

Intergranular dissipation processes induced by nanodefects in (Bi,Pb):2223 HTS superconductor

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By using solid state reaction method, a lot of (Bi,Pb):2223 high temperature superconducting materials were obtained. The effects of partial double substitution for Cu by 3d elements on phase purity and the dynamic of intergranular vortices have been investigated by using X-ray diffraction and a.c.magnetic susceptibility measurements function of temperature and a.c.field amplitude. The effect of 3d ions concentration and the nanodefects located at grain boundaries on the vortex pinning was characterised quantitatively by using the temperature dependence of imaginary part of a.c. magnetic susceptibility for a.c. amplitudes up to 1000 A/m.

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1. Introduction

The Bi:2223 phase has attracted considerable interest due to its higher T_c and the potential for applications.

One of the reasons for the low critical current densities J_c is the granular nature of this sintered HTS compound. Extensive studies are still continuing to improve the critical current density by optimizing the processing for Bi:2223 [1,2].

The competition of intra-and intergranular conductivity is strongly temperature dependent and is differently affected by 3d ions which substitute in Cu positions [3,5]. It is difficult to prepare single phase material for Bi:2223 phase. The Bi:2212 was frequently observed as a major impurity phase in Bi:2223 samples because of its greater thermodynamic stability with respect to the Bi:2223 phase. The partial substitution of Pb in Bi sites enhances chemical stability and promotes the formation of the 2223 phase [3].

A.C. susceptibility measurements $\chi = \chi' + i\chi''$ are very useful for characterizing the HTS [7,8]. Below T_c , the sharp decrease in the real part $\chi'(T)$ is a manifestation of diamagnetic shielding and the peaks in the imaginary part $\chi''(T)$ represents the A.C. losses. The effect of 3d element substitution for Cu in (Bi,Pb):2223 ceramic superconductor by using a.c. susceptibility measurements shows that the intergrain pinning force density and J_c are influenced by the nature of 3d ions [9,10].

The parameters obtained from a.c. susceptibility data depend on the sample composition as well as the ceramic processing variables for the (Bi,Pb):2223 system [11,12].

The magnetically modulated microwave absorption investigation of (Bi, Pb):2223 phase doped by Li was studied in [13]. A slight increase of T_c have been observed.

In this paper we report the effect of codoping Zn and Fe as impurities in the Cu position of (Bi,Pb):2223 superconductor on the intergranular properties of ceramic system, by using AC susceptibility measurements as a function of temperature and AC field amplitude.

2. Experimental

The complex $(\text{Bi}_{1.8}\text{Pb}_{0.46})\text{Sr}_{1.88}\text{Ca}_{2.06}(\text{Cu}_{1-x-y}\text{Zn}_x\text{Fe}_y)_3\text{O}_z$ ($x=0.00;0.02;0.05$ and $y=0.00;0.01$) samples were prepared by the conventional solid state reaction method of appropriate amounts of the metal oxides and carbonates of 99.99% purity. Appropriate amounts of Bi_2O_3 , PbO , SrCO_3 , CaCO_3 , and CuO were mixed in agate mortar and calcined at 800 °C for 36 hours. The calcinated powder was pressed into pellets and presintered at 845 °C for 200 hours and finally sintered at 850 °C for 150 hours. High temperature heat treatment has been demonstrated to be an effective way to enhance the yield of Bi:2223 phase in both Pb-substituted and Pb-free samples, because this procedure could convert Bi:2212 in the Bi:2223 phase [14-16].

X-ray diffraction (XRD) analysis was performed by reflection with a Siemens D5000 system working in Bragg-Brentano $\theta - 2\theta$ geometry. The X-ray generator was equipped with a Co anticathode and it was using $\text{Co}(K_\alpha)$ radiation ($\lambda = 0.17909 \text{ nm}$).

Powder XRD measurements were made in an effort to check the phase purity as well as to determine the crystal structure and lattice parameters of the phases. The measurements were conducted in a $\theta - 2\theta$ -scan mode and the step angle being 0.02°.

To simplify the discussions, the samples with $x=0.02$ Zn; $y=0.01$ Fe and $x=0.05$ Zn; $y=0.01$ Fe are noted AFeZn_2 and AFeZn_5 respectively. All the diffraction peaks for the

undoped sample ($x=y=0.00$) can be indexed to the Bi:2223 phase with the base-centered orthorhombic structure (lattice parameters $a = 0.5408$ nm, $b = 0.5420$ nm and $c = 3.7075$ nm). Similar results were reported for (Bi,Pb):2223 samples with $x=0.02$ Ca and $y=0.01$ Fe in reference [17]. The fact that no trace of the Bi:2212 phase can be detected from the powder XRD pattern indicate a nearly complete conversion of Bi:2212 into Bi:2223. XRD indicate that the Bi-2223 orthorhombic phase is the major phase in both AFeZn_2 and AFeZn_5 samples. Moreover, a Bi-2212 phase is present in the sample AFeZn_5 ($x=0.05$ Zn), in low proportions.

The real (χ') and imaginary (χ'') parts of the AC susceptibility were simultaneously collected with a Lake Shore Model 7000 AC susceptometer. The measurements were performed at a frequency of 1000Hz as a function of temperature at fixed AC magnetic field amplitude (H_{ac}) in a range from 0.4 to 800 A/m.

3. Results

Fig. 1 and Fig. 2 show the $\chi''(T)$ behavior for (a) $x=0.00$, $y=0.00$, (b) $x=0.02$ Zn, $y=0.00$ and (c) $x=0.02$ Zn, $y=0.01$ Fe samples at different AC field amplitudes H_{ac} ranging from 10 A/m to 100 A/m and from 200 to 600 A/m, respectively. The end of the diamagnetism in $\chi''(T)$ correspond to the intragrain critical temperature T_{cG} . The T_{cG} values are 109.5 K in $x=0.00, y=0$; $T_{cG} = 106.5$ K in $x=0.02$ Zn, $y=0$ and $T_{cG} = 102.5$ K in $x=0.02$ Zn, $y=0.01$ Fe respectively. In all samples, the imaginary part $\chi''(T)$ exhibit a single peak at T_p , which indicate the maximum hysteresis losses due to the motion of intergranular (Josephson) vortices.

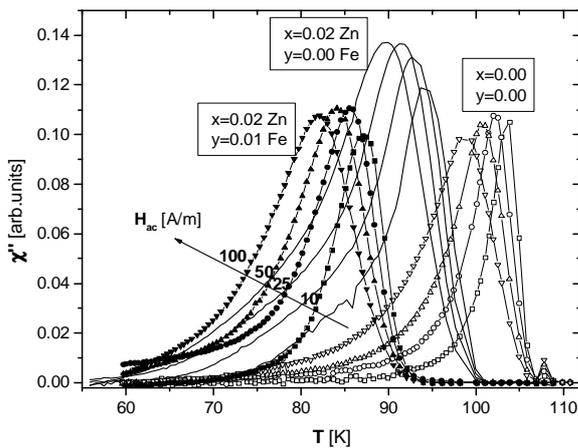


Fig. 1. Temperature dependence of the imaginary part χ'' of AC susceptibility for (a) $x=0.00$, $y=0.00$, (b) $x=0.02$ Zn, $y=0.00$ and (c) $x=0.02$ Zn, $y=0.01$ Fe samples at different AC field amplitudes H_{ac} ranging from 10 A/m to 100 A/m.

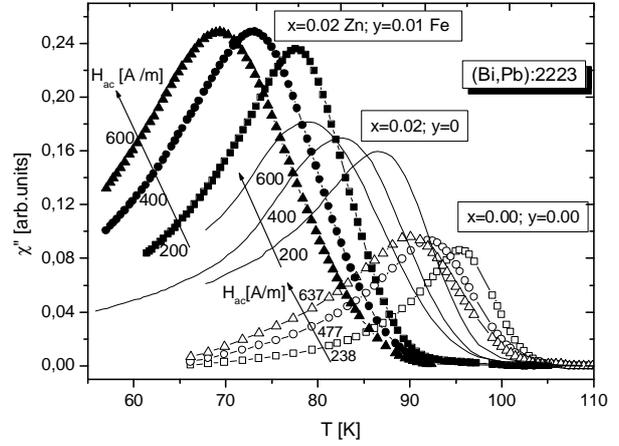


Fig. 2. Temperature dependence of the imaginary part χ'' of AC susceptibility for: (a) $x=0.00$, $y=0.00$; (b) $x=0.02$ Zn, $y=0.00$ and (c) $x=0.02$ Zn, $y=0.01$ Fe samples at different AC field amplitudes H_{ac} ranging from 200 A/m to 638 A/m.

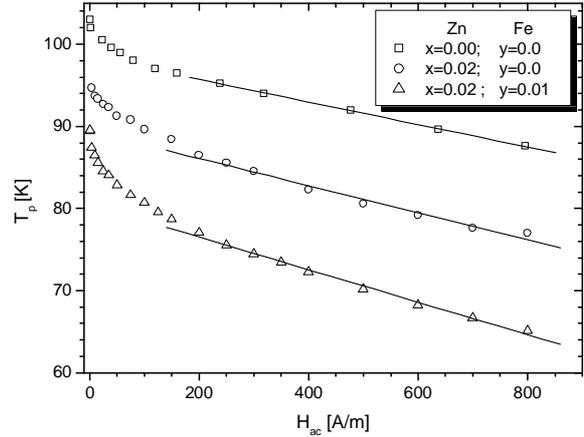


Fig. 3. Intergranular χ'' -peak temperature T_p versus AC field amplitude for $x=0.00$, $y=0.00$ (squares), $x=0.02$ Zn, $y=0.00$ (circles) and $x=0.02$ Zn, $y=0.01$ Fe (triangles).

In order to investigate the effect for partial substitution of Cu with Zn and Fe on the intergranular pinning force, we studied the T_p dependence as a function of H_{ac} .

As can be seen from Fig. 3, we got a linear dependence of T_p as a function of H_{ac} , in the a.c.amplitude range $200 \text{ A/m} < H_{ac} < 800 \text{ A/m}$, and a nonlinear dependence in the range $10 \text{ A/m} < H_{ac} < 100 \text{ A/m}$. The crossover field H_1^* between two regions is around 200 A/m for all samples.

The linear fits are:

$$T_p = 98.46 - 1.37 \times 10^{-2} H_{ac} \text{ [K]}, \text{ for } x=0.0, y=0.0 \quad (1)$$

$$T_p = 89.38 - 1.65 \times 10^{-2} H_{ac} \text{ [K]}, \text{ for } x=0.02 \text{ Zn}, y=0.00 \quad (2)$$

$$T_p = 80.47 - 1.98 \times 10^{-2} H_{ac} \text{ [K]}, \text{ for } x=0.02 \text{ Zn}, y=0.01 \text{ Fe} \quad (3)$$

4. Discussion

The (IL) line for granular HTS compounds is obtained from AC susceptibility measurements as a relation between the temperature T_p of the maximum in χ'' (T) and the amplitude H_{ac} of AC magnetic field [18-20]. As generally accepted, the irreversibility line can be described by the following power law [21-22]:

$$1-T_p/T_c = aH^q, \quad (4)$$

where T_c is the critical transition temperature and H the amplitude of the applied magnetic field. T_c is the temperature for the onset of diamagnetic response in the $H_{ac}=0.4$ A/m magnetic field.

Fig. 4 show the log-log plot of the reduced temperature $t=T_p/T_c$ as a function of H_{ac} in low magnetic field (up to 100 A/m). There exists two distinct regions for the nonlinear IL corresponding to two different power laws. The drawn lines is the power law given in eq.1 with slopes q_1 and q_2 and H_2^* is the crossover field. The slope q_1 decrease from 0.2 (sample $x=y=0.00$) to 0.05 (samples by $x=0.02$ Zn and $x=0.02$ Zn $y=0.01$ Fe). The slope q_2 decrease from 0.33 to 0.2 by increasing x and y .

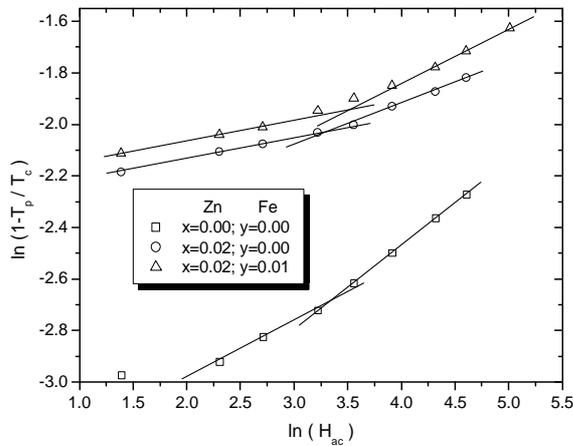


Fig. 4. The $\ln(1-T_p/T_c)$ as a function of $\ln(H_{ac})$ in the nonlinear region of irreversibility curves (the low field range, below 200 A/m).

After calculating the AC susceptibility response due to the intragranular and intergranular flux densities, Muller obtained the following equation for $T_p = T(H)$ [21]:

$$\left(1 - \frac{H_{ac}}{H_j}\right)^2 = 1 + \frac{2d \cdot f_j(T)}{\mu_0 \mu_{eff}(T) \cdot H_j^2} \quad (5)$$

where H_j is characteristic field of the Kim-Anderson critical state model, d the sample thickness, f_j is the pinning force density and μ_{eff} the effective permeability of grain medium.

An explanation of q values obtained in our samples for small H_{ac} is to take into account the following temperature dependence of the pinning force density:

$$f_j(T) = f_j(0) \left(1 - \frac{T}{T_c}\right)^{n/2} \quad (6)$$

For small fields, near T_c , $\mu_{eff}=1$, and we find from [eq. 5] the IL line described by eq. (4), with $q=2/n$ and

$$a_1 = \left[\frac{\mu_0 H_j}{f_j(0) \cdot d} \right]^{2/n}, \text{ where } H_j = \frac{\Phi_0}{4\mu_0 \lambda_g(0) R_g}.$$

For undoped sample in the nonlinear region the exponent decreases from $n_1=10$ to $n_2=6$, and suggest the decrease of intergranular pinning force density in the range of low magnetic fields. For doped samples, the exponent $n_1=40$ and n_2 around 10. These values suggest that for low magnetic fields 3d ions acts in the intergrain regions as strong pinning centers.

With increasing H_{ac} above 200A/m, the temperature T_p of intergranular peak position decreases. In this case we obtain a linear behaviour of the IL:

$$1 - T_p/T_c = a_2 H, \quad (7)$$

Eq. (7) was obtained in reference [23] by assuming that $f_j(T)/\mu_{eff}(T) = [f_j(0)/\mu_{eff}(0)](1-(T/T_c)^2)$ and $H_{ac} \gg H_j$,

$$\text{where } a_2 = \left[\frac{\mu_0 \cdot \mu_{eff}(0)}{2f_j(0) \cdot d} \right]^{1/2}.$$

The increase of slope a_2 (see Fig. 3) by increasing x and y , agree with the decrease of $f_j(0)$ of intergranular pinning by partial substitution of Cu by 3d element.

5. Conclusions

The Cu substitution simultaneously by Zn ($x=0.00, 0.02; 0.05$) and Fe ($y=0.00; 0.01$) in $(Bi_{1.8}Pb_{0.46})Sr_{1.88}Ca_{2.06}(Cu_{1-x-y}Zn_xFe_y)_3O_z$ bulk superconductor was performed.

Critical transition temperature T_c decreases by increasing the concentration x and y for the Zn and Fe ions.

X-ray diffraction analysis of the (Bi,Pb)(Sr,Ba):2223 samples shows that they contain Bi-2223 phases as majority phase.

A.c. susceptibility measurements as a function of temperature and amplitude of a.c. field is a powerful technique to study the influence of Zn and Fe ions on the irreversibility line of the (Bi,Pb):2223 superconductor.

The irreversibility line $T_p(H)$ for the intergranular peak is found to obey the $(1-T_p/T_c) = aH^q$ law, with a crossover from nonlinear (at low fields) to linear behaviour (high fields).

Partial atomic substitution of Cu by 3d elements induced modification of the Josephson medium. For low magnetic fields, 3d element lead to the improvement of

pinning force density and above 200 A/m to the decrease of pinning force density.

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