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Research note

Investigation of particle size on the phase formation and microstructure of Bi-2223 /Ag wire

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Abstract

The effect of particle size and the heat treatment on precursor powder and subsequent phase formation of silver tube Bi-2223 wire has been studied. First, we have prepared a series of $Bi_{1.66}Pb_{0.34}Sr_2Ca_2Cu_3O_y$ samples with different sintering temperatures and studied the phase formation in these samples. Second, different particle size distributions were obtained by grinding powders of best formed Bi-2223 phase samples. PIT technique has been used to make wires of Bi-2223 phase using silver tube. The effects of precursor heat treatments and different particle size were identified by combination of XRD, SEM EDX, LPSA (Laser Particle Size Analyzer) electrical resistivity and Ac-susceptibility measurements technique. It has been shown that the small particle size promotes the formation of Bi-2223 phase and the critical current densities tend to increase when the size of sintered powder of Bi-2223 phase used in the formation of wire, decreases.

Keywords: Bi-2223 superconductor wires, particle size effect, precursor powders, synthesis

1. Introduction

With progressive improvements in current-carrying capacity and other properties, silver sheathed $(BiPb)_2Sr_2Ca_2Cu_3O_{10}$ (Bi-2223) superconducting wires and tapes are becoming attractive in power application such as transmission cables, transformers, current limiter, motors and generators. The Bi-2223 system possesses a very complex phase diagram and the synthesis of a single-phase of Bi-2223 phase is difficult due to the problem of intergrowths. The phase field was further complicated due to addition of extra elements, Pb, which is necessary to stabilize the Bi-2223 phase [1, 2].

From the point of view of practical applications, power transmission with negligible loss is an area where high-T_c superconductors have a great potential. The proposed devices are tapes and wires, which are usually manufactured in dense tube method where the precursor powder is densely filled into Ag tubes and drawn into the necessary shape. The products are then subjected to different mechanical and thermal processes.

Since the discovery of Bi-2223 phase superconductor, great progress has been made in enhancing the transport properties of this high-T_c superconductor. However, the limitations of the Bi-2223 phase superconductor applications are the intergrain weak links and weak flux pinning capability. It was shown that one of the major current limiting factors in Bi-2223 system is the presence of the residual secondary phase [3]. Sample analysis revealed that the Bi-2201 phase is located mainly between the superconducting grains, preventing the super-current [3]. It was shown that Jc was raised as the Bi-2212 phase decreased in Bi-2223 tapes [4]. Deng et al. [5], using SEM, have observed that the residual Bi-2212 phase is situated at grain boundaries of Bi-2223 phase.

The authors recently reported on the effect of sintering temperature on the intergranular properties of polycrystalline of Bi-2223 phase. Our pervious results on adding Ag to a bulk BSCCO superconductors show a significant improvement in the J_c in these compounds. In this paper we report on the particle size effect on the Bi-2223 phase polycrystalline and wire samples, using PIT technique and silver tube. The precursor powders of Bi-2223 superconductor phase were prepared using the optimum sintering condition obtained in our previous work [6].

2. Experimental procedure

$Bi_{1.66}Pb_{0.34}Sr_2Ca_2Cu_3O_y$ powder samples were prepared using conventional solid state reaction method. Extra pure powders of Bi_2O_3, $SrCO_3$, PbO, $CaCO_3$, and CuO were well mixed in stoichiometric proportions calcinated at 810°C for 24h, three times. In order to obtain a series of powders with different particle sizes, the calcinated powders were subjected to high energy ball milling in a

Table 1. Volumes fraction of Bi-2201, Bi-2212, and Bi-2223 Phases

Sample	Bi-2223 (%)	Bi-2212 (%)	Bi-2201 (%)
S1	70	20	10
S12	81	10	9
S24	84	9	7
S48	86	8	6
S88	87	7	6

Table 2. Powder grinding time and average particle size and dimension of the wires.

Sample	Grinding time (h)	Average particle size (μm)	Dimension of wires(mm)
B24	24	2.63	-
B48	48	2.52	-
B72	72	1.93	-
BAG	-	4.55	-
WB24	24	-	0.7×0.6
WB48	48	-	0.8×0.7
WB72	72	-	0.7×0.7
WBAG	-	-	0.7×0.6

cylindrical bowl, with seven 10 mm agate balls for different time intervals, from 1 to 88 h, at a rotational speed of 400 rpm using a planetary ball mill. The resulting powders were repeletized and sintered for 180 h at 865 °C . The sintered ceramic samples made from 1h, 12h, 24, 48h, and 88h milled precursor powders were named S1, S12, S24, S48, and S88, respectively. The detail of preparation is given in[6].

As a result of our previous work [7] a test sample of Bi-2223/Ag (20% Ag doped Bi-2223 phase) have been prepared with the same thermal process and ground for 1h.

A group of powders, namely 24h, 48h, 72h, and Ag doped ground powders have been selected. These powders were taken as powders B24, B48, B72 and BAG and were processed by powder-in-tube method with the same processing parameters. High purity silver tube was used, the dimensions of which were 6mm externally and 4mm internally in diameter. The precursor powder was loaded into the silver tube and densified by means of a steel piston. The powder density inside the tubes before deformation was 50% of theoretical density. The tubes were closed, and were deformed using two two-hammer swaging machines, down to a diameter of about 2mm. The above processes took place in two stages with an intermediate sintering of the tubes in 865°C for 180h. The extortion method was used to further decrease the diameter of above wire down to about 0.5mm. The wires made from powders of B24, B48, B72 and BAG, were named WB24, WB48, WB72 and WBAG, respectively. The 0.7 mm diameter wires were sintered in air at a temperature of865° C for 180h.

The Ac susceptibility measurements were performed, down to 77K, using a Lake Shore susceptometer, Model 7000. The resistivity measurements were carried out by the four probe method, using modified Lake Shore susceptometer, down to 77K, and a Leybold Closed Cycle refrigerator, down to 10K. The microstructures of the sintered powder and selected wire samples were examined by scanning electron microscopy (Phillips XL30). The critical current density was measured by the standard four-probe technique.

3. Results and Discussions

The X-ray diffraction patterns of samples which are sintered for 180 h at 865 °C are reported on [8]. Using the technique of "all peaks of major phases" (namely, Bi-2223, Bi-2212, and Bi-2201) [9, 10], we have estimated the volume fractions of these phases. The estimation of fractional volumes of Bi-2223, Bi-2212, and Bi-2201 phases are given in Table 1. As one can see clearly, by the extension of grinding time, the volume percentage of Bi-2223 phase will increase from 70% to 87%, but the volume fractions of Bi-2212 and Bi2201 phases are decreased from 20% to 7% and from 10% to 6%, in sintered samples, respectively. From the XRD pattern one can assume that the small particle size promotes the formation of Bi-2223 phase. Due to the fact that a homogeneous and small particle size powders enhance the reaction in sintering process, this effect will increase the speed of phase transformation and the formation of Bi-2223 phase.

Within the sensitivity of our XRD machine, there was almost no noticeable trace of impurity phase in the XRD patterns. With the extension of grinding time, the powder became finer and the XRD peaks became broader. The average particle sizes of selected powders and the dimension of them are listed in Table 2.

Figure 1 shows the results of SEM observation on the microstructure of the wires sintered in air at 865°C for 180h. These micrographs show a c-axis oriented and dense structure of Bi-2223 plate-like grains. But, the WB72 sample is highly c-axis oriented and is denser than the others. As pointed out by R. Yamamoto [11], Colds Isostatic pressing (CIP) will improve the superconductivity of the Bi-2223 phase, but a small

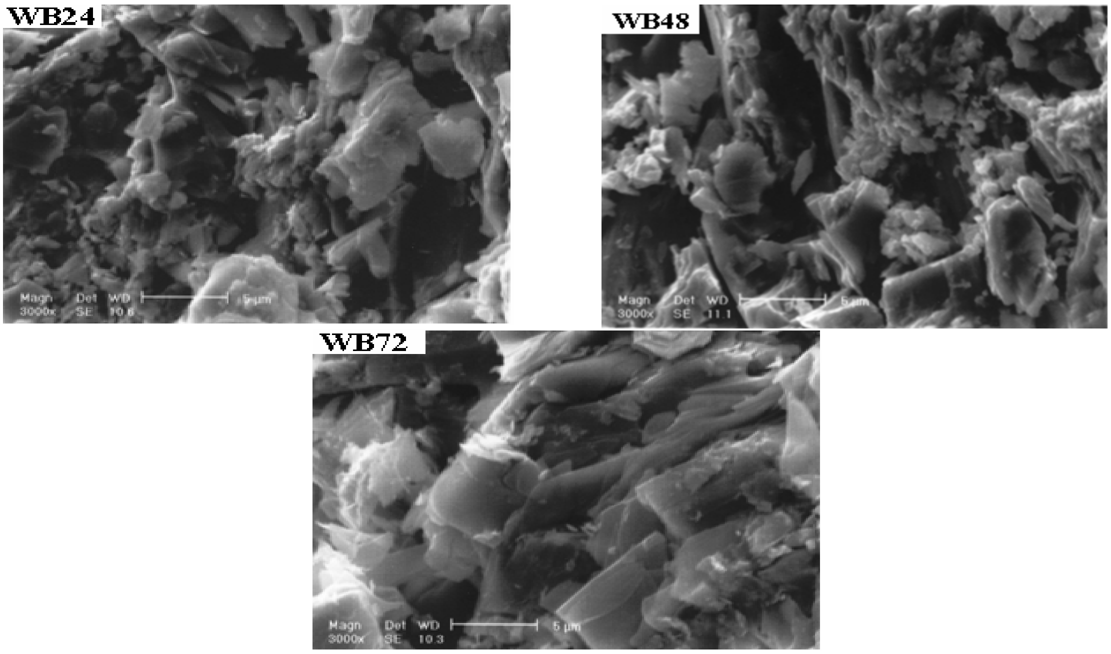

Figure 1. The SEM micrographs of WB24, WB48, and WB72 samples .

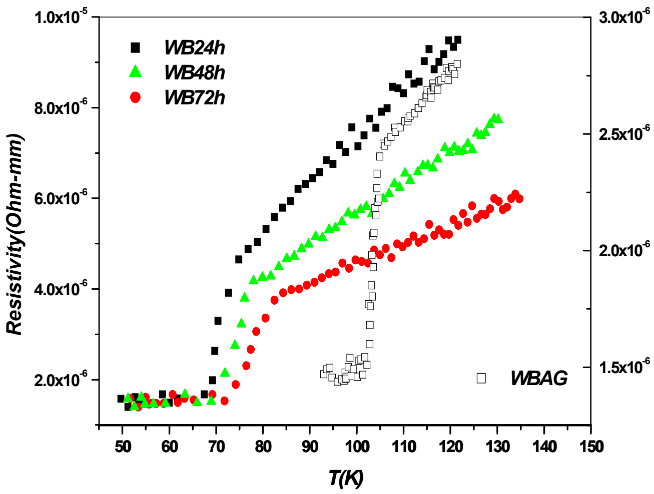

Figure 2. Temperature dependence of resistance of WB24, WB48, WB72 and WBAG samples at 30mA.

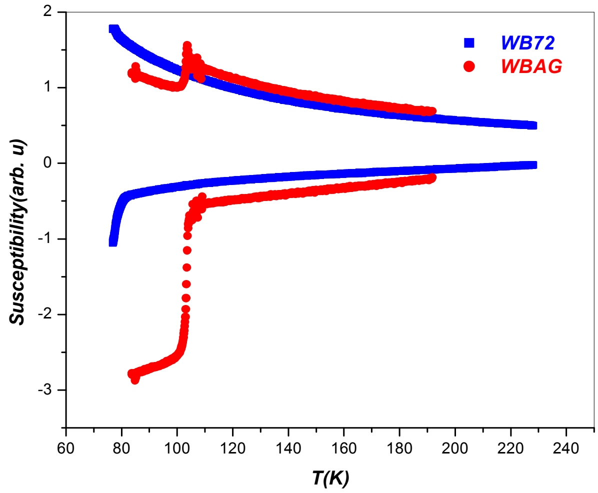

Figure 3. Temperature dependence of Ac susceptibility for WB72 and WBAG samples in ac amplitude 0.8 A/m at frequency of 333.3 Hz

particle size also enhances this process

Figure 2 shows the results of resistivity measurements for WB24, WB48, WB72 and WBAG. As the results of these measurements clearly show, all samples show a metallic behavior above theirs transition temperature. But with the extension of the grinding time, the normal resistivity of wires have dropped significantly. The transition temperature of wires seems to improve with decreasing the particle size of precursor powders.

Figure 3 shows the temperature dependence of real, χ', and imaginary, χ", parts of Ac susceptibility of WB72 and WBAG samples in ac amplitude 0.8 A/m at frequency of 333.3 Hz. It is to be noted that WB24 and WB48 didn't show any sign of transition in the Ac susceptibility measurements, down to 80K. This results show that the transition temperature of the test sample, namely Bi-2223/Ag series, is about 100K. To confirm this, we have carried out the resistivity measurement on this sample. The result of this measurement is presented in figure 2.

Finally, the results of direct measurements of critical current density of WB72 and WBAG at 77K are shown in figure 4. The measured values of J_c for WB72 and WBAG samples are about 1000A/cm^2 and 1050 A/cm^2, respectively. As we have shown in our previous work [7], addition of Ag up to 20% will increase the J_c of Bi-2223 phase. It has been reported by numerous authors, including us [7], that the Ag reside between the grains of Bi-2223 phase as a separate phase and is no interrupted by the formation of Bi-2223 phase. More work needs to

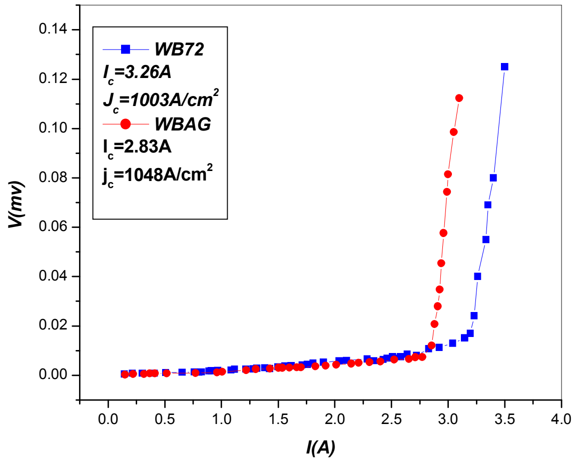

Figure 4. DC voltage-current characteristics of WB72 and WBAG samples.

be done to elaborate on the effect of particle size and heat treatments in the presence of silver in the Bi-2223 superconductor phase.

4. Conclusion

The phase formation, microstructure evolution, and critical current density are sensitive to the precursor particle size distribution. Our results indicate that the transition temperature and the critical current of wire with smallest particle size was most sensitive to the annealing temperature, and led to the fastest formation of Bi-2223 phase. So, it is suggested that the small particle size promotes the formation of the liquid phase and accelerates the melting of intermediate phase. Our new studies on Bi-2223/Ag wire shows that the presence of Ag enhances the transition temperature and the critical current of Bi-2223 phase superconductor wire. More investigation of the effect of precursor particle size on the critical current density of Ag doped Bi-2223 phase is underway.

Acknowledgments

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References

1. P J Ford and J A Saunders, *Contemp. Phys*. **38** (1997) 63.
2. P Majewski, S Kaesche, F Aldinger, *J. Am. Ceram. Soc*. **80** (1997)1174.
3. Iwasa, *Case study in superconducting magnet*: Design and operational issues. Plenum press (1994) 203-236.
4. J Jiang, X Y Cai, J G Chandler, A A Polyanskii, Y Yuan, E E Hellestier, *IEEE. Trans. Appl. Supercond*. **13** (2003) 3018.
5. H Deng, P Hua, W Wang, C Dong, H Chen, F Wu, H Wang, Y Zhou, G Yuon, *Physica* C **339** (2000) 181.
6. P Kameli, H Salamati, M Eslami, *Solid State Commu*. **137** (2006) 30.
7. H Salamati, A Babaei and M Safa, " Investigation of week link and the role of silver addition on YBCO and BSCCO superconductors ", *Proceeding of First Regional Conference on Magnetic and Superconducting Materials, world Scientific*, Tehran, Iran (1999) 181-188.
8. I Abdolhosseini, H Salamati, P Kameli, *IJPR* **6** (2006) 169.
9. I V Driessche, A Buekenhoudt, K Konstantinov, E Brueneel, S Hoste, *Appl. Supercond*. **4** (1996) 185.
10. I Karaca, S Celebi, A Varilci, A I Malik, *Supercond. Sci. Technol*. **16** ((2003) 100.
11. R Yamamoto, S Yoshizawa, S Hirano, Y Hishinuma, A Nishimura, A Matsumoto and H Kumakura, *Physica* C **378-381** (2002) 664-668.