



Mössbauer and magnetic studies of $(\text{La}_{0.8}\text{Ca}_{0.2})_{1-x}\text{Bi}_x\text{FeO}_3$ perovskites

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Abstract

The effect of Bi doping of $(\text{La}_{0.8}\text{Ca}_{0.2})_{1-x}\text{Bi}_x\text{FeO}_3$ perovskites on structural and magnetic properties was investigated. X-ray diffraction, magnetometry and Mössbauer spectroscopy measurements were performed on the samples. The sol gel prepared samples were annealed at 800 °C, 900 °C and 1200 °C. The samples crystallize in an orthorhombic structure with a PNMA space group. The LCBFO800 samples have Mössbauer spectra with large sextets in accordance with the small grain size. The several spectral components observed in the sample series are in accordance with the ones found in literature. Hyperfine magnetic fields decrease slightly with Bi content and increase with annealing temperature. Isomer shifts and quadrupole splitting stay constant with composition and annealing temperature. Magnetometry measurements show that the antiferromagnetic behavior is dominant over the ferromagnetic one. Bi doping increases coercivity and decreases remanent magnetization, while annealing temperature increases coercivity and remanent magnetization.

Keywords Perovskite-type oxides · Bismuth-substituted materials · Magnetism

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1 Introduction

Perovskite compounds of the type ABO_3 (A, B = metals) exhibit interesting properties as good catalysis performance, solid state electrodes, gas sensing, and are already being used in several technological applications [1–3]. Besides, the partial substitution of both sites can improve their properties.

In recent years, one of the most studied ferrites is the perovskite $La_{1-x}Ca_xFeO_3$ which has improved magnetic, dielectric and catalytic properties [4, 5].

Combining a low temperature transition from the ferromagnetic state to the paramagnetic state at $T_C = 670$ K [6] with being a good candidate for gas sensor applications, make it the subject of several studies on the effects of substitution by non-magnetic elements such as bismuth, the most diamagnetic element on Earth.

We present a study on $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ ($x = 0.00, 0.05, 0.10, 0.15, 0.20$ and 0.25) compounds prepared by sol-gel. The samples were annealed at different temperatures as 800, 900 and 1200 °C and then structural and magnetic studies were performed.

2 Experimental

The samples were prepared by the sol-gel method [7] using as raw materials lanthanum nitrate, bismuth nitrate, calcium nitrate, ferric nitrate and citrate acid (all analytically pure).

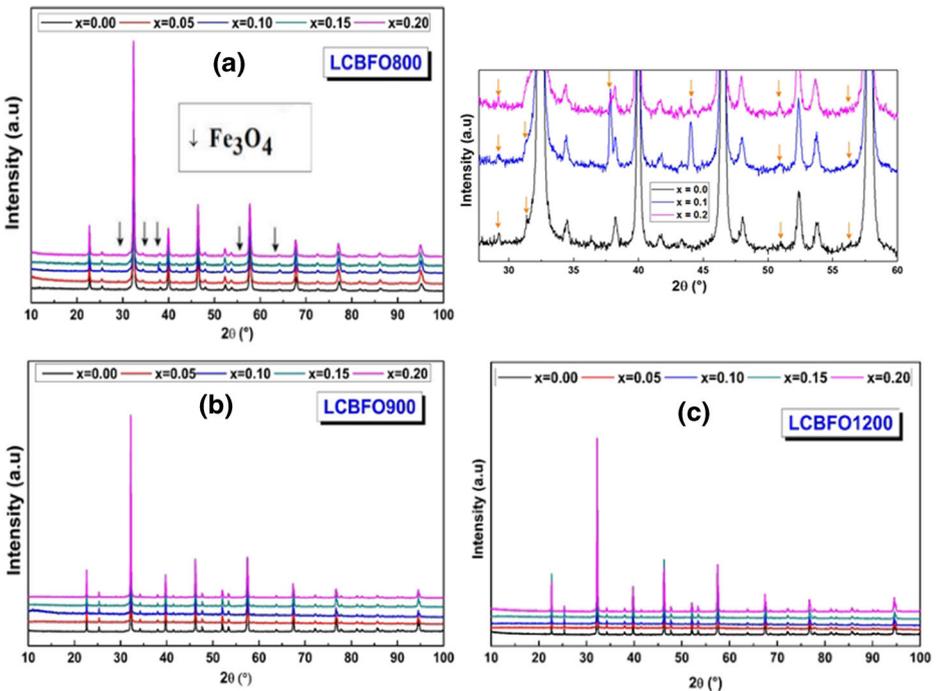


Fig. 1 XRD diffractograms for indicated (a) LCBFO800, (b) LCBFO900, (c) LCBFO1200 samples. On top right is a zoom of LCBFO800 patterns showing the peaks of Fe_3O_4 oxide

Table 1 Grains size as determined from diffraction patterns by Scherrer formula

Annealing temperature/composition	x = 0.0	x = 0.1	x = 0.2
800 °C	33.6(2)	36.4(2)	37.6(2)
900 °C	53.2(2)	55.7(2)	58.7(2)
1200 °C	60.4(2)	60.5(2)	61.7(2)

Annealing was done at 800 °C, 900 °C and 1200 °C and thus LCBFO800, LCBFO900 and LCBFO1200 samples were obtained.

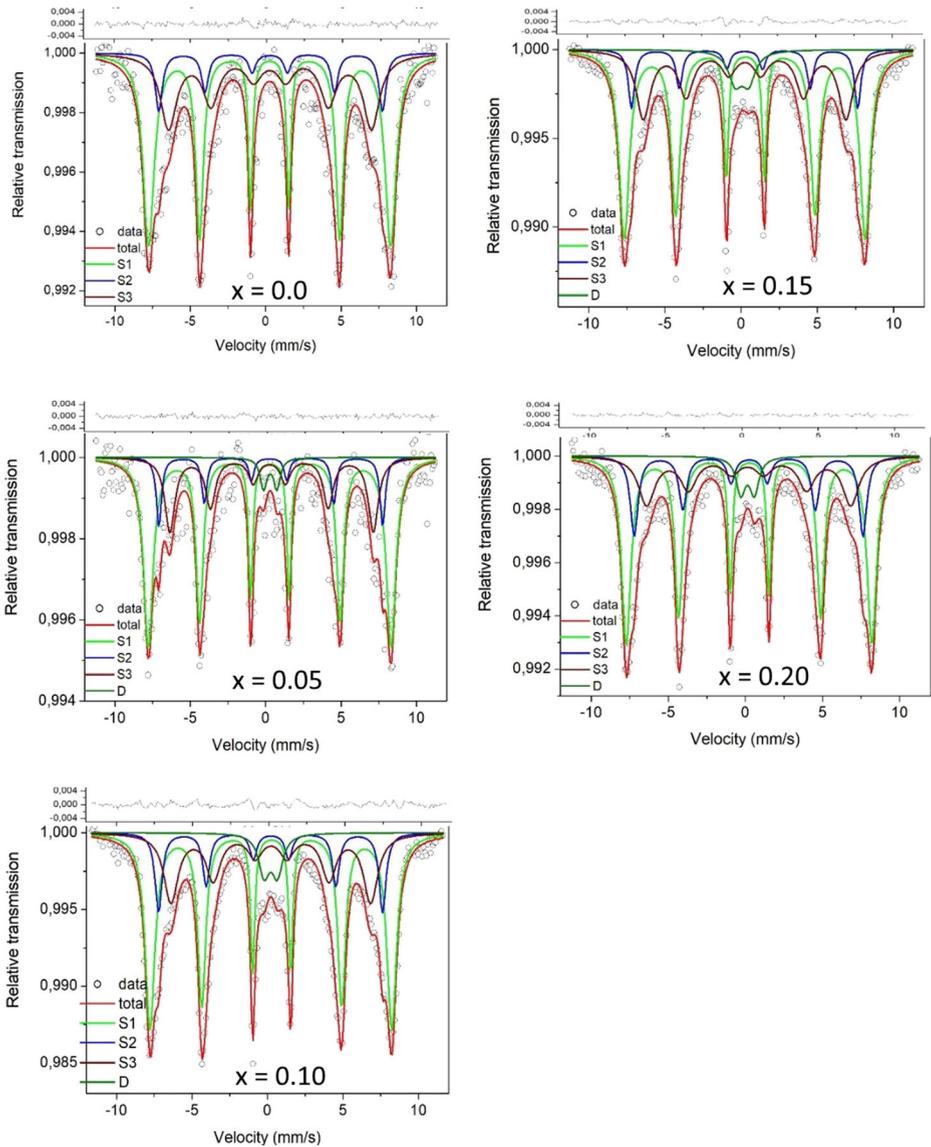


Fig. 2 Room temperature Mössbauer spectra of the $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ samples prepared at 800 °C. S stands for sextets and D for doublets

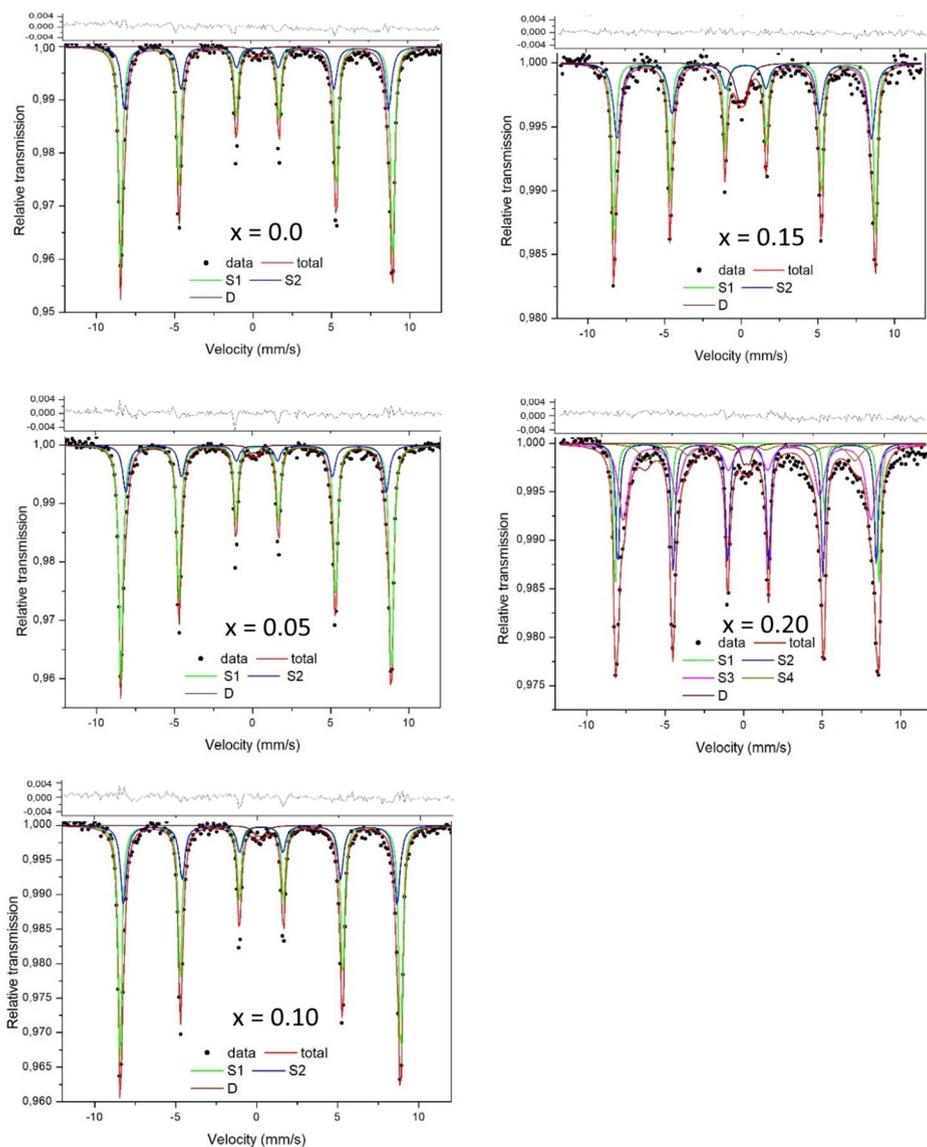


Fig. 3 Room temperature Mössbauer spectra of the $(\text{La}_{0.8}\text{Ca}_{0.2})_{1-x}\text{Bi}_x\text{FeO}_3$ samples prepared at 900 °C. S stands for sextets and D for doublets

The phase purity, homogeneity, lattice structure and cell parameters of the synthesized compounds were checked by X-ray diffraction (XRD) analysis, using a *BRUKER 8D ADVANCE* X-ray powder diffractometer, with $\text{CuK}\alpha$ radiation ($\lambda = 0.154184$ nm), in θ - 2θ Bragg-Brentano geometry. The acquisition was in the 2θ range of 5 – 100° , with a step of 0.02° and an acquisition time for each step of 1 s. The XRD data were also used to obtain the lattice parameters by means of Rietveld analysis [8] using the *FULLPROF* program, and also the crystallite sizes.

Mössbauer spectra were recorded at room temperature by a constant acceleration type spectrometer (*WissEL*) in transmission geometry, using the ^{57}Fe as isotope and a source

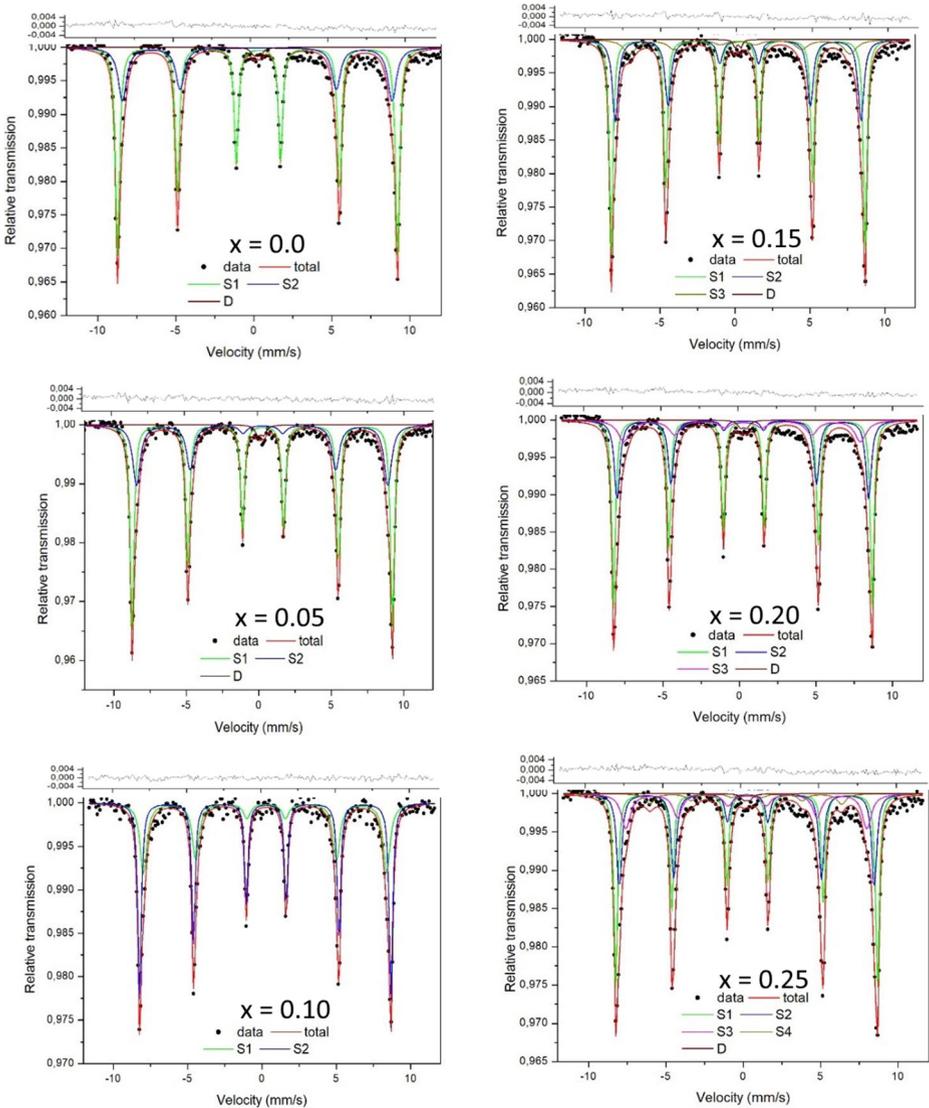


Fig. 4 Room temperature Mössbauer spectra of the $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ samples prepared at 1200 °C. S stands for sextets and D for doublets

of ^{57}Co (energy of 14.4 keV) in a matrix of Rh with an activity of about 20 mCi. The spectra were fitted by a set of Lorentzian lines determined by the least squares method, applying the *NORMOS* program distributed by WissEL GmbH. Isomer shifts are given, as usually, relatively to α -Fe measured at room temperature.

Magnetic measurements were done in a Vibrating Sample Magnetometer (VSM) with a cryogen-free *Dynacool PPMS*, operating at a vibration frequency of 40 Hz and amplitude of 2 mm in the central area of the coils. The samples were in a rod-shaped sample holder made of Perspex.

Table 2 Hyperfine parameters resulted from the fitting procedure of the spectrum showed in Fig. 2 for $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ samples prepared at 800 °C. Isomer shifts are given relatively to α -Fe

	IS (mm/s)	QS (mm/s)	B (T)	W (mm/s)	%	Subspectra
$x = 0,00$	0,37(1)	0,0002(1)	49,8(1)	0,34(1)	56,5	Sextet 1 (Fe3+)
	0,37(1)	-0,001(1)	46,2(1)	0,56(1)	11,1	Sextet 2 (Fe3+)
	0,38(1)	0,023(1)	41,7(1)	1,30(1)	32,4	Sextet 3 (Fe3+)
$x = 0,05$	0,38(1)	0,009(1)	49,9(1)	0,34(1)	59,7	Sextet 1 (Fe3+)
	0,39(1)	-0,045(1)	46,0(1)	0,42(1)	12,1	Sextet 2 (Fe3+)
	0,35(1)	0,026(1)	41,8(1)	0,90(1)	24,8	Sextet 3 (Fe3+)
$x = 0,10$	0,31(1)	0,88(1)	-	0,54(1)	3,4	Doublet (Fe3+)
	0,37(1)	0,055(1)	49,6(1)	0,36(1)	52,6	Sextet 1 (Fe3+)
	0,32(1)	-0,031(1)	46,1(1)	0,56(1)	15,0	Sextet 2 (Fe3+)
$x = 0,15$	0,34(2)	-0,017(1)	41,0(1)	1,14(1)	26,6	Sextet 3 (Fe3+)
	0,32(1)	0,89(1)	-	0,87(1)	5,8	Doublet (Fe3+)
	0,37(1)	0,046(1)	49,0(1)	0,34(1)	56,1	Sextet 1 (Fe3+)
$x = 0,20$	0,34(1)	-0,010(1)	46,0(1)	0,50(1)	10,4	Sextet 2 (Fe3+)
	0,34(2)	-0,003(1)	41,2(1)	1,13(1)	27,6	Sextet 3
	0,20(1)	0,88(1)	-	1,07(1)	5,9	Doublet (Fe3+)
$x = 0,20$	0,37(1)	0,035(1)	49,4(1)	0,35(1)	53,4	Sextet 1 (Fe3+)
	0,33(1)	-0,029(1)	46,2(1)	0,61(1)	18,4	Sextet 2 (Fe3+)
	0,29(2)	-0,078(1)	41,0(1)	1,28(1)	23,3	Sextet 3
	0,25(1)	0,89(1)	-	0,72(1)	4,9	Doublet (Fe3+)

IS- isomer shift; QS- quadrupole splitting; B- hyperfine magnetic field; W- full width at Lorentzian half maximum; %- percentage of site

3 Results and discussion

Figure 1 shows X-ray diffraction patterns of the LCBFO samples. They crystallize in the orthorhombic structure with Pnma space group. For LCBFO800 samples, also Fe_3O_4 secondary phase was identified with “X’Pert High Score Plus” program. The peaks attributed to this phase have small intensity (Fig. 1, top right) therefore the samples

Table 3 Hyperfine parameters resulted from the fitting procedure of the spectrum showed in Fig. 3 for $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ samples prepared at 900 °C. Isomer shifts are given relatively to α -Fe

	IS (mm/s)	QS (mm/s)	B (T)	W (mm/s)	%	Subspectra
$x = 0,00$	0,38(1)	-0,07(1)	53,7(1)	0,28(1)	64,6	Sextet 1 (Fe3+)
	0,36(1)	-0,06(1)	52,6(1)	0,54(1)	33,8	Sextet 2 (Fe3+)
	0,33(1)	0,39(1)	-	0,45(1)	1,6	Doublet (Fe3+)
$x = 0,05$	0,37(1)	-0,07(1)	53,7(1)	0,29(1)	68,5	Sextet 1 (Fe3+)
	0,37(1)	-0,07(1)	52,3(1)	0,58(1)	29,8	Sextet 2 (Fe3+)
	0,25(1)	0,39(1)	-	0,45(1)	1,7	Doublet (Fe3+)
$x = 0,10$	0,38(1)	-0,06(1)	53,6(1)	0,28(1)	68,5	Sextet 1 (Fe3+)
	0,38(1)	-0,07(1)	52,4(1)	0,54(1)	29,4	Sextet 2 (Fe3+)
	0,30(1)	0,39(1)	-	0,51(1)	2,1	Doublet (Fe3+)
$x = 0,15$	0,37(1)	-0,06(1)	52,1(1)	0,23(1)	16,2	Sextet 1 (Fe3+)
	0,37(1)	-0,07(1)	51,0(1)	0,29(1)	35,1	Sextet 2 (Fe3+)
	0,31(1)	0,37(1)	-	0,45(1)	2,4	Doublet (Fe3+)
$x = 0,20$	0,38(1)	-0,07(1)	52,9(1)	0,27(1)	52,0	Sextet 1 (Fe3+)
	0,37(1)	-0,09(1)	51,4(1)	0,56(1)	39,7	Sextet 2 (Fe3+)
	0,20(2)	0,42(1)	-	0,68(1)	8,3	Doublet (Fe3+)

IS- isomer shift; QS- quadrupole splitting; B- hyperfine magnetic field; W- full width at Lorentzian half maximum; %- percentage of site

Table 4 Hyperfine parameters resulted from the fitting procedure of the spectrum showed in Fig. 4 for $(\text{La}_{0.8}\text{Ca}_{0.2})_{1-x}\text{Bi}_x\text{FeO}_3$ samples prepared at 1200 °C. Isomer shifts are given relatively to $\alpha\text{-Fe}$

	IS (mm/s)	QS (mm/s)	B (T)	W (mm/s)	%	Subspectra
$x = 0,00$	0,38(1)	-0,064(1)	55.6(1)	0,32(1)	69.2	Sextet 1 (Fe3+)
	0,36(1)	-0,060(1)	53.5(1)	0,65(1)	29.3	Sextet 2 (Fe3+)
	0,33(1)	0,39(1)	-	0,45(1)	1.5	Sextet 3 (Fe3+)
$x = 0,05$	0,38(1)	-0,064(1)	55.6(1)	0,31(1)	65.5	Sextet 1 (Fe3+)
	0,37(1)	-0,055(1)	53.7(1)	0,62(1)	32.6	Sextet 2 (Fe3+)
	0,35(1)	0,026(1)	41,8(1)	0,90(1)	1.9	Sextet 3 (Fe3+)
$x = 0,10$	0,25(1)	0,39(1)	-	0,45(1)	3,4	Doublet (Fe3+)
	0,36(1)	-0,073(1)	50.7(1)	0,55(1)	38.9	Sextet 1 (Fe3+)
	0,37(1)	-0,061(1)	52.4(1)	0,32(1)	61.1	Sextet 2 (Fe3+)
$x = 0,15$	0,36(1)	-0,6(1)	52.4(1)	0,34(1)	53.9	Sextet 1 (Fe3+)
	0,36(1)	-0,07(1)	50.7(1)	0,50(1)	33.2	Sextet 2 (Fe3+)
	0,38(2)	-0,13(1)	45.4(1)	1,13(1)	12.0	Sextet 3 (Fe3+)
$x = 0,20$	0,31(1)	0,04(1)	-	0,32(1)	0,9	Doublet (Fe3+)
	0,37(1)	-0,056(1)	49,4(1)	0,32(1)	52.7	Sextet 1 (Fe3+)
	0,36(1)	-0,055(1)	46,2(1)	0,44(1)	29.0	Sextet 2 (Fe3+)
$x = 0,25$	0,33(2)	-0,0712(1)	41,0(1)	0,91(1)	17.0	Sextet 3 (Fe3+)
	0,39(1)	0,40(1)	-	0,45(1)	1.3	Doublet (Fe3+)
	0,37(1)	-0,062(1)	52,4(1)	0,32(1)	43.6	Sextet 1 (Fe3+)
$x = 0,25$	0,37(1)	-0,059(1)	51.1(1)	0,39(1)	30.9	Sextet 2 (Fe3+)
	0,35(2)	-0,055(1)	48.3(1)	0,71(1)	18.9	Sextet 3 (Fe3+)
	0,31(1)	-0,070(1)	38.4(1)	0,70(1)	5.6	Sextet 4 (Fe3+)
	0,35(1)	0,37(1)	-	0,45(1)	1.1	Doublet (Fe3+)

IS- isomer shift; QS- quadrupole splitting; B- hyperfine magnetic field; W- full width at Lorentzian half maximum; %- percentage of site

are considered as single phased. The samples annealed at 900 °C and 1200 °C do not show this secondary phase.

The increase in the annealing temperature promotes the increase in grain size, as observed by XRD and SEM [9]. The linewidth of diffraction peaks decreases with increasing of annealing temperature, and the grain size decreases concomitant. The sizes of crystallites were determined by Scherrer formula [10] and are displayed in Table 1 for samples with compositions $x = 0.0$, $x = 0.1$ and $x = 0.2$.

To have a detailed insight on the charge states and local environment of Fe nuclei, we carried out Mössbauer spectroscopy measurements of the LCBFO800, LCBFO900 and LCBFO1200 compounds. Each obtained spectrum was fitted by magnetic sextets and a central quadrupole doublet. The RT spectra are shown in Figs. 2, 3 and 4 and resulting fitting parameters are given in Tables 2, 3 and 4.

It is observed that within each series the spectra do not change significantly with increasing bismuth concentration.

The compounds are antiferromagnetic at room temperature and yield six-line spectra. A parametric Fe^{3+} site is also observed, probably due to an impurity not seen in XRD because of its small amount. The best resolved six-line spectra are for LCBFO900 and LCBFO1200 compounds. The LCBFO800 series has large sextets in accordance with the small grain size obtained by XRD. There is a great amount of magnetite in each of these samples, as already seen in literature for these type of compounds [6]. A Fe^{3+} ion sextet with a large linewidth and a hyperfine magnetic field of about 41.0 T is also observed, belonging to the perovskite compound [6]. For $x = 0.0$ and $x = 0.05$ this Fe^{3+} site is in tetrahedral coordination and for $x \geq 0.10$ changes to octahedral coordination. In addition, there is a paramagnetic doublet for $x \geq$

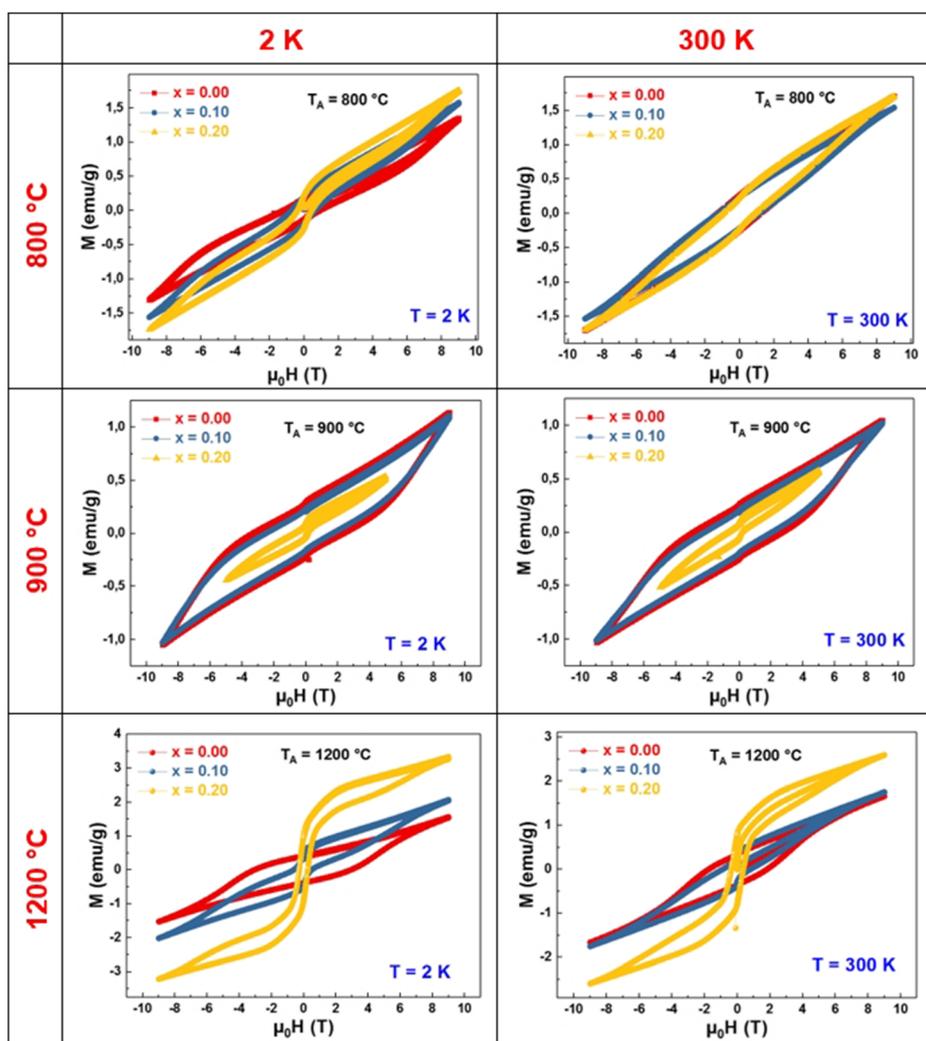


Fig. 5 Hysteresis curves obtained at 2 K and 300 K for the LCBFO samples annealed at 800 °C, 900 °C and 1200 °C

0.05. The hyperfine values of this Fe^{3+} doublet is also in accordance with literature [6]. No Fe^{4+} is found within the fitting procedure.

For the other series, LCBFO900 and LCBFO1200, only the sextets attributed to the compounds are observed. The parameters are of Fe^{3+} ions in accordance with literature [11–13].

In the case of LCBFO900 compounds there are two sextets in octahedral coordination with hyperfine magnetic fields of 52–54 T and 53–51 T, showing different environments for the Fe ions. For the LCBFO1200 series the values of the hyperfine magnetic fields differ in a certain extend and for $x \geq 0.15$ another sextet with a lower field appears with $H = 46\text{--}48$ T. At $x = 0.25$ a fourth sextet with $H = 38.4$ T exists. The appearance of several sextets corresponding to several Fe environments has been seen for these perovskites' compounds before [12–14].

Table 5 Magnetic parameters taken from hysteresis curves shown in Fig. 5. H_c , M_r , and M_{9T} represent magnetic coercive field, remanent magnetization and magnetization at 9 T, respectively

Composition	Measured T (K)	Annealing T (K)	H_c (T)	M_r (emu/g)	M_{9T} (emu/g)
x = 0.0	2	800	-1.308(2)	-0.125(2)	1.341(1)
		900	-3.302(2)	-0.224(2)	1.137(1)
		1200	-3.138(2)	-0.379(2)	1.547(1)
	300	800	-0.272(2)	-1.211(2)	1.714(1)
		900	-0.262(2)	-2.905(2)	1.055(1)
		1200	-2.055(2)	-0.408(2)	1.656(1)
x = 0.1	2	800	-0.853(2)	-0.208(2)	1.566(1)
		900	-2.421(2)	-0.159(2)	1.086(1)
		1200	-0.598(2)	-0.397(2)	2.058(1)
	300	800	-0.213(2)	-1.219(2)	1.539(1)
		900	-0.167(2)	-2.228(2)	1.019(1)
		1200	-1.202(2)	-0.254(2)	1.754(1)
x = 0.2	2	800	-0.427(2)	-0.237(2)	1.745(1)
		900	-0.642(2)	-0.047(2)	0.889(1)
		1200	-0.294(2)	-0.896(2)	3.335(1)
	300	800	-0.246(2)	-1.024(2)	1.698(1)
		900	-0.058(2)	-0.721(2)	0.980(1)
		1200	-0.411(2)	-0.761(2)	2.594(1)

As a summary, we have fitted the spectra in general with 3 sextets and one doublet. The obtained hyperfine fields increase with increasing annealing temperature, in accordance with the increase of grain size as observed by XRD, and at the same time linewidths of Lorentzians decrease. The area of the sextet with higher magnetic field (more than 50%) decreases with increase of Bi and increase with increase of annealing temperature. IS and QS are nearly constant with composition and annealing temperature.

Figure 5 shows $M(H)$ curves for the LCBFO compounds. It is observed that the hysteresis cycles reveal the presence of more than one contribution, being the antiferromagnetic interactions dominate (weak ferromagnetism). Table 5 resumes the parameters obtained from the hysteresis curves.

The increase of Bi doping enhances the ferromagnetic contribution which is confirmed by almost reaching a saturation for $x = 0.2$. The hysteresis curves at 4.2 K show that magnetization increases linearly with increasing Bi content. Moreover, Bi doping increases coercivity (H_c) and decreases remanent magnetization (M_r) for samples $x = 0.0$ and $x = 0.1$.

Concerning the effect of annealing temperature, T_A , it can be observed that T_A increases coercivity (H_c) and remanent magnetization (M_r). Regarding magnetization and particularly its value at a field of 9 T, it is higher at 1200 °C but lower at 900 °C, comparing to the $T_A = 800$ °C.

4 Conclusions

In this work, the effect of Bi doping of $(La_{0.8}Ca_{0.2})_{1-x}Bi_xFeO_3$ perovskites on structural and magnetic properties, including Mössbauer spectroscopy, was investigated. The annealing at various temperatures promote different grain sizes on the samples that

originate different properties of the sample batches. The samples crystallize in a orthorhombic structure with a PNMA space group. The LCBFO800 samples have Mössbauer spectra with large sextets in accordance with the small grain size. The several spectral components observed in the sample series are in accordance with found in literature. In general, the spectra were fitted with 3 Fe^{3+} sextets and a Fe^{3+} doublet. The area of the sextet with higher magnetic field is more than half of the spectra and in general decreases with increase of Bi content. With the increase of annealing temperature these area increase. Also, the hyperfine magnetic fields increase with the annealing temperature. Isomer shifts and Quadrupole Splittings stay nearly constant with composition and annealing temperature, and in accordance with literature values. Magnetometry measurements show that the antiferromagnetic behavior is dominate over the ferromagnetic one. Bi doping increases coercivity and decreases remanent magnetization, while annealing temperature increases coercivity and remanent magnetization.

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