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ARTICLE

Density Enhancement for the Fabrication of Bi-2212 Superconducting Tapes

Zhang Shengnan, Li Chengshan, Feng Jianqing, Xu Xiaoyan, Hao Qingbin, Zhang Pingxiang

Northwest Institute for Nonferrous Metal Research, Xi'an 710016, China

Abstract: $Bi_{2.1}Sr_{1.96}CaCu_{2.0}O_{8+\delta}$ (Bi-2212) precursor powders were synthesized with co-precipitation process, and Bi-2212 single filament tapes were fabricated with powder-in-tube (PIT) technique. Based on SEM observation, the primary grain size of Bi-2212 precursor powders was 3~5 μm. However, after sintering, Bi-2212 grains clustered together and the particle size distribution became quite wide, from 10 μm to over 180 μm. By dividing the particles into four groups with different average particle sizes, the influences of average particle size on the filament density, microstructures and current capacity of final Bi-2212 tapes were investigated. Results show that with the decrease of particle size, the filament density increases over the entire cold-working process and heat treatment process. Therefore, the intergrain connection is enhanced, which leads to the obvious increase of current capacity.

Key words: high temperature superconductor; Bi-2212; filling density; critical current density

Although many high temperature superconductor (HTS) systems have been discovered since the first report of cuprate HTS at 1986, only $YBa_2Cu_3O_7$ (YBCO)[1] and Bi-based superconductors (including $Bi_2Sr_2Ca_2Cu_3O_{10+\delta}$ (Bi-2223) and $Bi_2Sr_2CaCu_2O_{8+\delta}$ (Bi-2212))[2-4] are considered to be practically applicable HTS materials. Bi-2212, as the only HTS material, so far, which can be made into round wires with isotropic cross sections, attracts more and more attentions [5-7], because the round wires configuration can greatly simplify the winding process of cables and magnets. Meanwhile, the high upper critical field and weak field dependence of current capacity can both ensure the applications of Bi-2212 under low-temperature and high magnetic field. Nowadays, Bi-2212 insert coils for the manufacturing of high field (~30 T) magnets were proved to be practical[8-11]. Therefore, it is necessary to consider the industrial fabrication of Bi-2212 tapes and wires for further applications.

However, there are still some factors limiting the further enhancement of current capacity of Bi-2212 wires. One is the anisotropic transport properties due to the intrinsically layered structure of Bi-2212. When the magnetic field is perpendicular to the *ab* plane, the weak Abrikosov pinning leads to a rapid decrease of J_c in the low magnetic field, which greatly restricts the practical performance of Bi-2212[12-14]. Another is the bubbles existing in the filament[5]. Generally speaking, there are two sources for the bubbles formation. One is the precipitated gas during sintering[15-17]. An over-pressure process was designed to constrain the expansion of Ag sheath during sintering process[5], which greatly enhanced the current capacity of Bi-2212 wires for over 5 times. The other source is the space formed during powder packing process, which is the major contribution[6,18]. There have been many methods reported to reduce the residual bubbles to enhance the filament density. Jiang et al first introduced a cold isotropic pressure process after packing, which leads to the double of critical current density. Then by introducing a swaging process, they further enhanced both the filament density and the uniformity

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Corresponding author: Zhang Shengnan, Ph. D., Senior Engineer, Superconducting Materials Research Center, Northwest Institute for Nonferrous Metal Research, Xi'an 710016, P. R. China, Tel: 0086-29-86231079, E-mail: snzhang@c-nin.com

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of current capacity distribution[6]. Hao et al set up a device to vacuum the packed tapes before cold working process, which successfully lead to the decrease of bubbles and increase of critical current of >50%[18]. Based on the previous study above, it can be deduced that the decrease of residual gas before sintering is effective to enhance the filament density of Bi-2212 wires.

Particle size is a very important parameter for the packing process of powders and exhibits great influence on the packing density. The precursor powders synthesized by co-precipitation technique are generally composed of particles with a wide particle size distribution in different shapes. Therefore, it is hard to control the packing density and the uniformity. In this study, precursor particles were divided into four groups with different average particle sizes. The filament density change during cold working process was analyzed, and the effect of average particle size on the final current capacity of sintered tapes was discussed.

1 Experiment

$Bi_{2.1}Sr_{1.96}CaCu_{2.0}O_{8+\delta}$ precursor powders were prepared by a modified co-precipitation process[19,20] with the starting materials of Bi_2O_3, $SrCO_3$, $CaCO_3$, and CuO (> 99.9%). After a series of calcination processes in air at 800 °C/12 h, 820 °C/20 h, and 850 °C/20 h with intermediate grinding, the precursor powders were obtained. Four groups of precursor powders were divided by screen openings of 80, 240 and 400 mesh, which corresponding to the particle size of 1#: d>180 μm, 2#: 180>d>60 μm，3#: 60>d>35 μm, and 4#: d<35 μm. The precursor powders were then densely packed into Ag tubes with the outer diameter of 10 mm, and inner diameter of 8 mm. With the same drawing and rolling process step by step, single filament tapes with the thickness of ~300 μm, width of ~4 mm were obtained. The partial melting process was performed with the O_2 flowing velocity of 0.4 m^3/h, and the maximum heat treatment temperature, T_{max} of 892 °C. The tapes were kept at T_{max} for 20 min, then cooled down with the cooling rate of 5 °C/h to 840 °C with the dwell time of 20 h, and then cooled down to room temperature with furnace.

Polycrystalline X-ray diffraction (XRD) patterns of precursor powders and tapes were taken on an X-ray diffractor (XRD, Bruker D8 ADVANCED) with Cu-Kα radiation (λ=0.154 nm). The microstructure of precursor powders was observed with field-emission scanning electron microscopy (FESEM, JSM-6700F). The critical current, I_c, was measured at liquid nitrogen temperature (77 K) on a computer-aided apparatus using a DC four-probe method with the criterion of 1 μV/cm under self field.

2 Results and Discussion

The microstructure of precursor powders is shown in Fig.1. The different particle size distribution can be obviously observed. In Fig.1a and 1b, the powders are mainly composed of large clusters with spheroidicity shape. The average particle size are 330 and 91 μm, respectively, which are consistent with the screen opening size of the mesh. These clusters are formed during the sintering process, and it is hard to crush these clusters into smaller particles. In Fig.1c and 1d, small clusters in irregular shapes can be observed. The average particle sizes based on statistics results are 38 and 11 μm, respectively. Meanwhile, it is also noteworthy that regardless of the different particle sizes, all the large and small clusters are composed of small and uniform grains with the size of 3~5μm, which can be considered as the primary Bi-2212 grains.

In order to make sure the chemical and phase compositions of all the four groups are the same, so that to confirm the df ference of current capacity is not caused by these differences, X-ray diffraction patterns are taken on the four samples. As shown in Fig.2, the major phase of all the powders is Bi-2212, as marked on the patterns. There is no obvious texture of the randomly distributed particles, and no detectable secondary phase is observed. As shown in the inset no obvious shift of the patterns can be detected. Meanwhile, since the full-width at half-maximum (FWHM) values are sensitive to the primary particle size, which are almost the same for the four samples, no obvious differences of FWHM can be observed, either. Therefore, it can be confirmed that all the samples exhibit almost the same chemical and phase composition and the only difference is the cluster size as observed in SEM images.

One important influence of the particle size is the tap density. And the tap density is a major contribution to the

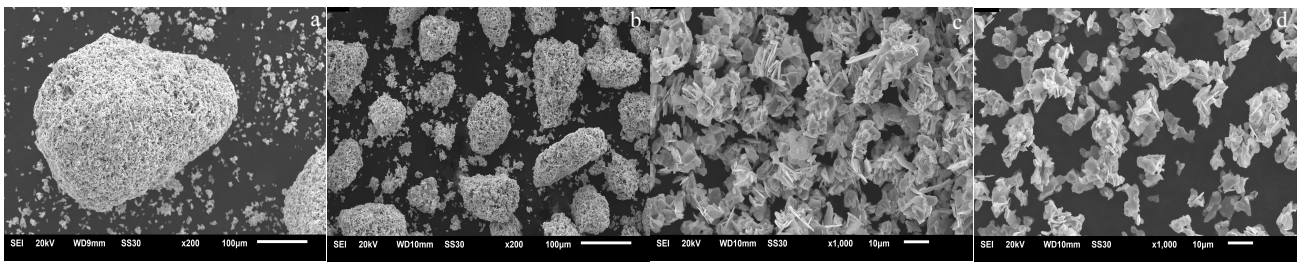

Fig.1 SEM images of Bi-2212 precursor powders with different particle sizes: (a) 1#: d>180 μm，(b) 2#: 180>d>60 μm, (c) 3#: 60>d>35 μm, and (d) 4#: d<35 μm

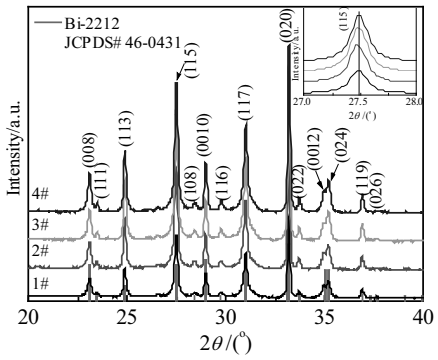

Fig.2 XRD patterns of Bi-2212 precursor powders with different particle sizes

filament density of the final wires and tapes. So it is quite necessary to analyze carefully the effects of different particle sizes and different morphology as shown in SEM images on the tap density of the precursor powders. The tap density of different powders was measured by packing and taping the precursor powders with the same weight in a graduate. As shown in Fig.3, obviously different volumes are obtained on different precursor powders. The tap densities are calculated as 0.77, 0.87, 1.00 and 1.05 g·cm^{-3}, corresponding to the precursor powders of 1#~ 4#, respectively. It is interesting to see that the tap densities of both 1# and 2# are obviously smaller than those of 3# and 4#, which could be attributed to the great difference of morphology. It is more difficult for the particles with spheroidicity shape to get high tap density than those with irregular shapes. Thus it can be concluded that higher tap density can be achieved with the precursor powders with the average particle size smaller than 40 μm.

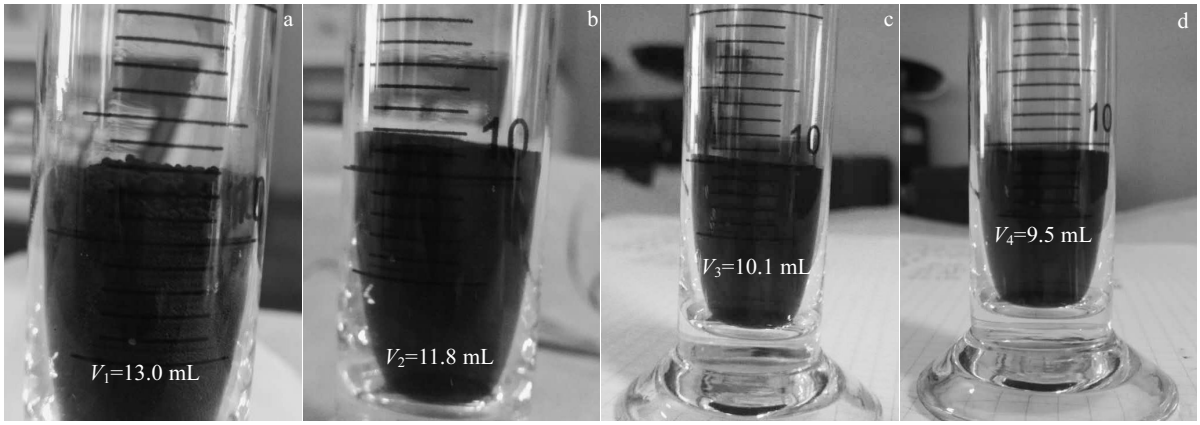

Fig.3 Taping volume of the precursor powders with different particle sizes and the same weight

Powder in tube processes are performed with different precursor powders and four tapes also numbered 1~4 # are obtained correspondingly. Filament densities are measured on the samples obtained during the cold working process: two samples after the drawing process with the diameter decreasing to 2.12 and 1.84 mm, one after the rolling process to the thickness of 4 mm, and one after the sintering process of tapes. As shown in Fig. 4, all the filament densities increase with the cold working process, which proves that the cold working process including drawing and rolling is an effective densification process. Obvious differences between the filament densities of tape 1# and 2#, and those of tape 3# and 4# can also be observed. The increase of filament density from 1# to 4# is above 20%. Generally speaking, with the decrease of particle size and the change of particle shapes, the filament density increases. While, when the average particle size is smaller than 40 μm, the increase of filament density is not quite obvious. Thus, it is indicated that the particle size exhibits an obvious influence on the packing density of tapes, when the average particle size is larger than 40 μm.

The heat treatment process for Bi-2212 wires and tapes is called the partial melting process, during which the filaments density get higher and the texture structure is formed. As shown in Fig.5, (00*l*) texture is formed in these four tapes, with only (00*l*) diffraction peaks can be observed on the patterns. There is no obvious secondary phase detected. And there is no shift of the peaks either, which suggests that there is no phase and chemical composition difference appearing during heat treatment process. On the other hand, all the patterns are fitted and the FWHM values are calculated as shown in the inset of Fig.5. It can be noticed that the FWHM values of all the diffraction peaks decrease with decreasing particle sizes, which suggests a larger Bi-2212 grain size and a better crystallization with smaller particle size. Considering that before the heat treatment process, obvious filament density enhancement can be observed, it is easier for denser filament to crystallize. The existence of bubbles tends to interfere the growth process of grains. Therefore, larger grains can be achieved with higher filament density, which then lead to strong intergrain connections, and will be beneficial to the current transport process.

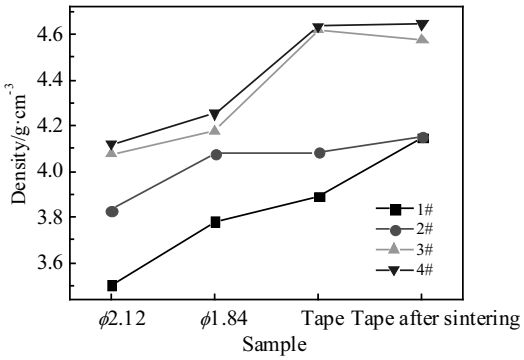

Fig.4 Filament density values of the tapes with different precursor powders during different cold working process

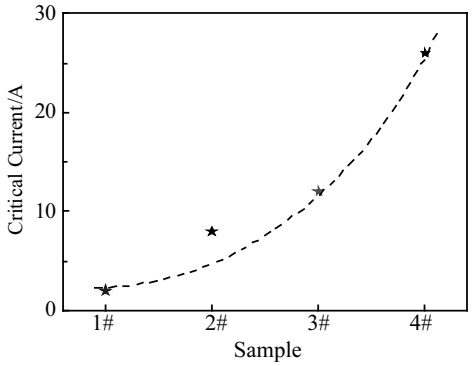

Fig.6 Critical current values of different Bi-2212 tapes after the same PIT and heat treatment process

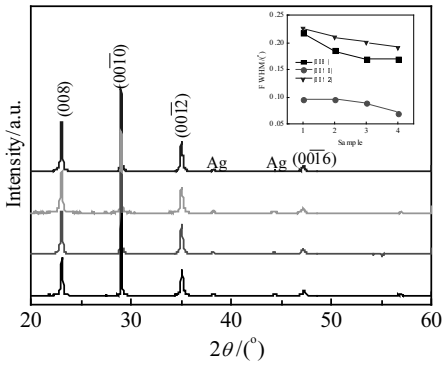

Fig.5 XRD patterns of sintered Bi-2212 tapes with the FWHM values of different peaks shown in the inset

After the partial melting process, the critical currents of all the four tapes are measured with transport method, in order to study the influences of filament packing density on the current capacity of Bi-2212. As shown in Fig.6, the critical currents of all the four tapes are plotted, which increase monotonously with decreasing average particle size. The maximum critical current of ~26 A is achieved on the 4# tape with the smallest average particle size. This change is consistent to the change of FWHM values in Fig.5 and filament densities in Fig.4. Therefore, it can be deduced that with the increase of filament density and enhancement of intergrain connections, the current capacity can be enhanced by tuning the particle size of precursor powders. Thus it is quite necessary to optimize the average particle size before packing process of precursor powders for industrial fabrication of Bi-2212 superconducting wires.

3 Conclusions

1) Bi-2212 precursor powders are divided into four groups with different average particle sizes of ~11 to ~330 μm and the influences of particle size on the packing process and the current capacity of final tapes are discussed.

2) With the decrease of particle size, the packing density of the powders increases during the entire cold working and sintering processes.

3) Higher texture could also be achieved with smaller particle size, and then the intergrain connections can be enhanced due to the less pores.

4) A higher critical current is obtained on the tapes with the smallest particle size.

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增强芯丝密度对 Bi-2212 高温超导带材性能的影响

张胜楠，李成山，冯建情，徐晓燕，郝清滨，张平祥
(西北有色金属研究院，陕西 西安 710016)

摘　要：采用共沉淀工艺制备Bi-2212前驱体粉末，并通过粉末装管法(PIT)制备了Bi-2212单芯带材。通过对装管粉末颗粒尺寸的控制，分别获得了具有不同芯丝密度的带材，并对其所引起的超导带材的微结构和超导载流性能的变化进行了系统的分析。SEM照片显示，前驱体粉末是由3~5 μm的初级颗粒组成的，但是由于烧结过程中发生硬团聚，有大量的大尺寸团簇颗粒产生，颗粒尺寸由10到180 μm不等。通过过筛将前驱体粉末按照颗粒尺寸分为4组，分别进行装管。结果表明，随着平均颗粒尺寸的减小，在冷加工和热处理过程中带材的芯丝密度均有明显增加。因此最终带材的晶间连接性较强，从而获得带材载流性能较高。本研究可以作为装管工艺优化的一个重要理论依据，为Bi-2212超导线材性能的进一步提高奠定基础。

关键词：高温超导材料；Bi-2212；装管密度；临界电流密度

作者简介：张胜楠，女，1983 年生，博士，高级工程师，西北有色金属研究院超导材料研究所，陕西 西安 710016，电话：029-86231079，E-mail: snzhang@c-nin.com