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ARTICLE

Microstructure and Superconductivity of Bi-2223 Tape Prepared by Precursor Powders with Different Particle Sizes

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Abstract: The effects of particle size of precursor powders on microstructure and superconductivity of Bi-2223 tape were investigated. Three kinds of precursor powders with different particle sizes (8, 2, <1 μm) were prepared by a spray pyrolysis method via adjusting the concentration of metal nitrates solution and ball milling. All powders were composed of Bi-2212, $(Sr, Ca)_xCu_yO_\delta$ (AEC) and CuO phase after heat treatment. Results show that AEC phase dimension and content increase with decreasing of particle size of the precursor powders. However, the powder with an average particle size of 2 μm has the minimal dimension and content of CuO phase. Among the 37-filaments Bi-2223 tapes fabricated from the three kinds of precursor powders, the one from the powder with average particle size of 2 μm achieves the highest critical current (I_c), which also has the most $(Bi, Pb)_3Sr_2Ca_2CuO_x$ (Pb-3221) and least AEC phase compared to other tapes. The particle size of the precursor powders mainly affects dimension and content of AEC and CuO phase, which further causes difference of I_c and non-superconducting phases in Bi-2223 tape.

Key words: precursor powder; particle size; Bi-2223; superconductivity

Owing to high superconducting transition temperature and high critical current of $(Bi, Pb)_2Sr_2Ca_2Cu_3O_x$ (Bi-2223) tape at 77 K, many efforts have been devoted to achieve commercial application. Critical current density (J_c) has reached 70 kA/cm^2 up to now [1]. However, the extremely high J_c of 170~180 kA/cm^2 and 1000 kA/cm^2 have been reported in magneto-optic and single-crystal thin films of Bi-2223 [2,3], which implies that there is larger room to further increase J_c of Bi-2223 tape.

The final superconducting properties of Bi-2223 tape depend strongly on process parameters, such as precursor powder, mechanical deformation and thermo-mechanical treatment. The precursor powder, including preparation and heat treatment is the most key step because of complicated phase relationships in Bi-Pb-Sr-Ca-Cu-O system[4]. Phase composition, particle size, homogeneity and reproducibility are regarded as important factors for an ideal precursor powder, especially for commercial application. It is generally believed that phase assemblages of Bi-2212, Ca_2PbO_4, Ca_2CuO_3 and CuO or (Bi,Pb)-2212, AEC and CuO are beneficial to high J_c of Bi-2223 tape. The homogeneity and reproducibility mainly depend on preparation methods, which has been demonstrated by comparison studies of solid state reaction, co-precipitation, spray drying and spray pyrolysis[5], and spray pyrolysis method is considered as an ideal method. Jiang and Li et al suggested that a small particle size plays an important role in obtaining high J_c of Bi-2223 tape based on solid state reaction and the co-precipitation method[6,7]. However, there have been

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few systemic reports on effects of particle size on superconducting properties for the spray pyrolysis method.

In this work, the precursor powders with different particle sizes were prepared by the spray pyrolysis method. Phase assemblages especially secondary phase were analyzed after heat treatment of precursor powders. 37-filaments Bi-2223 tapes were fabricated by well-known powder in the tube technique to study effects of particle size on superconducting properties.

1 Experiment

Precursor powders, with the nominal composition of $Bi_{1.87}Pb_{0.34}Sr_{1.9}Ca_{2.1}Cu_{3.04}O_x$, were prepared by the spray pyrolysis method using a mixed nitrate solution. Three precursor powders were preparedto obtain different particle sizes. Powder 1 was prepared with 2 mol/L solution concentration, powder 2 and powder 3 were prepared with 0.5 mol/L solution concentration, and then powder 3 was milled for 3 h using ethanol as a liquid medium. Three precursor powders were heat treated at 780 °C for 10 h in a flowing 0.1%O_2/balance N_2 atmosphere.

The 37-filaments Bi-2223 tapes were fabricated by the established powder in the tube technique as reported elsewhere[8]. Three types of tapes (A, B, C) were designated according to the filled precursor powder 1, 2 and 3. All short tapes were sintered at 824 °C for 20 h in a flowing 8% O_2/balance N_2 atmosphere. Then tapes were rolled in one pass to reduce 25% tape thickness. After that, all tapes were sintered at 824 °C for 80 h and 780 °C for 24 h.

The phase composition was determined by X-ray diffraction (XRD). Microstructures of powders and tapes were analyzed by scanning electron microscope (SEM). The critical currents of tapes were measured by the standard four-point probe technique in nitrogen liquid.

2 Results and Discussion

Fig.1 shows typical SEM micrographs of precursor powders prepared by the spray pyrolysis method using different nitrate solution concentrations. As shown in this figure, both original powder 1 and powder 2 are spherical and well distributed. The average particle size of powder 1 is about 8 μm because of large solution concentration, while that of powder 2 is 2 μm. Powder 3 was prepared by milling of powder 2 for 3 h using ethanol as a liquid medium, and the average particle size is less than 1 μm. After heat treatment at 780 °C for 10 h, all powders show plate-like grain crystal, which are Bi-2212 phase and have average dimension less than 3 μm.

Three kinds of powders were pressed into bulks under the same pressure, which were polished carefully, to study the evolution of non-superconducting secondary phases with decreasing of particle size of precursor powders after heat treatment. Fig.2 shows SEM micrographs of three bulks. It is clearly shown that the dimension of AEC phase increases with decreasing of particle size of precursor powders. Compared to powder 1 and powder 3, powder 2 has a minimal dimension for CuO phase. It is noted that the dimension of AEC and CuO phases grows up obviously, when the particle size of precursor powder is less than 1 μm, as shown in Fig.2. It seems that a small particle size of precursor powder is beneficial to growing- up of AEC and CuO phases.

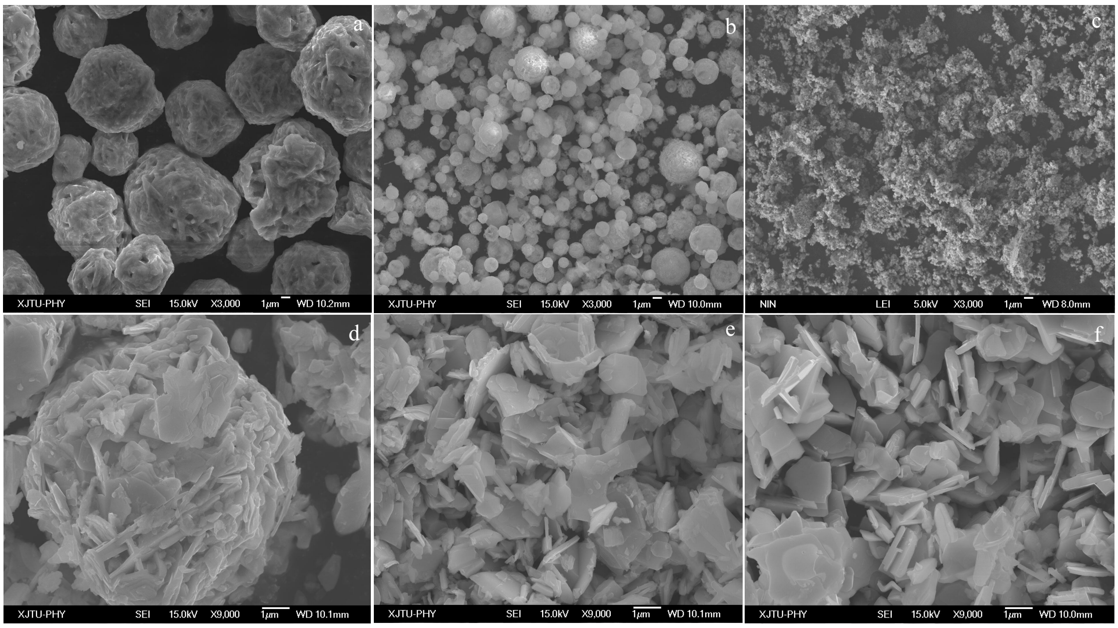

Fig.1 SEM images of original precursor powders (a~c) and precursor powders after heat treatment at 780 °C for 10 h in a flowing 0.1% O_2/ balance N_2 atmosphere (d~f): (a, d) powder 1, (b, e) powder 2 and (c, f) powder 3

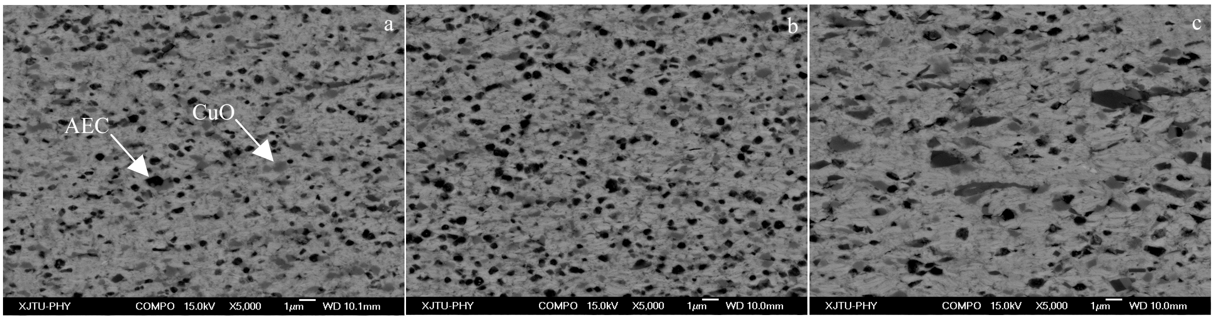

Fig.2 SEM images of bulks pressed by precursor powders after heat treatment: powder 1 (a), powder 2 (b), and powder 3 (c)

Fig.3 shows the X-ray diffraction patterns of all three powders after heat treatment at 780 °C for 10 h in a flowing 0.1% O_2/balance N_2 atmosphere. All powders have the same phase assemblages, which are composed of (Bi, Pb)-2212, AEC, CuO and a little Bi-2201 phase. It has been confirmed that Pb doping can enhance formation of Bi-2223, and Pb can form different compounds which mainly depends on the heat treatment atmosphere. Generally speaking, Ca_2PbO_4 phase can be formed when the precursor powder is heat treated in air atmosphere. However, Pb can be doped into Bi-2212 phase instead of 0.1% O_2/balance N_2 atmosphere, which is beneficial to higher texture and can be characterized by the splitting of peak about 33° and disappearing of peak of Ca_2PbO_4 at 18.4° as shown in Fig.3. It can be seen that powder 2 has minimal intensity of X-ray diffraction pattern of CuO phase, and the intensity of X-ray diffraction pattern of AEC phase increases with the decreasing of particle size of precursor powders.

P. Kameli et al introduce a method to estimate volume fraction of phases, according to X-ray diffraction patterns [9]. Volume fractions of phases were estimated by this method to study the effect of particle size on characterization of phase assemblages, as shown in Table 1. Powder 2 has the highest Bi-2212 phase content (96.31%) and the lowest CuO phase content (1.25%). AEC phase increases from 1.93% to 2.78% with decreasing of particle size. FWHM values of Bi-2212 phase (0012) decreases with the decreasing of particle size, which means more sufficient reaction. It can be also concluded that the reaction time of precursor powders decreases with the decrease of particle size.

37-filaments tapes (A, B, C) were fabricated from powder 1, powder 2 and powder 3, respectively, by a tube method. The critical currents of tapes were measured by the standard four-point probe technique in nitrogen liquid and self-field, as shown in Fig.4. The critical current of tape A is about 84 A. Tape B has highest critical current about 110 A, which is attributed to the average particle size of 2 μm of precursor powder. However, tape C has the lowest critical current about 75 A. Considering above results, it seems that CuO phase is a key parameter to affect superconductivity of Bi-2223 tape. We

Table 1 Characterization of phase composition of precursor powder after heat treatment

Sample	Bi-2212/vol%	AEC/vol%	CuO/vol%	FWHM
Powder 1	94.27	1.93	3.8	0.225
Powder 2	96.31	2.44	1.25	0.198
Powder 3	94.74	2.78	2.48	0.167

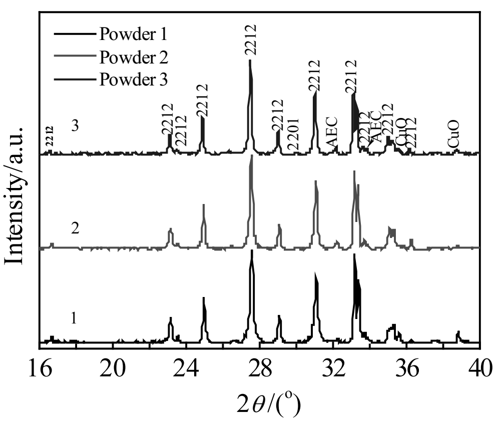

Fig.3 XRD pattern of powder 1, powder 2 and powder 3 after heat treatment at 780 °C for 10 h in a flowing 0.1% O_2/balance N_2 atmosphere

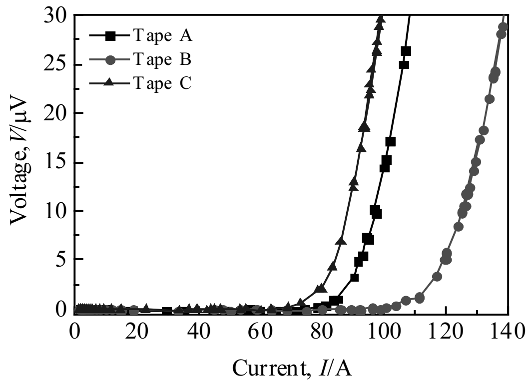

Fig.4 *I-V* curves of 37-filaments tapes (A, B, C), corresponding to powder 1, powder 2 and powder 3

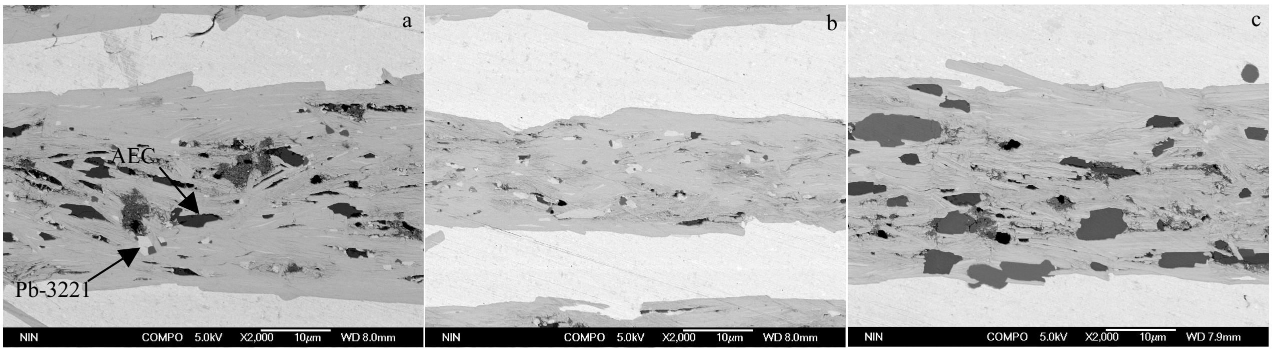

Fig.5 SEM images of three kinds of tapes: (a) tape A from powder 1, (b) tape B from powder 2, and (c) tape C from powder 3

should decrease content and dimension of CuO phase in precursor powder to achieve high critical current. Compared to CuO phase in precursor powder, AEC phase has less important effect on superconductivity of Bi-2223 tape.

Fig.5 shows microstructures of three kinds of tapes after the same heat treatment. It can be seen that there are obvious dƒ ferences among these tapes. Compared to tape C, tape A and tape B have more Pb-3221 phase, which are formed at low temperature post anneal stage of 780 °C/24 h. It is confirmed that the formation of Pb-3321 phase has a significant effect on improving critical current. What's more, tape B has the lowest AEC phases both in the dimension or content, which is unbeneficial to critical current because of elements deviation of Bi-2223 phase and blocking of current flowing path. The dimension of AEC phase of tape A is larger than that of tape B, but smaller than that of tape C. tape C has the least Pb-3221 phase, but most AEC phase compared with tape A and tape B. It seems that Pb-3221 phase and AEC phase mainly affect superconductivity of Bi-2223 tape. We should increase the content of Pb-3221 phase and decrease the content and dimension of AEC phase to obtain a high critical current, which can be achieved by adjusting the content and dimension of CuO phase in precursor powder.

3 Conclusions

1) Three kinds of precursor powders with different particle size (8, 2, <1 μm) are prepared by the spray pyrolysis method via adjusting the concentration of metal nitrates solution and ball milling.

2) After heat treatment of precursor powders with different particle sizes, the distinction is mainly in the dimension and content of AEC and CuO phase.

3) The dimension and content of AEC phase increase with the decreasing of particle size of the precursor powders. There is a minimal dimension and the lowest content of CuO phase for precursor powder with average particle size of 2 μm. Both AEC and CuO phases have the largest dimension for precursor powder with average particle size less than 1 μm.

4) For precursor powder with average particle size of 2 μm, Bi-2223 tape has the highest critical current, and it could be attributed to minimal dimension and the lowest CuO phase content in precursor powder, which further results in higher Pb-3221 and less AEC phase. With increasing or decreasing of particle size of precursor powder, the critical current decreases because of less Pb-3221 and more AEC phase.

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前驱粉粒度对 Bi-2223 带材微观结构和超导电性的影响研究

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摘　要：主要研究了前驱粉粒度对 Bi-2223 带材微观结构和超导电性的影响。通过调整溶液浓度和机械球磨，采用喷雾热分解方法制备了 3 种不同粒度的前驱粉（8，2，<1 μm）。3 种粒度的前驱粉经热处理后的相组分均为 Bi-2212，$(Sr, Ca)_xCu_yO_\delta$ (AEC)和 CuO。研究发现，AEC 相的尺寸和含量均随着前驱粉粒度的减小而增加。在平均粒度为 2 μm 的前驱粉中，CuO 相的含量和大小均为最小。采用这 3 种不同粒度的前驱粉制备的 37 芯带材，前驱粉平均粒度为 2 μm 的带材具有最高的临界电流，同时相对于另外两根带材，该带材中有最高的 Pb-3221 相和最少的 AEC 含量。研究结果表明，前驱粉粒度的大小主要影响了前驱粉中 AEC 和 CuO 两种相的大小和含量，进而导致了所制备带材的临界电流及芯丝中非超导相的变化。

关键词：前驱粉；粒度；Bi-2223；超导电性

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