effective flux pinning centers in the matrixes compared with P-A.

4. Summary

We have investigated the influence of the different starting precursor powder on the properties of Bi2223 Ag-sheathed tapes. It is found the precursor powder of P-B, which was employed more long time ball milling, have proved to be more reactive than P-A, which is coarse in the particle size. After full thermomechanical process, the Bi-2223 phase purity, grain size and texture degree in the tape of P-B almost get to optimum, and it results in the largely improvement of I_c compared with tape of P-A. The measurement of J_c in the applied magnetic filed also prove that P-B tape have reduced the weak links and possibly enhance the flux pinning ability. Optimization of precursor powder is very effective to improve the properties of final tapes.

References

[1] L. Hua, J.M. Yoo, J.W. Ko, H.D. Kim, H.S. Chung, G.W. Qiao, Physica C 308(1998)105-114.

[2] S. Koyama, U. Endo, and T. Kwai, Jpn. J. Appl. Phys. 27(1988) L1861.

[3] L. Pierre, J. Schneck, D. Morin, J.C. Toledano, J. Primot, L. Daguet, and H. Savary, J. Appl. Phys. 68(1990)2296.

[4] S.E. Dorris, B.C. Prorok, M.T. Lanagan, N.B. Browning, M.R. Hagen, J.A. Parrell, Y. Feng, A. Umezawa, D.C. Larbalestier. Physic C 223(1994)163.

[5] Q. Li, K. Brodersen, H.A. Hjuler, T. Freltoft, Physica C 217(1993) 360.

[6] Y. Hakuraku and X. Mori, J. Appl. Phys. 73(1992) 309.

[7] N.V. Vo, J.O. Willis, D.E. Peterson, H.K. Liu and S.X. Dou, Physica C 299(1998) 315-326.

[8] J.M. Yoo, H.S. Chung, J.W. Ko, H.D. Kim, J. Sha, Physica C 267(1996) 53.

[9] J.W. Ko, H.D. Kim, B.T. Lee, S.M. Yoo, J.M. Yoo, H.S. Chung, in: H. Hayakama, Y. Enomoto(Eds), Advances in SuperconductivityVIII, 1995, p839.

[10] Q.Y. Hu, H.D. Liu, S.X. Dou, Physica C 250(1995) 7.

[11] Y.L. Wang, W. Bian, Y. Zhu, Z.X. Cai, D.O. Welch, R.L. Sabatini, M.Suenaga, T.r. Thurston, Apply. Phys. Lett. 69(1996) 580.

[12] K. Ficher, T. Fahr, A. Hutten, E. Muller, M.Schubert, D. Schlafer, IEEE Transactions on applied superconductivity, vol. 7, No.2, June 1997

[13] R.L. Perterson and J.W. Ekin, Physica C, 157(1989)325

[14] B. Hensel, J.C. Grivel, A. Jeremie, A. Perin, A. Pollini and R. Flukiger, Physica C, 205(1993) 329