Physical, mechanical and magnetic properties of the Yb substituted $Bi_2Sr_2Ca_1Cu_2O_y$ textured superconductor

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Abstract

In this study, samples with nominal composition of $Bi_2Sr_2Ca_1Cu_{2-x}Yb_xO_y$, where x = 0.0, 0.05, 0.1 and 0.25 have been textured using the Laser Floating Zone technique. The effects of different Yb doping on the structure have been investigated by electrical resistivity, scanning electron microscopy, XRD, mechanical and dc-magnetization techniques. It has been found that with increasing Yb^{3+} doping for Cu, the physical and superconducting properties of structure gradually decrease. J_c values, calculated using the Bean's model, significantly change, although T_C is approximately the same for all samples.

Keywords: Bi-2212 · LFZ technique · XRD · Resistivity · Magnetic Properties · Critical Current

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1. Introduction

Since the discovery of high-temperature superconductor, many substitution studies have been made on HT_C-BSCCO system in Cu sites to improve the physical and magnetic properties of the superconducting phase [1-4]. The effect of substitution on the Cu sites is more important than on other ones since the transition temperatures in ceramic superconductors depends on the charge carrier concentration in the Cu-O planes. It is well known that holes in the Cu-O planes created by electron doping constitute an important parameter to achieve superconductivity. There are three well known phases in the Bi-based superconductors with general formula $Bi_2Sr_2Ca_{n-1}Cu_nO_y$ where n = 1, 2, and 3. The transition temperatures of these different phases are related to the number of the Cu-O layers per unit cell.

In addition, the material preparation techniques for the BSCCO systems play a crucial role, as important as the substitutions which can be made in BSCCO. Moreover, this system possesses a high crystallographic anisotropy which can be exploited by an adequate grain alignment to produce high quality tapes and wires, as well as bulk materials with improved electrical properties. Laser Floating Zone Melting (LFZ) technique is the one of best methods used to texture these bulk superconductor samples [5]. This method provides well aligned Bi-2212 grains, with their c-axis nearly perpendicular to the growth direction, and very small amount of secondary phases [6-7].

In this work, the effect of the Yb substituting for Cu on the mechanical, physical and superconducting properties of samples prepared by using the LFZ method has been investigated. The prepared samples have been characterized using X-ray powder diffraction (XRD), electrical resistivity, scanning electron microscopy (SEM) and magnetic measurements. In addition, it is important to know how Yb addition affects the mechanical properties of the LFZ grown Bi-2212 materials. Thus, Flexural strength tests have also been made to determine the mechanical behaviour of samples.

2. Experimental Details:

$Bi_2Sr_2CaCu_{2-x}Yb_xO_y$ samples, with x =0.0, 0.05, 0.1 and 0.25 Yb additions, have been prepared from commercial $Bi(CH_3COO)_3$ (≥ 99.99%, Aldrich), $Sr(CH_3COO)_2 \cdot 1/2H_2O$ (99%, Panreac), $Ca(CH_3COO)_2 \cdot H_2O$ (98%, Alfa Aesar), $Cu(CH_3COO)_2 \cdot H_2O$ (98%, Panreac), and $Yb(CH_3COO)_3 \cdot H_2O$ (99.9%, Alfa Aesar) powders. They were dissolved in a mixture of glacial acetic acid (PA, Panreac) and distilled water. To the resulting clear light blue solution, polyethyleneimine (PEI) (Aldrich, 50 wt% water) was added and the solution turned dark blue immediately, indicating the polymer-cations coordination through the nitrogen atoms. After partial evaporation (~80 vol. %) of water and acetic acid in a rotary evaporator, the concentrated solution was placed on a hot plate at 50 ºC until a very dark blue thermoplastic paste appeared. Further heating produced a slow combustion with the release of brown fumes (nitrogen oxides), producing a very fine powder. This product was then milled in an agate mortar, and calcined twice at 750 and 800 °C for 12 h with an intermediate manual milling. These prereacted powders were then used to prepare cylindrical precursors, approximately 120 mm long and 3 mm diameter by cold isostatic pressing with an applied pressure of 200 MPa during 1 minute. The green ceramic cylinders were subsequently used as feed in a LFZ melting installation described elsewhere [8]. Fibers were grown using a growth rate of 15 mm/h and a relative rotation of 18 rpm between the seed and feed. Using these growth conditions and adjusting the laser power input to obtain a molten zone of

1–1.5 times the rod diameter, it is possible to obtain a stable solidification front, which allows the fabrication of homogeneous textured bars.
Bi-2212 ceramic presents incongruent melting and, in consequence, after the directional solidification process, it is necessary to perform a thermal treatment in order to form the Bi-2212 superconducting phase [9,10]. This annealing process was performed under air, and consisted in two steps: 60 h at 860 ºC, followed by 12 h at 800 ºC and, finally, quenched in air to room temperature.
Phase determination has been performed on annealed samples using X-ray powder diffraction measurements (Rigaku D/max-B) with 2θ ranging between 10º and 60º. Microstructural characterization was made on polished longitudinal cross-sections of the samples, after the annealing process, in a scanning electron microscope (SEM, JEOL JSM 6400) equipped with an energy dispersive spectroscopy (EDX) system. Electrical measurements were performed by the conventional four-point probe configuration on about 30 mm long samples. The magnetic measurements of samples were performed with a 7304 model LakeShore VSM at temperatures of 10 and 25 K and an applied field of ± 1 T. Finally, mechanical characterization has been performed by flexural strength, using the three-point bending test in an Instron 5565 machine with a 10 mm loading span fixture and a punch displacement speed of 30 μm/min.

3. Results and Discussion

After texturing processes all the bars posses similar diameter, but after annealing it has been observed an important change on the 0.25Yb substituted samples. Their diameter increases from ~2.0 to 2.3 mm while the other samples maintain very similar diameters before and after annealing. This effect can be clearly observed in Fig. 1, where SEM images corresponding to longitudinal polished sections of samples after annealing, are displayed. In this figure it can be seen that samples diameter is similar in all cases, except for the 0.25Yb substituted sample, which is higher than for the rest of samples. Careful observation indicates that the amount of secondary phases (mostly observed as dark contrasts, corresponding to the $(Sr,Ca)CuO_2$ secondary phase) is increased when the amount of Yb is raised.
In order to explain the different behaviour of the 0.25Yb substituted samples, secondary electrons SEM micrographs have been recorded and a representative one is displayed in Fig. 2. In this figure it can be observed that these samples posses a high amount of porosity, which explains the increase of their diameter after annealing.

In order to identify the phases present in the samples, powder XRD has been performed on all textured samples. The obtained patterns are displayed in Fig. 3. They show that main peaks (indicated by +) in all samples correspond to the Bi-2212 phase, which clearly indicates that the performed thermal treatment is adequate to produce the superconducting phase as the major one. However, some minor peaks corresponding to the $CaCuO_2$ phase (indicated by a *) have also been detected.

Figure 4 shows the temperature dependence of the resistivity. In this figure it is clear that T_c is similar for all the Yb-doped samples, but slightly lower than the obtained for the undoped ones. On the other hand, for temperatures higher than the superconducting to normal state transition, all samples show a metallic behaviour.

M-H hysteresis loops have been measured at different temperatures: 10, 15, 20 and 25 K. In Figs. 5 and 6 the results at 10 and 25 K, respectively, are presented for clarity, as the samples evolution is the same in all cases. All those M-H hysteresis loops were obtained after cooling the sample in zero magnetic field (ZFC) and measured between the applied external fields of ± 10 kOe. The hysteresis curves of samples performed at different temperatures demonstrate that the pure sample exhibits the largest area which decreases when Yb content is increased.

The J_C values of the samples were calculated from these data using the Bean's model [11]:

$$J_C = 30 \frac{\Delta M}{d}$$

where J_C is the magnetization current density in ampéres per square centimeter. $\Delta M = M_+ - M_-$ is measured in electromagnetic units per cubic centimeter, d is the thickness of sample. Figure 7 shows the calculated critical current densities as a function of the applied field for all samples, at T=10 K. J_C calculations reveal that the pure sample exhibits better superconducting properties and has the biggest J_C values. These results indicate that J_C values significantly decrease when magnetic field is raised and the amount of Yb is increased.

In addition to the above measurements, the mechanical behaviour of samples has been studied by three-point bending tests. The mean mechanical stress, together with its standard error bars, is represented in Fig. 8 for the different samples. From this figure, it is clear that while Yb values increases the mechanical stress decreases due to the formation of secondary phases and the decrease on the grain orientation (see Fig. 1). Moreover, the mechanical stress decreases dramatically for 0.25Yb substituted samples due to the formation of a high amount of internal porosity (see Fig. 2).

4 Conclusions

In the present study, $Bi_2Sr_2CaCu_{2-x}Yb_xO_y$ (x = 0.0, 0.05, 0.1 and 0.25) samples were successfully textured using the LFZ method. Resistivity results pointed out that the T_c of samples slightly changes with increasing Yb concentration. From XRD patterns, it has been found that the Bi-2212 phase is the major one in all samples. Magnetic hysteresis and calculated J_c values show that the magnetic properties and critical current values of the samples decrease with increasing of the substitution level. Mechanical stress is found to be highly sensitive to the doping level as it decreases when Yb content increases.

Acknowledgements

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Figure captions

Figure 1. SEM micrographs of polished longitudinal sections of the Yb doped Bi-2212 textured materials after annealing: x = (**a**) 0.0; (**b**) 0.05; (**c**) 0.1; and (**d**) 0.25 Yb. Dark contrasts correspond to $(Sr,Ca)CuO_2$ secondary phases.

Figure 2. SEM micrographs of polished longitudinal sections of the 0.25Yb doped Bi-2212 textured materials after annealing, taken with secondary electrons in order to observe porosity in the samples.

Figure 3. X-ray diffraction patterns of the $Bi_2Sr_2CaCu_{2-x}Yb_xO_y$ superconducting samples. (+) Bi-2212 phase, and (*) $(Sr,Ca)CuO_2$ secondary phases.

Figure 4. Resistivity measurement vs. temperature for all samples.

Figure 5. M-H hysteresis loops of samples at T=10K and applied external fields of ±10kOe.

Figure 6. M-H hysteresis loops of samples at T=25K and applied external fields of ±10kOe.

Figure 7. Calculated J_C results of samples at T=10K from their hysteresis loops.

Figure 8. Mean maximum mechanical stress (three–point flexure strength), together with their standard error, as a function of Yb amount.

Figure 1

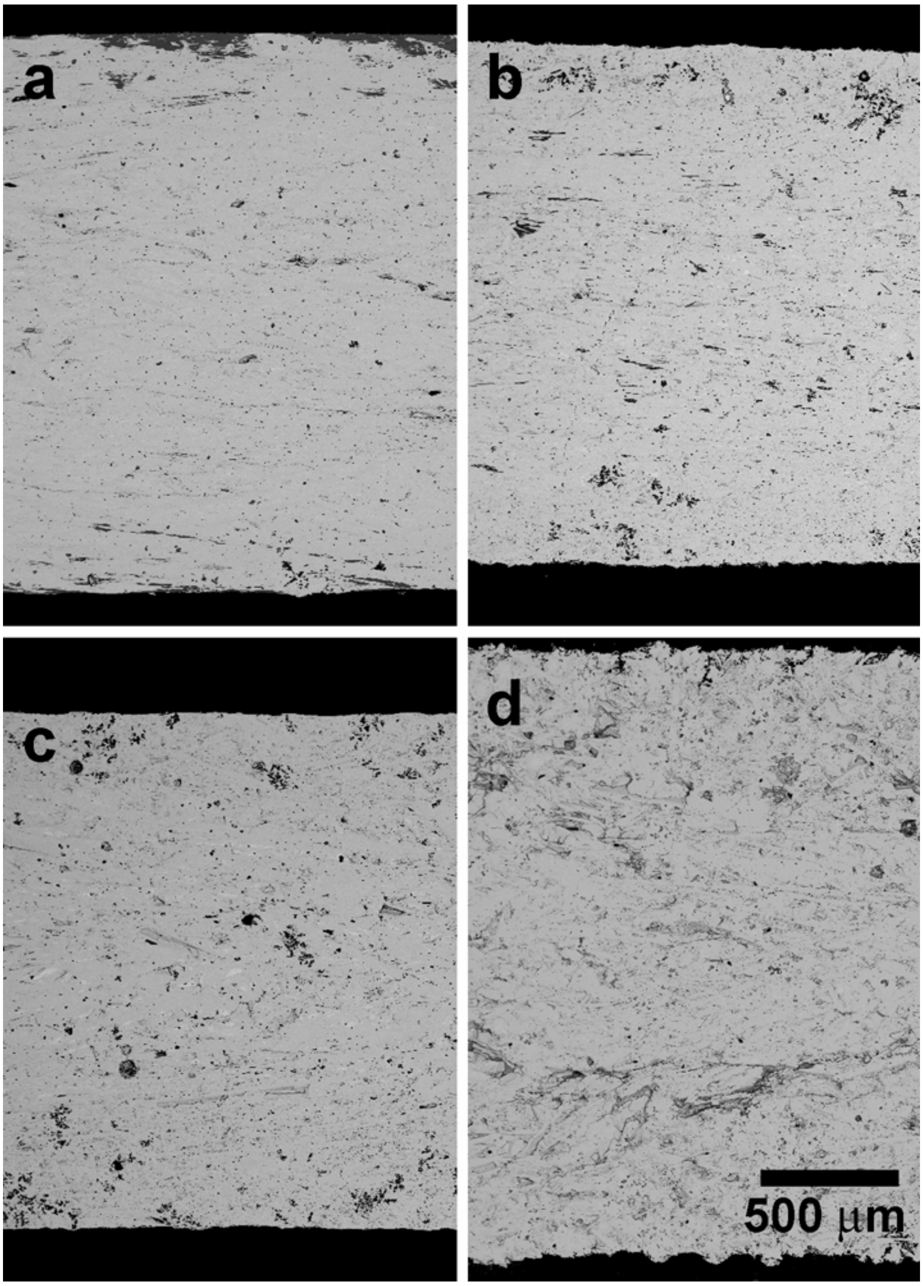


Figure 2

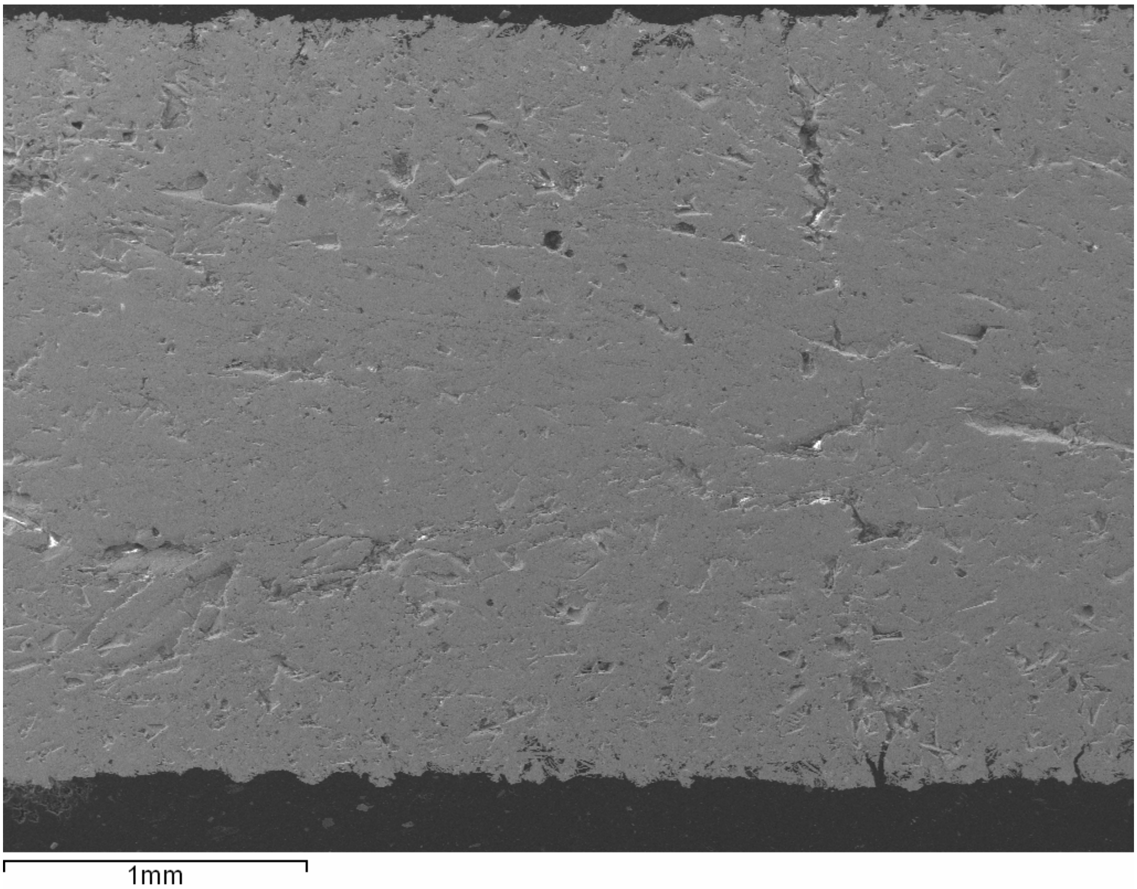
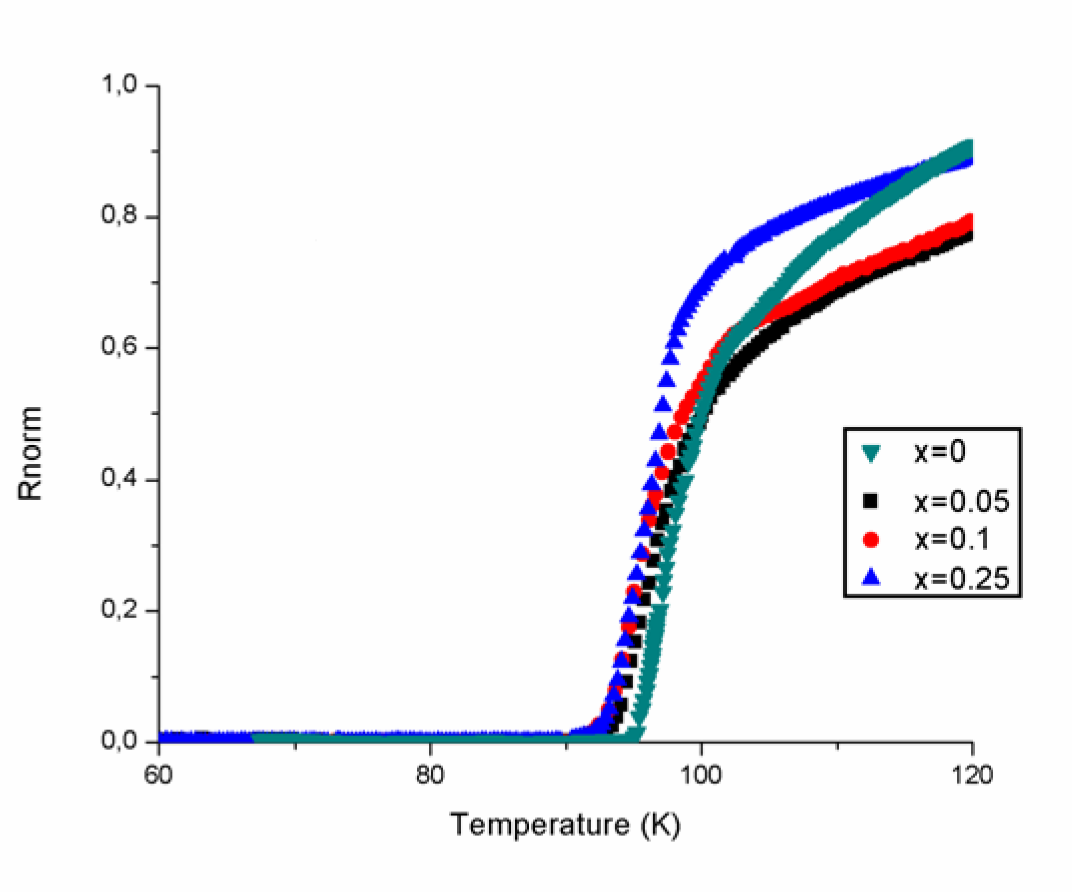
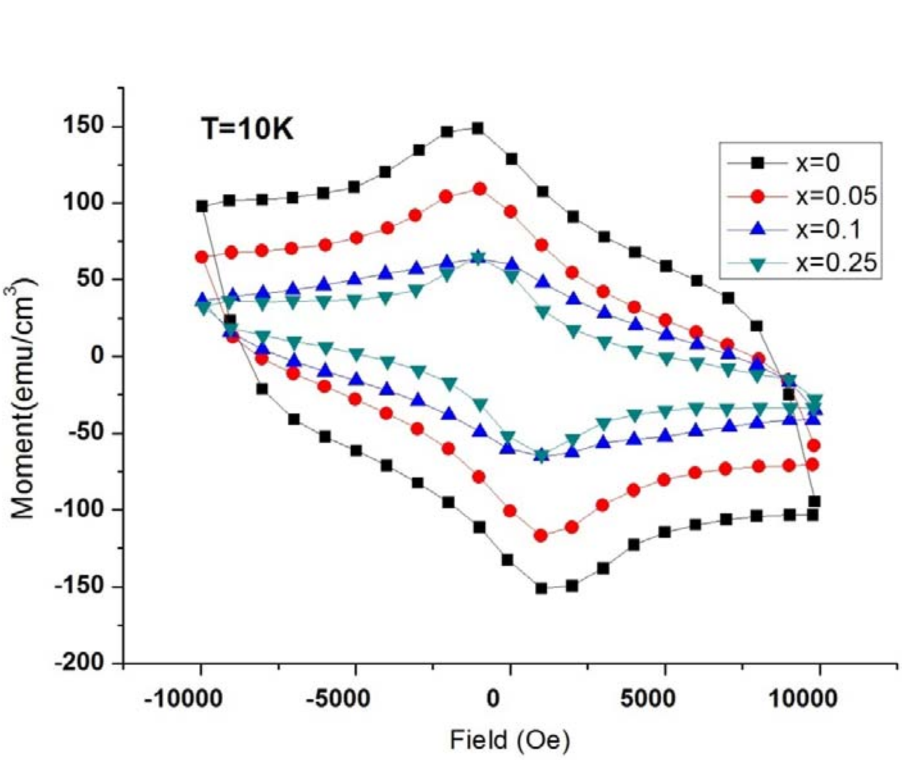


Figure 3



1,0
0,8
0,6
0,4
0,2
0,0
Rnorm
60
80
100
120
Temperature (K)
x=0
x=0.05
x=0.1
x=0.25

Figure 4



T=10K
x=0
x=0.05
x=0.1
x=0.25
Moment(emu/cm^3)
Field (Oe)
150
100
50
0
-50
-100
-150
-200
-10000
-5000
0
5000
10000

Figure 5

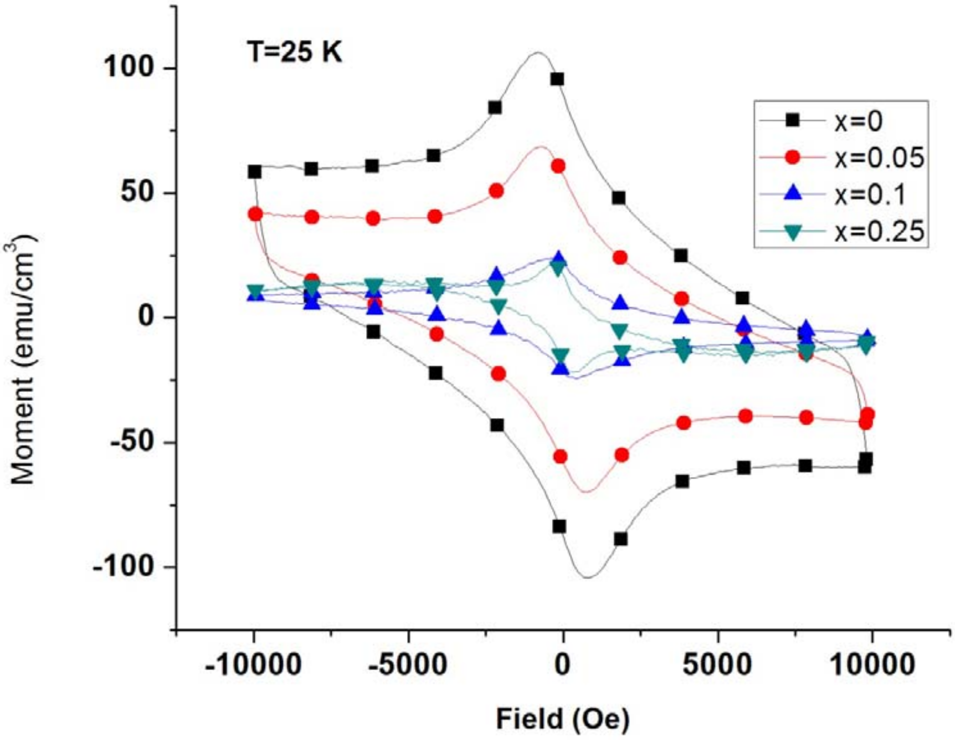
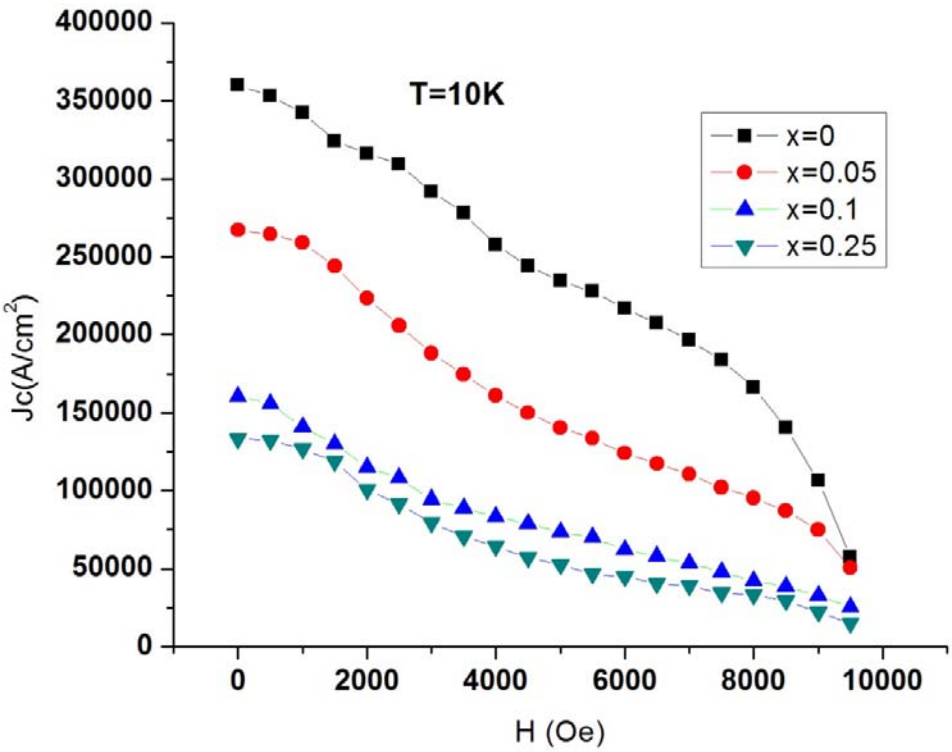

T=25 K
x=0
x=0.05
x=0.1
x=0.25
Moment (emu/cm³)
100
50
0
-50
-100
-10000
-5000
0
5000
10000
Field (Oe)

Figure 6



T=10K
x=0
x=0.05
x=0.1
x=0.25
Jc(A/cm^2)
H (Oe)
400000
350000
300000
250000
200000
150000
100000
50000
0
2000
4000
6000
8000
10000

Figure 7