

# Phase transition of (Bi, Pb)-2223 superconductor induced by Fe<sub>3</sub>O<sub>4</sub> addition

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## Abstract

We investigated the effect of Fe<sub>3</sub>O<sub>4</sub> addition on the critical temperature of (Bi, Pb)-2223 polycrystalline samples. Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> + *x* wt. % Fe<sub>3</sub>O<sub>4</sub> (*x* = 0.0, 0.2, 0.4, 0.6, and 0.8) samples were prepared by using a solid-state reaction method. The analysis of X-ray diffraction data indicates that as Fe<sub>3</sub>O<sub>4</sub> is added, dominant phase of the sample changes from Bi-2223 to Bi-2212 with an increasing Bi-2201 phase. The transition temperature of the samples drastically decreased with the Fe<sub>3</sub>O<sub>4</sub> addition. The resistance data of samples with *x* = 0.2 and 0.4 showed a double transition indicating a coexistence of Bi-2223 and Bi-2212 phase while the samples with *x* = 0.6 and 0.8 showed a single transition with a semiconducting behavior. This phase transition may originate from changes in local structure of the Bi-2223 system by Fe<sub>3</sub>O<sub>4</sub> addition. Analysis of the pair distribution function of the Cu-O pair in the CuO<sub>2</sub> plane calculated from extended X-ray absorption fine structure data revealed that the oxygen coordination of copper ion changes from CuO<sub>4</sub> planar type (*x* = 0.0 - 0.4) to CuO<sub>5</sub> pyramidal type (*x* = 0.6, 0.8). The correlated Debye-Waller factor, providing information on the atomic disorder within the CuO<sub>2</sub> plane, shows an inverse relation to the coordination number. These results indicate that addition of Fe<sub>3</sub>O<sub>4</sub> changes the oxygen distribution around Cu in the CuO<sub>2</sub> plane, causing a phase transition from Bi-2223 to more stable Bi-2212/Bi-2201 phases.

**Keywords:** (Bi,Pb)-2223, Fe<sub>3</sub>O<sub>4</sub>, critical temperature, EXAFS, coordination number, FWHM

## 1. INTRODUCTION

After the discovery of the Bi-Sr-Ca-Cu-O (BSCCO) high *T<sub>c</sub>* superconductor, studies on the application of this system has attracted a huge attention. BSCCO superconductor is classified into three phases according to the number of Cu-O<sub>2</sub> planes *n*, and the critical temperature (*T<sub>c</sub>*) varies depending on the phases such as Bi-2201 (*n* = 1, *T<sub>c</sub>* ~ 20 K), Bi-2212 (*n* = 2, *T<sub>c</sub>* ~ 85 K), and Bi-2223 (*n* = 3, *T<sub>c</sub>* ~ 110 K) [1, 2]. Fabrication of an isolated phase of Bi-2223 is found to be difficult due to the fact that the Bi-2212 phase grows prior to the Bi-2223 phase during synthesis [3], therefore, Bi-2212 phase necessarily occupies a certain volume fraction in the fabrication of Bi-2223 phase. For the application of the BSCCO system, not only the critical current density but also *T<sub>c</sub>* must be considered. The Bi-2212 phase has a higher critical current density than the Bi-2223 phase due to fewer weak links, but has a much lower *T<sub>c</sub>*, which is close to the liquid nitrogen temperature [4]. Since low critical current density and low *T<sub>c</sub>* have significant limitations in application of superconductors, various methods have been tried to improve the both characteristics.

Partial substitutions or additions of certain elements at different cationic sites have been conducted to enhance the

Superconducting properties of the Bi-2223 system. In particular, it is known that substitution of Pb in the Bi sites or addition of Pb in the BSCCO composition can effectively increase the volume fraction of the Bi-2223 phase [5]. As another approach, in order to improve the critical current density of (Bi, Pb)-2223, additions of impurity materials have been tried to introduce artificial pinning centers [6]. Especially, ferromagnetic Fe<sub>3</sub>O<sub>4</sub> doping in (Bi, Pb)-2223 system has been tried to improve the critical current density, however, the values of *T<sub>c</sub>* are reported to decrease abruptly as the Fe<sub>3</sub>O<sub>4</sub> content increases [7].

In this research, we investigate the mechanism of *T<sub>c</sub>* decrease in Fe<sub>3</sub>O<sub>4</sub>-added Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> ((Bi, Pb)-2223) polycrystalline samples by using extended X-ray absorption fine structure (EXAFS) spectroscopy. Variation in *T<sub>c</sub>* of cuprates is reported to relate to the change in the local structure of the CuO<sub>2</sub> plane. EXAFS is known to be a powerful tool to identify the distribution of atomic displacements. Especially, EXAFS data measured at the Cu K-edge provide useful information on both the local structure of the CuO<sub>2</sub> plane and the structural phase variation in Fe<sub>3</sub>O<sub>4</sub>-added (Bi, Pb)-2223 system. In addition, the coordination number and distribution of oxygen atoms around copper are determined by using of the Cu-O pair distribution function (PDF) [8].

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## 2. EXPERIMENT

Samples with starting composition of Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> (Bi, Pb)-2223 were prepared using the solid-state reaction method. The Pb content was chosen to be 0.4 as this is the optimized value for the highest  $T_c$ . Bi<sub>2</sub>O<sub>3</sub>, PbO, SrCO<sub>3</sub>, CaCO<sub>3</sub> and CuO powders of high purity (99.99%) were mixed in appropriate amounts and ground before being calcined in four steps of 670 °C / 48 hours + 750 °C / 48 hours + 800 °C / 48 hours + 820 °C / 48 hours. After adding 0.0, 0.2, 0.4, 0.6, and 0.8 wt. % of Fe<sub>3</sub>O<sub>4</sub> as magnetic material, several times of milling and intermittent grindings were performed to ensure homogeneity of the sample. The powders were then pressed and sintered in air at 850 °C for 160 hours [9]. The Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+δ</sub> +  $x$  wt. % Fe<sub>3</sub>O<sub>4</sub> superconductors fabricated for this study were named as Pure, F02, F04, F06, and F08 according to the Fe<sub>3</sub>O<sub>4</sub> content.

The powder X-ray diffraction (XRD) (D8 Discover with GADDS) and energy dispersive X-ray spectroscopy (EDX) (FlatQuard) were used to determine the resultant phase and the compositional distribution, respectively. To determine the critical temperature of samples, the resistance measurements as a function of temperature were carried out by using a four-probe technique in a closed loop helium system [10]. In order to observe the local structure around Cu atoms, the Cu-K $\alpha$  (8980.0 eV) X-ray absorption spectra were collected in the Pohang Accelerator Laboratory (PAL) 8C nanoprobe beam line at room temperature.

The collected Cu K-edge XAFS spectra were normalized to the atomic energy absorption by using the EXAFS method [11], and collected EXAFS data were analyzed by the ATHENA code of the IFEFFIT software program [12]. For an analysis of the oxygen distribution in the Cu-O<sub>2</sub> layer, the atomic PDF is calculated on the 1<sup>st</sup> Cu-O shell by using a regularization method [13].

## 3. RESULTS AND DISCUSSION

Figure 1 shows the powder X-ray diffraction (XRD) patterns of the samples Pure, F02, F04, F06, and F08. The peaks belong to 2223, 2212, and 2201 phases are marked as H, L, and\*, respectively, and impurities such as Ca<sub>2</sub>PbO<sub>4</sub> and Fe<sub>2</sub>O<sub>3</sub> are separately indicated. Ca<sub>2</sub>PbO<sub>4</sub> impurity is found only in the pure sample, and it disappears in all the samples in which Fe<sub>3</sub>O<sub>4</sub> is added. In addition, as Fe<sub>3</sub>O<sub>4</sub> is added to (Bi, Pb)-2223, a number of peaks of 2223 phase disappear, and a new peak of Fe<sub>2</sub>O<sub>3</sub> starts to appear in samples of F06 and F08. The volume fraction of Fe<sub>2</sub>O<sub>3</sub> in F06 is 0.7 % respect to the total volume, and the relative volume fraction of Fe<sub>2</sub>O<sub>3</sub> in F08 increases to 1.0 %.

As can be seen in Fig. 1, the addition of Fe<sub>3</sub>O<sub>4</sub> seems to promote a phase transition from 2223 to 2212 and 2201 phases and to induce a peak shift. All three phases are found to coexist in the samples with Fe<sub>3</sub>O<sub>4</sub> except Pure sample. The volume fractions of the superconducting phase are calculated from the diffraction intensities of 2201, 2212, and 2223 by using the equation (1) and listed in Table I. The 2223 volume fraction for the pure sample is

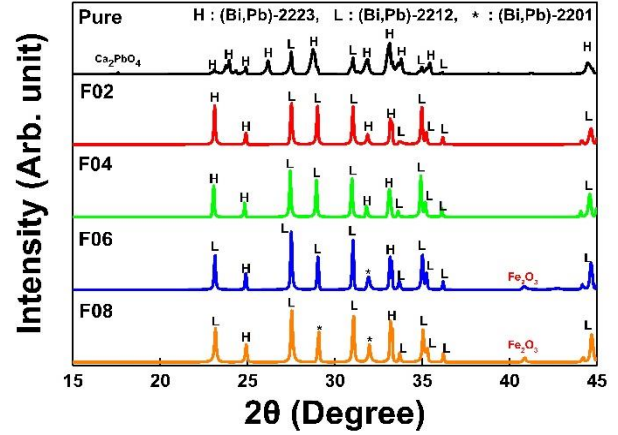


Fig. 1. X-ray diffraction patterns of (Bi, Pb)-2223 samples with Fe<sub>3</sub>O<sub>4</sub> addition. 2201, 2212, 2223 phases are indicated as \*, L, and H, respectively.

TABLE I  
SUPERCONDUCTING VOLUME FRACTION OF (Bi, Pb)-2223 WITH Fe<sub>3</sub>O<sub>4</sub> ADDITION.

Sample	Volume Fraction (%)		
	2201	2212	2223
Pure	0.0	32.8	67.2
F02	4.3	68.0	27.6
F04	5.0	70.0	27.6
F06	11.5	74.5	14.0
F08	19.4	64.1	16.4

67.2% and the other phase is 2212. As Fe<sub>3</sub>O<sub>4</sub> is added, the 2223 volume fraction drastically decreases and 2212 becomes a major phase with increasing 2201 phase.

$$V_{2201} = \frac{\sum I_{2201}(\text{peak})}{\sum I_{2223}(\text{peak}) + \sum I_{2212}(\text{peak}) + \sum I_{2201}(\text{peak})} \times 100$$

$$V_{2212} = \frac{\sum I_{2212}(\text{peak})}{\sum I_{2223}(\text{peak}) + \sum I_{2212}(\text{peak}) + \sum I_{2201}(\text{peak})} \times 100 \quad (1)$$

$$V_{2223} = \frac{\sum I_{2223}(\text{peak})}{\sum I_{2223}(\text{peak}) + \sum I_{2212}(\text{peak}) + \sum I_{2201}(\text{peak})} \times 100$$

Figure 2 shows electrical resistance as a function of temperature of all of the samples. Pure, F02, and F04 exhibit a metallic behavior in the normal state, whereas F06 and F08 exhibit a semiconducting behavior in the normal state. Similar results of semiconducting resistance due to the Fe substitution for Cu in Bi-2212 system had also reported [14]. It could infer the possibility of partial substitution of Fe for Cu. The value of  $T_{c,\text{onset}}$  of pure is 112.3 K, showing the highest critical temperature. A close inspection on the resistances of F02 and F04 reveals double transitions, a weak transition around 106 K, but a dominant transition at 62.7 K and 61.7 K, respectively, which may indicate a coexistence of 2223 and 2212 phases. As the Fe<sub>3</sub>O<sub>4</sub> content further increases, the transition becomes a single and the values of  $T_{c,\text{onset}}$  reduce to near 46 K for F06 and F08. In addition, a systematic increase in the transition width  $\Delta T_c$  as well as a systematic decrease in the residual resistance ratio (RRR) with increasing the Fe<sub>3</sub>O<sub>4</sub> content indicate an increase of impurity phase.

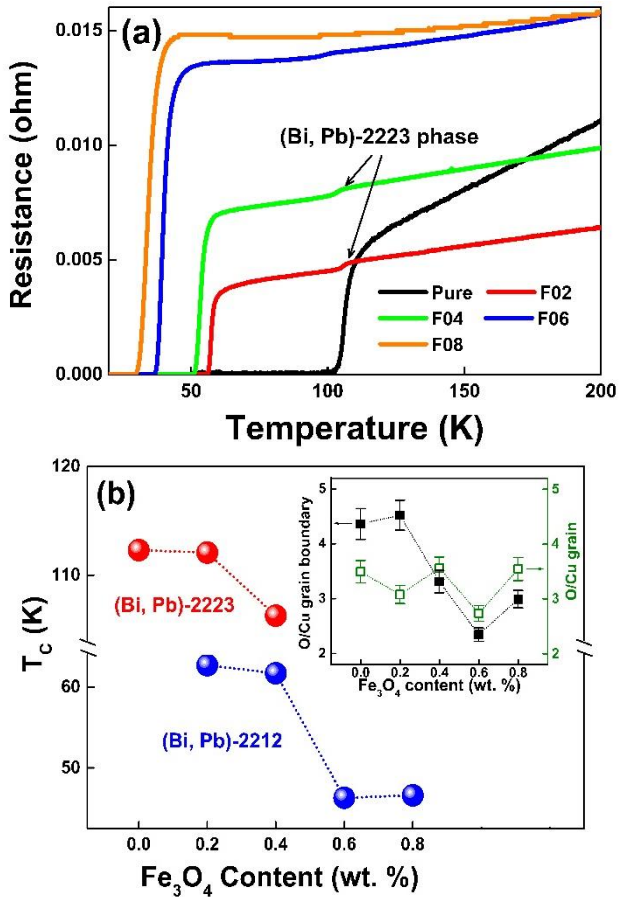


Fig. 2. (a) Resistance of (Bi, Pb)-2223 samples as a function of temperature with Fe<sub>3</sub>O<sub>4</sub> addition (b) Critical temperature as a function of Fe<sub>3</sub>O<sub>4</sub> content. Phase transition from 2223 to 2212 and 2201 is obvious as the Fe<sub>3</sub>O<sub>4</sub> content increases. Inset: atomic % of oxygen relative to copper at grain boundaries and inside grains of (Bi, Pb)-2223.

TABLE II  
THE CRITICAL TEMPERATURE OF (Bi, Pb)-2223 WITH Fe<sub>3</sub>O<sub>4</sub> ADDITION.

Sample	Critical Temperature T <sub>c</sub> (K)			
	T <sub>c onset</sub>	T <sub>c zero</sub>	ΔT <sub>c</sub>	RRR
Pure	112.3	102.4	9.9	2.00
F02	62.7	55.8	6.9	1.55
F04	61.7	50.8	10.9	1.38
F06	46.3	36.0	10.3	1.27
F08	46.6	29.6	17.0	1.18

In order to check variation in the elemental composition of (Bi, Pb)-2223, EDX analysis is performed. The inset of Fig. 2(b) shows the relative atomic ratio of oxygen to copper both inside grains and at the grain boundaries. The oxygen ratio inside grains is fluctuating around 3.2 irrespective of the Fe<sub>3</sub>O<sub>4</sub> content. However, the ratio of oxygen to copper at grain boundaries decreases abruptly with increasing Fe<sub>3</sub>O<sub>4</sub> content. For example, it starts from near 4.2 in Pure and approaches to about 3 for sample F08.

The abrupt decrease of the relative ratio of oxygen to copper by increasing Fe<sub>3</sub>O<sub>4</sub> content seems to be correlated

with the phase transition from Bi-2223 to Bi-2212.

Therefore, information on the distribution of oxygen in the Cu-O<sub>2</sub> plane may provide an explanation on the appearances of lower T<sub>c</sub> phases (Bi-2212 or Bi-2201) [15].

Using EXAFS measurements, it is possible to identify the oxygen distribution around Cu atoms in the Cu-O<sub>2</sub> plane, and an advanced PDF method is capable of providing an accurate information of isolated contribution of Cu-O pairs.

To examine the oxygen distribution in the Cu-O<sub>2</sub> plane, EXAFS data obtained at the Cu K-edge are analyzed. The collected EXAFS signal is weighted by  $k^3$  without the background spectrum. The transform window is set to  $k = (2.8 - 11.6) \text{ \AA}^{-1}$ , and the Hanning window is given as  $dk = 0.2 \text{ \AA}^{-1}$ . A typical Fourier transformed EXAFS spectra is shown in the inset of Fig. 3. Fourier transformed EXAFS oscillations represent that the 1<sup>st</sup> shell locates at a distance of  $\sim 1.5 \text{ \AA}$ , which corresponds to the contribution of the Cu-O scatterings. However, due to the possibility of multiple scattering effect from other shells, it cannot provide an accurate information in CuO<sub>2</sub> plane by fitting process. In order to evaluate an isolated contribution of Cu-O pairs in the Cu-O<sub>2</sub> plane, single contribution of Cu-O pairs was extracted from the EXAFS signals. The identical shapes of oscillations in Fig. 3 showed the well-extracted contribution of Cu-O pairs without any informational distortion from the other scatterings. Through this data, the atomic pair distribution function (PDF) of Cu-O pairs is calculated based on the EXAFS regularization method [16].

Typical PDFs of Cu-O pairs of (Bi, Pb)-2223 samples with Fe<sub>3</sub>O<sub>4</sub> addition are shown in Fig. 4. The line shapes of PDF for all of the samples are well separated into two groups; (i) Pure, F02, F04 and (ii) F06, F08. Especially, PDF for each group shows that integrated areas are different, where the area of the Gaussians corresponds to the oxygen coordination number adjacent to the photo absorbing Cu atoms [17]. An increase of the area of the distribution seems to have a correlation with Fe<sub>3</sub>O<sub>4</sub> content. Thus, to identify the oxygen coordination number, the Gaussian amplitude fitting to PDF is performed.

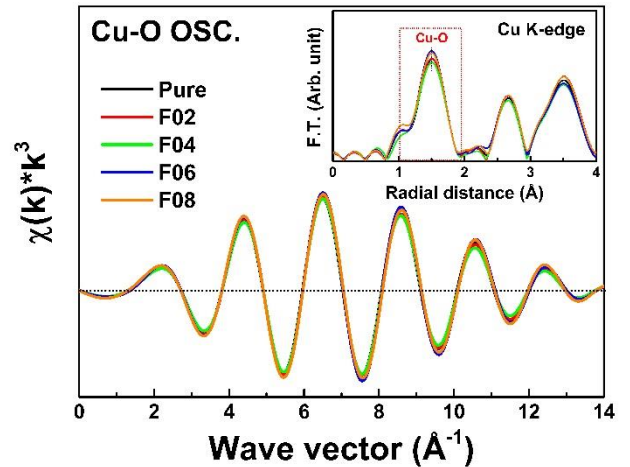


Fig. 3. Filtered Cu-O EXAFS signal in the range of  $R = (1.02 \text{ to } 1.96 \text{ \AA})$ . Inset: the Fourier transforms of Cu-K edge EXAFS spectra of all the samples.

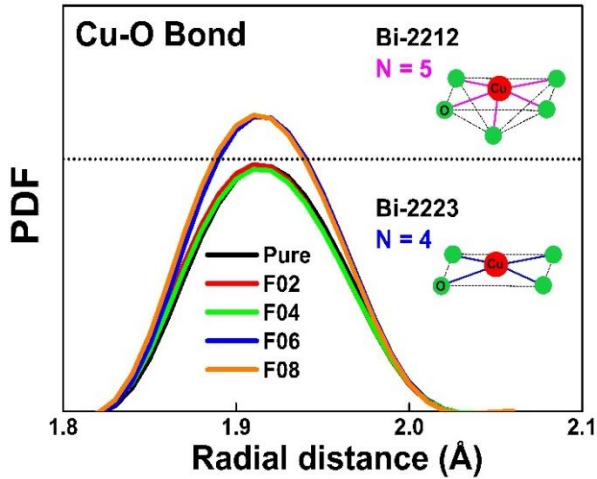


Fig. 4. The Cu-O pair distribution function of (Bi, Pb)-2223 with Fe<sub>3</sub>O<sub>4</sub> addition.

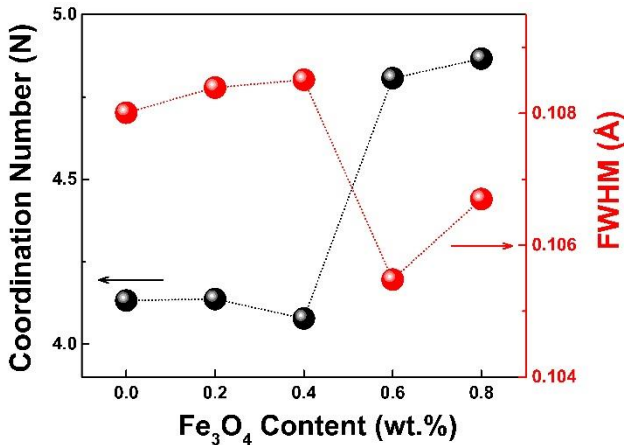


Fig. 5. The number of adjacent oxygen atoms around Cu and the full width at half maximum of (Bi, Pb)-2223 as a function of Fe<sub>3</sub>O<sub>4</sub> content.

Figure 5 shows the expected coordination numbers of oxygen for the average crystallographic structure, obtained from the PDF fitting, as a function of Fe<sub>3</sub>O<sub>4</sub> content. The abrupt increase of the coordination number from ~ 4.1 for Pure to ~ 4.9 for F06 and F08 can be clearly observed. Since the coordination numbers are associated with the distribution of oxygen in the CuO<sub>2</sub> plane, this result indicates that the oxygen coordination of copper ion changes from the CuO<sub>4</sub> planar type to the CuO<sub>5</sub> pyramidal type. It seems that partial vanish of CuO<sub>4</sub> square planes in the Bi-2223 phase induces average structural transformation to the CuO<sub>5</sub> pyramids of Bi-2212 and/or Bi-2201. In other words, vanish of stacked CuO<sub>4</sub> plane due to Fe<sub>3</sub>O<sub>4</sub> addition alters the superconducting phase from Bi-2223 to Bi-2212, which is in good agreement with the analysis of X-ray diffraction data.

The full width of half maximum (FWHM) of PDF curves, which corresponds to the correlated Debye-Waller factors providing information on the atomic disorder within the CuO<sub>2</sub> plane, are also calculated and displayed in Fig. 5. The values of FWHM are found to have an inverse relation with the coordination number, inferring that the system becomes more stable with Fe<sub>3</sub>O<sub>4</sub> addition. It is generally known that among this family, the Bi-2212 phase

has larger structural stability than the Bi-2223 phase [18]. As a result, it can be concluded that a unstable structure of the (Bi, Pb)-2223 system relaxes to another stable structure by the phase transition from Bi-2223 to Bi-2212 as the Fe<sub>3</sub>O<sub>4</sub> content increases beyond  $x = 0.6$ .

#### 4. CONCLUSION

In this study, the origin of  $T_c$  drop in the Fe<sub>3</sub>O<sub>4</sub>-added Bi<sub>1.6</sub>Pb<sub>0.4</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10+ $\delta$</sub>  ((Bi, Pb)-2223) system is investigated. (Bi, Pb)-2223+  $x$  wt. % Fe<sub>3</sub>O<sub>4</sub> ( $x = 0.0, 0.2, 0.4, 0.6,$  and  $0.8$ ) polycrystalline samples was fabricated by a solid-state reaction method. The analysis of X-ray diffraction data indicates that as Fe<sub>3</sub>O<sub>4</sub> is added, a dominant phase of the sample changes from Bi-2223 to Bi-2212 with an increasing volume fraction of the Bi-2201 phase. The values of  $T_c$  of the samples drastically decrease with the Fe<sub>3</sub>O<sub>4</sub> addition. The  $R$ - $T$  curves of the samples with  $x = 0.2$  and  $0.4$  show double transitions indicating a coexistence of Bi-2223 and Bi-2212/Bi-2201 phases. On the other hand, the samples with  $x = 0.6$  and  $0.8$  show a single transition with a semiconducting behavior.

This phase transition may be originated from changes in local structure of the CuO<sub>2</sub> plane in the (Bi, Pb)-2223 system by adding ferromagnetic Fe<sub>3</sub>O<sub>4</sub>. EXAFS measurements were carried out at the Cu K-edge to identify the effect of Fe<sub>3</sub>O<sub>4</sub> of addition on the local structure. Analysis of PDF of the Cu-O pair in the CuO<sub>2</sub> plane calculated from EXAFS reveals that the oxygen coordination of copper ion changes from the CuO<sub>4</sub> planar type ( $x = 0.0 - 0.4$ ) to the CuO<sub>5</sub> pyramidal type ( $x = 0.6, 0.8$ ). The atomic disorder within the CuO<sub>2</sub> plane is found to decrease as the Fe<sub>3</sub>O<sub>4</sub> content increases. These results indicate that addition of Fe<sub>3</sub>O<sub>4</sub> changes the oxygen distribution around Cu in the CuO<sub>2</sub> plane, causing a phase transition from Bi-2223 with high  $T_c$  to more stable Bi-2212/Bi-2201 phases with low  $T_c$ .

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