

# AC susceptibility study of the intergranular flux motion in the Fe doped $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$ high temperature superconductor

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By using solid state reaction method, Fe doped  $(\text{Bi,Pb})_2\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  high temperature superconductor samples were synthesized. The phase purity and the morphology of samples were obtained from XRD and SEM measurements. The temperature dependencies of real and imaginary parts of AC susceptibility were performed by using different values of AC field amplitude. Temperature dependencies of the irreversibility line for intergranular flux motion were obtained.

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*Keywords:*

## 1. Introduction

High-temperature superconducting (HTS) materials are in general not a homogeneous continuum but rather a network of linked grains [1]. In HTS the weak links occur easily in preparation and growth, being weakened further by irradiation, by impurities and by disorder. Cu-O plane weak links are the major obstacle for HTS currents, both dc and radio frequency. Exponential degradation of the supercurrent transport is observed at a function of misalignment angle between  $\text{CuO}_2$  planes. One of the reasons for this behavior is the d-symmetry of SC order parameter [2], and an extra degradation is believed to arise from structural defects such as dislocations [3] and derivations from stoichiometry. In particular, the loss of oxygen at the grain surfaces [4] leads to a decrease of doping with respect to the grain bulk value and thus a local degradation of SC properties. Manufacturing of technically applicable HTS materials require well-defined preparation techniques to obtain clean grains with small misorientation of their crystal axes.

The critical current density,  $J_c$  (and other superconducting parameters) of  $(\text{Bi,Pb}):2223$  high-temperature superconductors (HTS) is influenced by the Bi:2212 ratio. The inclusions of 2212 in the 2223 matrix may act as weak links or as pinning sites and this depends on the 2212 ratio, its morphology, etc. [5]. However, an obvious correlation between  $J_c$  and the ratio of 2212 was not yet established. The manner in which the 2223 matrix includes the 2212 phase is not completely understood.

The increase of  $J_c$  was possible by addition of Ag or iodine and atomic substitutions:

Because (HTS) consists of numerous grains, and this Josephson medium has strongly nonlinear magnetic sensitivity, the bulk material is used in the magnetosensitive sensors. The possibility to fabricate a magnetomodulation sensor of a weak magnetic field based on the Bi:2223 HTS ceramics with the operational temperature  $T = 77\text{ K}$  was investigated [6].

The ac susceptibility measurements on bulk samples reside in the fact that it makes possible the separation of the inter- and intra-granular signals, as well as the separation of the response coming from different superconducting phases (by example 2212 and 2223). In the  $(\text{Bi,Pb}):2223$  superconducting ceramic samples the competition of intra- and intergrain conductivity is strongly temperature dependent and is differently affected by the nanodefects induced by the 3d ions which substitute in the Cu positions [7-10].

In this paper we report the effect of the partial substitution for Cu by  $x=0.02$  Fe on the inter- and intragrain  $\chi''$  peak of  $(\text{Bi,Pb}):2223$  superconductor by using AC susceptibility measurements as a function of temperature and AC field amplitude.

## 2. Experimental

The  $(\text{Bi}_{1.6}\text{Pb}_{0.4})(\text{Sr}_{1.8}\text{Ba}_{0.2})\text{Ca}_2(\text{Cu}_{1-x}\text{Fe}_x)_3\text{O}_y$  samples where  $x = 0.00$  and  $0.02$  were synthesized by solid state reaction of appropriate amounts of the metal oxides and carbonates of 99,99% purity. The characterization of the phase was carried out by X-ray diffraction (XRD) with Bruker equipment by using  $\text{Cu K}\alpha$  radiation. The XRD analysis confirmed the presence of a single "2223" phase in  $x = 0.00$  sample. In  $x = 0.02$  Fe sample most of the peaks belong to the "2223" phase with a few of low intensities belonging to the "2212" phase. The volume fraction of the "2223" phase obtained by using the XRD peak intensity of  $(0010)$  in "2223" and the  $(008)$  peak intensity in "2212" was around 96% vol.

SEM measurements were performed by using a JEOL JSM 6100 scanning electron microscope. The morphology observed is fairly uniform, consisting of platelets larger than  $10\mu\text{m}$ . No agglomerates of the raw constituents were found.

The real ( $\chi'$ ) and imaginary ( $\chi''$ ) 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 and discussion

For  $x=0.00$  and  $x=0.02$  samples the real part  $\chi'(T)$  show a two step behavior, characterizing the flux penetration in the intergranular matrix and in the grains respectively. The inflection point in the lower drop in the  $\chi(T)$  curve may be assigned to the intergrain critical temperature while the end of the upper step (the end of the superconductor diamagnetism) correspond to the intragrain critical temperature  $T_{cG}$ .

Fig. 1 shows  $\chi'(T)$  behavior of real susceptibility for  $x=0.02$  Fe sample.

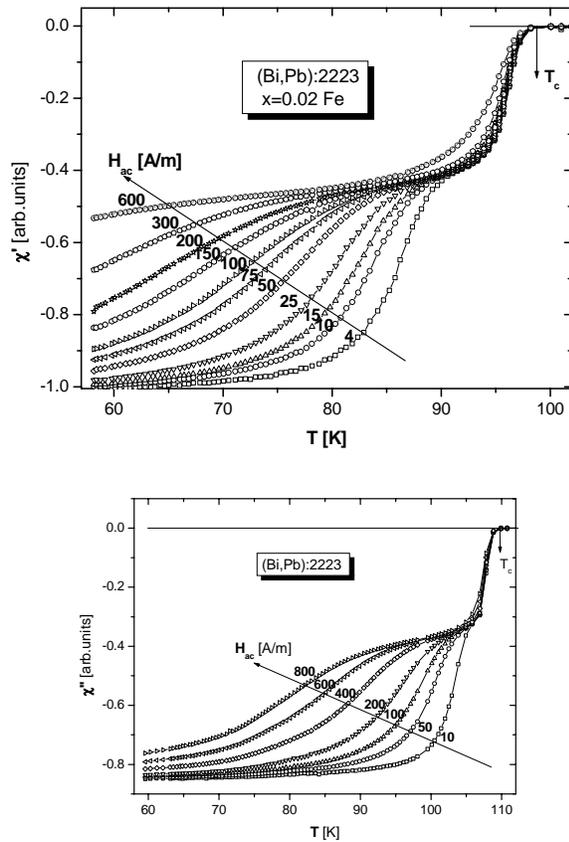


Fig. 1. Real part  $\chi'(T)$  at different  $H_{ac}$  amplitudes for  $x=0.00$  and  $x=0.02$  Fe in (Bi,Pb):2223 sample.

The  $T_{cG}$  values are 109,5K in  $x=0.00$  and 99K in  $x=0.02$  Fe. The decrease of  $T_{cG}$  in our  $x=0.02$  bulk samples agree with that found in Fe and Ni doped Bi:2212 single crystal [13].

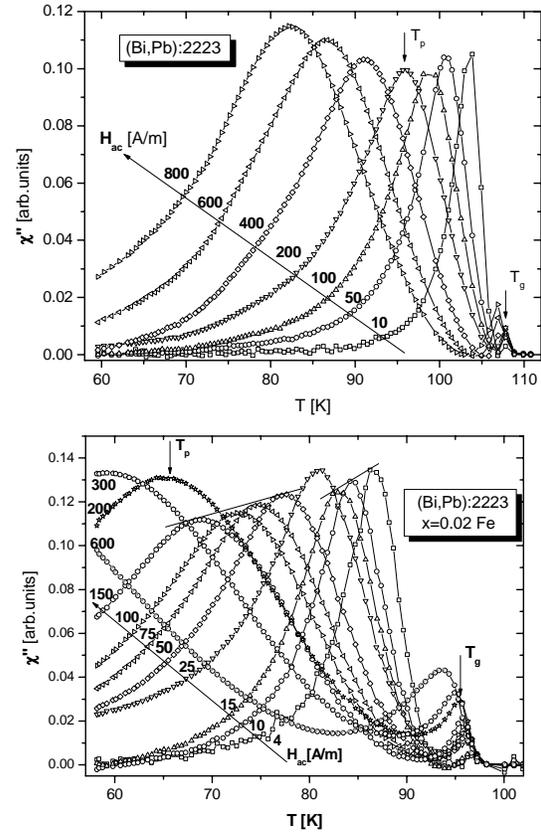


Fig. 2. Imaginary part  $\chi''(T)$  at different  $H_{ac}$  amplitudes for  $x=0.00$  and  $x=0.02$  Fe in (Bi,Pb):2223 sample.

The  $T_{cG}$  values are 109,5K for undoped sample ( $x=0.00$ ) and  $T_{cG}=98.5$  K for  $x=0.02$  Fe doped sample, respectively.

An important feature of HTS is the existence of „irreversibility line” (IL) where magnetic irreversibility sets in. The (IL) line is obtained from AC susceptibility measurements as a relation between the temperature  $T_p$  of the maximum in  $\chi''(T)$  and the amplitude  $H_{ac}$  of AC magnetic field. [12].

For  $x=0.00$  and  $x=0.02$  Fe samples, imaginary part of AC susceptibility,  $\chi''(T)$ , exhibit two peaks at  $T_p$  and  $T_g$ , which indicates inter- and intragranular dissipation.

For undoped sample, a small intergranular peak appear near  $T_g$  because the motion of intragrain Abrikosov vortices, and a large peak of  $T_p$  assigned to the intergranular Josephson vortices moving along the grain boundaries, respectively.

The effect of partial substitution of Cu by  $x=0.02$  Fe, lead to the increase of intensities of the intra- and intergranular dissipation peaks (Fig. 2), are shifted to temperatures lower than in  $x=0.00$  sample.

Fig. 3 shows  $T_p$  dependence function of  $H_{ac}$  for our samples. Above  $H_{ac}=200$  A/m, for all samples, the relation  $T_p(H_{ac})$  seems to be linear, while a departure from this linear behavior of IL line is observed to the crossover field  $H^*=200$  A/m for  $x=0.00$  and around  $H^*=100$  A/m for  $x=0.02$  Fe, respectively.

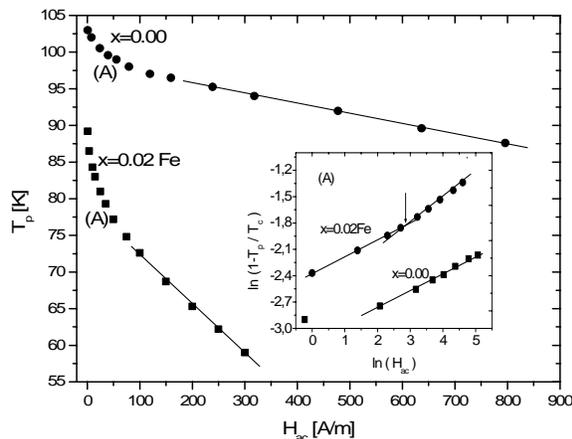


Fig. 3. Intergranular  $\chi''$ -peak temperature  $T_p$  versus AC field amplitude for  $x=0.00$ ,  $y=0.00$  (squares),  $x=0.02$  Zn,  $y=0.00$  (circles) and  $x=0.02$  Zn  $y=0.02$  Fe (triangles).

As generally accepted, the irreversibility line can be described by the following power law [13]:

$$1-T_p/T_c = a H^q, \quad [1]$$

where  $T_c$  is the critical transition temperature and  $H$  the amplitude of the applied magnetic field.

The IL line  $T_p(H_{ac})$  for our samples can be divided into two regions characterized by a different behavior: a linear relation ( $q=1$ ), and a nonlinear  $T_p(H_{ac})$  below the crossover field  $H^*$ .

The increase of slope  $a$  for  $q=1$  for  $x=0.02$  Fe (see Fig. 3), agree with the decrease of intergranular pinning force  $f_i$  by partial substitution of Cu by Fe.

The insert of Fig. 3 shows the log-log plot of the reduced temperature  $t=T_p/T_c$  as a function of  $\ln H_{ac}$  in low magnetic field. There exists two distinct regions for the nonlinear IL, with slopes  $q=0.19; 0.28$  for  $x=0.02$  Fe and a single region with  $q=0.19$  for  $x=0.00$  sample.

#### 4. Conclusions

The partial substitution of Cu by  $x=0.02$  Fe in (Bi,Pb)(Sr,Ba):2223 system leads to the decrease of  $T_c$  and shows that that -2223 phases is the majority phase.

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

Partial atomic substitution of Cu by Fe elements induced the decrease of inter- and intragranular pinning force density.

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