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Formation of nucleation centers for vortices in Bi-2223 superconducting core by dispersed Sn nanoparticles

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ABSTRACT

In this study, we investigate the effect of the Sn diffusion at different annealing temperature in the range of 650–850 °C on the microstructural, electrical, mechanical and superconducting properties of the $Bi_{1.8}Pb_{0.4}Sr_{2.0}Ca_{2.1}Cu_{3.0}O_y$ materials prepared by the standard solid state reaction method with the aid of bulk density, dc resistivity (ρ-T), transport critical current density (J_c), X-ray diffraction (XRD), scanning electron microscopy (SEM) and Vickers microhardness (H_v) measurements. It is found that all the properties given above are significantly dependent upon both the Sn nanoparticles inserted in the Bi-2223 superconducting matrix and diffusion annealing temperature. Namely, the dc resistivity measurements indicate that all the samples exhibit the metallic behavior as a result of the electron–phonon interaction in the Bi-2223 crystal structure or logarithmic divergence in density of states at the Fermi level. Further, the normal state resistivity decreases with the enhancement of the annealing temperature owing to the increment of the metallic connection between the superconducting grains due to the optimization of the hole density and possible changes in the lattice vibration. Likewise, onset and offset critical transition temperature values tend to enhance with the diffusion annealing temperature, confirming not only the improvement of crystallinity and especially connectivity between the superconducting grains but the increment in the average grain size and mobile hole concentration in the $Cu–O_2$ slabs of the Bi-2223 system, as well. The decrement in the degree of the broadening stems from the degradation of the porosity, grain boundary resistivity and grain boundary weak-links in the system. Moreover, the results of J_c show that the Sn inclusions (highly dispersed nanoparticles) form the effective flux pinning centers for the vortices in the crystal structure, and tightly bound to these nucleation centers. Thus, the J_c value steeply increases from 852 A cm^{-2} to 4307 A cm^{-2} with the diffusion annealing temperature. Similar findings are valid for the microstructural characteristics obtained from the SEM images belonging to the samples. The annealing temperature of 850 °C develops considerably the surface morphology and interaction between the superconducting grains. Hence, the sample annealed at 850 °C for 24 h exhibits the smoothest and densest surface morphology. Similarly, the bulk density analysis illustrates that the densities regularly rise from 5.582 to 6.102 g/cm^3 with increasing the annealing temperature, confirming that more and more Sn inclusions penetrate into the superconducting grains or over grain boundaries in the crystal structure. This is associated with the enhancement of the connectivity (interaction) between the superconducting grains. At the same time, the results of the XRD experiments display that all the samples composed of two superconducting phases (Bi-2223 and Bi-2212) present the polycrystalline superconducting phases but in different intensity of diffraction lines. With increasing the annealing temperature up to 850 °C, the intensity of diffraction lines belonging to the high-T_c phase (Bi-2223) becomes stronger and so the best superconducting properties are observed for the sample prepared at the annealing ambient of 850 °C for 24 h, being even favored by the largest (lowest) cell parameter c (a). Furthermore, Vickers microhardness measurements conducted at different applied indentation test load ($0.245\ N \leqslant F \leqslant 2.940\ N$) demonstrate that all the Bi-2223 ceramics obey the typical Indentation Size Effect (ISE) behavior, and the mechanical properties increase with the diffusion annealing temperature. The long and short of it is that the Sn inclusions and especially diffusion annealing temperature up to 850 °C are favorable for the formation velocity of Bi-2223 phase.

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1. Introduction

The research on the high temperature superconducting (HTCS) materials has enhanced for last decades due to their remarkable high transition temperature, smaller power losses, high magnetic field carrying capacity, optical and electronic properties [1–6]. Of the HTCS, Bi–Sr–Ca–Cu–O (BSCCO) materials have a special place with their simple preparation procedure, high critical transition temperature and especially good mechanical performance [7,8]. These important properties make them use for the theoretical studies and practical applications [9–11].

Moreover, Bi-based superconducting materials are composed of the layered structures that make the mobile charge carriers confine to the $Cu–O_2$ layers [12,13]. The carriers enable the crucial properties such as onset and offset critical transition temperatures to appear in the superconducting material for the potential applications in technology and industry. In the Bi-based superconducting parents, there are three basic superconducting phases with respect to the number of $Cu–O_2$ (consecutively stacked) planes in the unit cell. The critical transition temperature values belonging to the phases change from to 20 K to 110 K. The more $Cu–O_2$ stacked planes a specimen has, the more critical temperature the material presents.

The transport critical current density that is in the charge of the practical applications for the superconducting samples is more important parameter as compared to the transition temperature. As well known the critical current density is lower level for the Bi-based superconductors; on the other hand, any induction of a disorder in the crystal structure is enough to improve the pinning of vortices in the crystal structure [14,15]. As a result, the circulated current in the material generates less energy dissipation owing to the formation of the nucleation centers for the vortices. In this current work, we try to form the effective flux pinning (nucleation) centers in the Bi-2223 bulk samples with the aid of highly dispersed nanoparticles (Sn individuals) which bind tightly the centers with each other. We analyze the role of the diffusion annealing temperature on the electrical, microstructural, mechanical and superconducting properties of the Sn diffused Bi-2223 bulk superconductors prepared by standard solid-state reaction technique with the aid of the standard characterization measurements such as the bulk density, dc electrical resistivity, transport critical current density, scanning electron microscopy, X-ray diffraction and microhardness experiments. All the evidences indicate that more and more Sn impurities enter into the grains or over grain boundaries in the Bi-2223 superconducting matrix, and form the nucleation centers for the vortices in the crystal structure. The long and short of it is that the diffusion annealing temperature is favorable for the Bi-2223 superconducting phase.

2. Experimental procedure

The poly-crystallized $Bi_{1.8}Pb_{0.4}Sr_{2.0}Ca_{2.1}Cu_{3.0}O_y$ (Bi-2223) superconducting samples are prepared in air by the standard solid-state reaction route from high-purity (99.9%) starting chemicals of Bi_2O_3, PbO, $SrCO_3$, $CaCO_3$ and CuO (Alfa Aesar Co., Ltd.). Prior the milling process, all the chemicals containing the oxides and carbonates are weighed in stoichiometric proportion by means of an electronic balance. The powders are mixed in a grinding machine for 15 h to obtain homogeneous mixture of ingredients. The resultant powder is filled into a rectangular bar of $1.5 \times 0.5 \times 0.2$ cm^3 in the air atmosphere under a force of 300 MPa at room temperature. The pelletized powders are exposed to the annealing process at the constant temperature of 850 °C for the duration of 24 h. Both the heating and cooling rates of the temperature are adjusted as 5 °C/min. The evaporation of the Sn nanoparticles on one face of the specimens is conducted by an AUTO 306 vacuum coater (EDWARDS) under pressure of 2×10^{-5} Torr by use of the SnO_2 chemical (Alfa Aesar Co., Ltd. 99.99% purity). Thickness of the Sn impurities is measured to be about 30 μm. After the evaporation process, the color of silvery-white residues on the (blackish) solidified ceramics confirms that the Sn diffusion is carried out perfectly. Additionally, we check that the Sn layer attached or not to the Bi-2223 superconducting materials by visual inspection and the mechanical damage method with the aid of a razor blade. Finally, the Sn layered superconducting ceramics are annealed at different temperatures such as 650, 700, 750, 800 and 850 °C. In forthcoming sections, the superconducting ceramics will be presented as Sn-650, Sn-700, Sn-750, Sn-800 and Sn-850, respectively. The standard experimental methods such as dc resistivity, transport critical current density, X-ray diffraction, scanning electron microscopy and Vickers microhardness measurements are performed for the identification of the samples prepared in this work.

The electrical properties of the samples are investigated by dc resistivity versus temperature measurements using 5 mA dc current throughout the specimen surface in the He gas contact (closed-cycle) cryostat from CRYO Industries in the temperature range from 90 K to 130 K. The four-point contacts are covered with the silver paint. A Keithley 220 programmable current source and a Keithley 2182A nano-voltmeter system are used for the standard four-probe measurements. Similar to the dc electrical resistivity measurements, the transport critical current (J_c) experiments in self-field are exerted using four-probe method in the home-made system at the constant temperature of 77 K in zero magnetic field. The experimental data obtained are recorded by Labview software. The J_c values are determined for a standard criterion of 1 μV/cm.

X-ray diffraction measurements are carried out using a Rigaku Multiflex + XRD diffractometer with Cu Kα target providing a monochromatic beam with wavelength of 1.54 Å in the range of 2θ = 4–60° at a scan speed of 3°/min and step increment of 0.02° at room temperature in air atmosphere. The cell parameters and volume fraction values are extracted from the XRD patterns. The accuracy in determining the lattice parameters (a and c) is found to be about ±0.01 Å. Besides, the average grain sizes of the samples prepared are computed by the Scherrer–Warren approach.

Moreover, the surface morphology, crystallinity, grain size distribution and connectivity between the superconducting grains in the Sn diffused Bi-2223 bulk superconductors are determined using a Jeol scanning electron microscope (SEM) JEOL 6390-LV, operated at 20 kV, with a resolution power of ≈3 nm.

As for the mechanical properties of the samples, the microhardness measurements are exerted on the sample surfaces at room temperature in the air atmosphere to using a digital microhardness tester (SHIMADZU, HVM-2). The indentation test load is applied for the duration of 10 s in the load range of 0.245–2.940 N. The measurements are performed at different regions of the surfaces due to the fact that we do not let the indentations overlap. Moreover, the Vickers microhardness, elastic modulus, yield strength and fracture toughness values being in charge of the technological and industrial applications are deduced from the microhardness curves.

3. Results and discussion

3.1. XRD examinations

In the present study, X-ray diffraction examinations of the samples prepared are conducted to discuss how the texturing (crystal plane alignment), crystal structure, phase purity, crystallite (grain) size and lattice parameters change with the Sn individuals in the Bi-2223 superconducting system. One can see the XRD patterns depicted in Fig. 1. The characteristic peaks belonging to the Bi-2212 and Bi-2223 superconducting phases are marked by H ($h\,k\,l$) and L ($h\,k\,l$) Miller indices. According to the curves in the figure, each sample presents the polycrystalline superconducting Bi-2223 and Bi-2212 phases within the variable intensity of diffraction lines. However, it is to be mentioned here that the characteristics peak intensities corresponding to the Bi-2223 phase improve regularly with the enhancement of the diffusion annealing temperature up to 850 °C as a consequence of the improvement in the texturing (crystal plane alignments) and crystallinity. This is attributed to the fact that the velocity of the Bi-2223 phase formation increases with the annealing temperature. Therefore, the characteristics peaks symbolizing the Bi-2223 phase such as H(002), H($00\underline{10}$) and H($00\underline{12}$) increase considerably, leading to the increase in the degree of the high phase. The change in the relative percentages of the high and low phases can be determined by means of the following equations (see Fig. 2).

$$f_{(2212)} = \frac{\sum I_{H(h\,k\,l)}}{\sum I_{H(h\,k\,l)} + \sum I_{L(h\,k\,l)}} \tag{1}$$

$$f_{(2201)} = \frac{\sum I_{L(h\,k\,l)}}{\sum I_{H(h\,k\,l)} + \sum I_{L(h\,k\,l)}} \tag{2}$$