www.amlett.org, www.amlett.com, DOI: <u>10.5185/amlett.2013.icnano.289</u> "ICNANO 2011" Special Issue Published online by the VBRI press in 2013

# Preparation and studies on $(1-x)BiFeO_{3-x}$ Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x = 0.25 and 0.5) multiferroic nanocomposites

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# **ABSTRACT**

Multiferroic nano-composite (1-x) BiFeO<sub>3-x</sub> Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x=0.25 and 0.5) have been successfully synthesized by mixing the two phases, prepared independently by two different methods followed by annealing at 600  $^{0}$ C. Existence of the two phases in the composite is confirmed by x-ray diffraction pattern. Average particle size is calculated to be about 45 nm for both of these phases. The saturation magnetization, remnant magnetization and coercive field increases linearly with increasing ferromagnetic phase (Li0.5Fe<sub>2.5</sub>O<sub>4</sub>) as investigated by VSM measurement. Local magnetic behaviors have been investigated by  $^{57}$ Fe Mössbauer spectroscopic studies. Large dielectric constant of the order of  $10^{3}$ - $10^{4}$  has been observed in these composites. Copyright © 2013 VBRI press

**Keywords:** BiFeO<sub>3</sub>; Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>; composite; multiferroic; VSM; 57Fe Mössbauer spectroscopy.



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## Introduction

Bismuth ferrite (BiFeO<sub>3</sub> or BFO) is one of the few multiferroic materials which show ferroelectric and ferromagnetic nature at room temperature. Such materials show magnetoelectric coupling where the magnetic properties can be controlled by applying electric field and electric properties can be controlled by applying magnetic field [1-3]. It shows G-type antiferromagnetic ordering below Neel temperature  $T_N = 370$  °C with a weak ferromagnetism and ferroelectric ordering below  $T_C = 830$ <sup>0</sup>C [4-6]. To increase the magnetism in BFO, many strategies like reducing the size of BiFeO<sub>3</sub> particles, doping in A or B-site have been proposed and investigated [7-12]. The magnitude of the magnetoelectric coupling, which can be written as  $(\varepsilon(H) - \varepsilon(0)) / \varepsilon(0)$  where  $\varepsilon(0)$  and  $\varepsilon(H)$  are values of dielectric constant at zero and H magnetic field, is still found to be small in the single phase BiFeO<sub>3</sub> at room temperature.

In search of high value of magnetoelectric coupling, researchers synthesized composites of ferroelectric and ferromagnetic compounds namely magnetoelectric composites. The value of ME coupling is greatly enhanced in these composite materials. The large value of the ME coupling was first predicted theoretically in multilayer magnetoelectric composites by Harshe et al. [13]. Since then ME properties in composite materials have been investigated in great details theoretically [14, 15]. Experimentally ME effects have been investigated in bulk, nano and thin film multilayer of composite materials. BaTiO<sub>3</sub>-CoFe<sub>2</sub>O<sub>4</sub> [16], Pb(Zr<sub>0.52</sub>Ti<sub>0.48</sub>)O<sub>3</sub>-NiFe<sub>2</sub>O<sub>4</sub> [17] thin films, BaTiO<sub>3</sub>-MgFe<sub>2</sub>O<sub>4</sub> [18], BaTiO<sub>3</sub>-BiFeO<sub>3</sub> [19] bulk

and Ni<sub>0.75</sub>Co<sub>0.25</sub>Fe<sub>2</sub>O<sub>4</sub>–BiFeO<sub>3</sub> [**20**], and ZnFe<sub>2</sub>O<sub>4</sub>–BiFeO<sub>3</sub> [**21**] nano composites, are some of the examples of the ME composites studied.

Lithium ferrites (Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>) is known to occur in two different crystalline form, (a) ordered Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (known as α- Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>) which crystallizes in spinel structure with space group P4<sub>3</sub>32 and is formed below 750 <sup>o</sup>C in solid state reaction method and (b) disordered Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> which crystallizes in inverse spinel structure with space group Fd3m and formed around 1000 °C in solid state reaction method [22-24]. The magnetic ordering temperature is above 900 K [24]. It has been used as an important component of microwave devices and memory core applications due to its high Curie temperature, high saturation magnetization and hysteresis loop properties which are suitable for this kind of performance [25-27]. No work has been reported on BiFeO3 and Li<sub>0.5</sub>Fe<sub>2.5</sub>O4 based ME nano-composites to the best of our knowledge. In this paper, we present synthesis and magnetic studies of (1-x) BiFeO<sub>3</sub>–x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x=0.25 and 0.5) nano-composite.

# **Experimental**

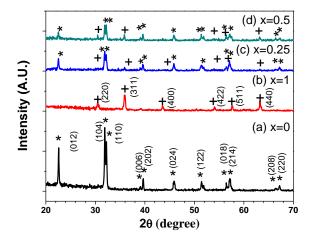
# Nanoparticles synthesis

Nanoparticles of Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> were synthesized by citric acid nitrate method. All the materials were of high purity (better than 99.9%) and used without further purification. Firstly, LiNO<sub>3</sub> was prepared by reacting required amount of Li<sub>2</sub>CO<sub>3</sub> in concentrated nitric acid (HNO<sub>3</sub>) with magnetic stiring. Stoichiometric amount of iron nitrate (Fe(NO<sub>3</sub>)<sub>3</sub>, 9H<sub>2</sub>O) and citric acid (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>) were then added to the LiNO<sub>3</sub> solution and stirred at 90°C for 1 hour. The solution was then kept in an oven at 80°C for 24 hours to evaporate the solution which resulted into solid precursor. The dried precursor was annealed at 600°C for 4 hours in a programmable box furnace to get nano-sized Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> particles. BiFeO<sub>3</sub> nanoparticles were prepared by combustion method without using any solvent. Bismuth and iron nitrate were taken in stoichiometric amount, mixed in presence of glycine (H<sub>2</sub>NCH<sub>2</sub>COOH) at 80<sup>o</sup>C for 30 minutes and then heated at 150 °C for combustion. The powder sample after combustion were collected, grounded using mortar-pastel and annealed at 600°C for 4 hours which results into BiFeO<sub>3</sub> nanoparticles. Nano-composite of (1-x) BiFeO<sub>3</sub>+x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> of composition (x=0.25 and 0.5) were prepared by mixing the required amount of BiFeO<sub>3</sub> and Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> synthesized nanoparticles in acetone medium for 2 hours and further annealing at 600°C for 4 hours with programmable heating.

# Characterization tools

In order to investigate the crystal structure and calculate the average crystallite size small amount of the annealed powder was characterized by x-ray diffraction (XRD) on a ARL X'TRA X-ray diffractometer (Thermo Electron Corporation) using Cu-  $K\alpha$  radiation (wavelength =1.54 Å). Crystallite size was calculated from the Scherrer formula using the peak broadening of XRD peaks after correcting for the instrumental broadening. Magnetization as a function of applied magnetic field (hysteresis loop) was measured using a vibrating sample magnetometer (VSM, ADE Technologies, USA) upto an applied field of

1.5 T at room temperature. About 60-70 mg of each powder sample was sandwiched between two pieces of cello tape inside a copper ring and used as absorber for the Mössbauer measurements in the transmission geometry using a conventional <sup>57</sup>Fe constant acceleration Mössbauer spectrometer employing 25 mCi <sup>57</sup>Co (embedded in Rh matrix) source. The recorded data were analyzed by using a least square program considering Lorentzian line shapes in the spectrum. The parameters were calculated by taking BCC iron spectrum as calibration.



**Fig. 1.** Room temperature powder XRD pattern of (1-x) BiFeO $_3$ - xLi $_0.5$ Fe $_2.5$ O $_4$  for x= (a) 0.00, (b) 1.00, (c) 0.25 and (d) 0.50 where the peaks marked with '\*' corresponds to BiFeO $_3$  phase and '+' corresponds to Li $_0.5$ Fe $_2.5$ O $_4$  phase.

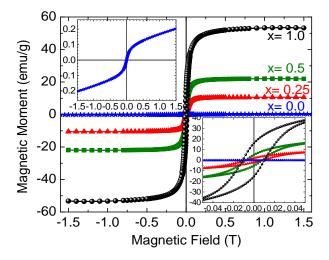
# Results and discussion

Structural analysis

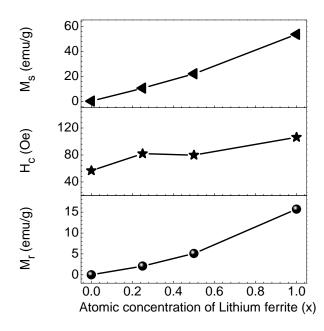
Fig. 1 shows the X-ray diffraction pattern of (1-x) BiFeO<sub>3</sub> $xLi_{0.5}Fe_{2.5}O_4$  for (x=0.00, 0.25 0.50 and 1.00) nano composites. All the peaks in Fig. 1a correspond to pure BiFeO<sub>3</sub> phase in distorted perovskite structure of space group R3c. No impurity peaks (like Bi<sub>2</sub>Fe<sub>4</sub>O<sub>9</sub>, α-Fe<sub>2</sub>O<sub>3</sub>, γ-Fe<sub>2</sub>O<sub>3</sub> and Fe<sub>3</sub>O<sub>4</sub>) has been found. The crystal parameters are calculated to be a=5.5829Å and c=13.8808Å for BiFeO<sub>3</sub> nanoparticles from the refinement of the XRD data. XRD peaks indexed as (220), (311), (400), (422), (511), (440) in Fig. 1b correspond to the spinel phase of Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> with space group P4<sub>3</sub>32. The peaks of both these two phases are broadened due to nano size structure of the samples. The XRD pattern of two composites namely x=0.25 and x=0.5 shows existence of both the phases. No impurity phases have been found during the preparation of composites from the individual phases. The average crystallite size has been calculated for all the samples from the peak broadening (from Scherrer's formula) after Correction from the instrumental broadening. The average crystallite size of the BiFeO<sub>3</sub> and Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> is calculated to be 44 and 40 nm respectively. The average crystallite sizes do not change much during preparation of final composites. The annealing temperature of the phases was chosen to be same (600°C) as that used to prepare the independent phases which could be the reason for no change in the particle size at the final composite making step.

#### Magnetic measurements

The variation of magnetization as a function of applied magnetic field (hysteresis curves), measured using a vibrating sample magnetometer (VSM) at 300K, for the samples (1-x) BiFeO<sub>3</sub>– x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>(x=0.00, 0.25 0.50 and 1.00) have been plotted in **Fig. 2**. Below inset of **Fig. 2** gives zoomed hysteresis loop in low field region to see the composite field clearly. The magnetic hysteresis loops for the pure BiFeO<sub>3</sub> nanoparticle is separately plotted in the above inset picture of **Fig. 2** with different scale.



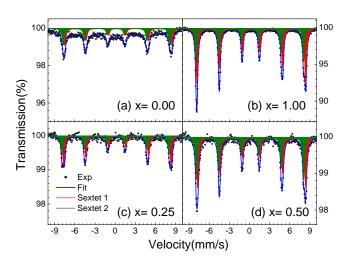
**Fig. 2.** Room temperature magnetic hysteresis curves of (1-x) BiFeO<sub>3- x</sub> Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> ( $x=0.00,\ 0.25,\ 0.5$  and 1.00) nano-composite. (Inset below) magnetic hysteresis curves at lower field region have been shown. (Inset above) Hysteresis loop of pure BiFeO<sub>3</sub> nanoparticles has been shown.



**Fig. 3.** Variation of the a) magnetization at highest applied magnetic field (1.75 T) (MS), b) remanent magnetization (Mr) and c) coercive field (HC) as function of atomic concentration (x) of the magnetic ( $\text{Li}_{0.5}\text{Fe}_{2.5}\text{O}_4$ ) phase.

It shows weak ferromagnetic behavior .The magnetization does not saturate upto highest applied fields investigated (1.5 T). Noticeable hysteresis with non-zero

remnant magnetization and coercive field of about ~ 56 Oe can be found. The value of magnetization is nearly double than that of bulk BiFeO<sub>3</sub> samples indicating fair amount of increase in the magnetization just by decreasing the particle size. The increase in magnetization in nano BFO can be due to suppression of spin spiral structure of periodicity 62 nm. The hysteresis loop for the Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> nanoparticles looks like a typical ferromagnetic loop with high saturation magnetization M<sub>s</sub>~ 53.53 emu/g. and coercive field value of 106 Oe. The room temperature saturation magnetization for Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> is reported to be 55 emu/g and shows magnetic transition at 912 K [24]. The magnetization of the nanocomposites with x=0.25 and x=0.5 is expectedly between that of BiFeO<sub>3</sub> and Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>. The variations of saturation magnetization (M<sub>s</sub>), remnant magnetization (M<sub>r</sub>) and coercive field (H<sub>c</sub>) for all the samples are plotted in Fig. 3. All these three quantities increase almost linearly with increasing Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> phase. The magneto-crystalline anisotropy constant can be written from Stoner Wohlfarth theory [28, 29] as  $K = H_c M_s \mu_0/2$  where  $\mu_0$  is the free space permeability and has the value of  $4\pi \times 10^{-7}$ . The magnetocrystalline anisotropy in these nano-composites increases with increasing the concentration of Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (magnetic phase) concentration as both M<sub>s</sub> and H<sub>c</sub> are increasing.



**Fig. 4.** Mössbauer spectrum of the (1-x) BiFeO3- x Li0.5Fe2.5O4 (x= 0.00, 0.25, 0.5 and 1.00) nano-composite measured at 300 K.

**Table 1.** Mössbauer parameters of (1-x) BiFeO<sub>3</sub>- x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x= 0.00, 0.25, 0.5 and 1.00) nano-composite measured at 300 K.

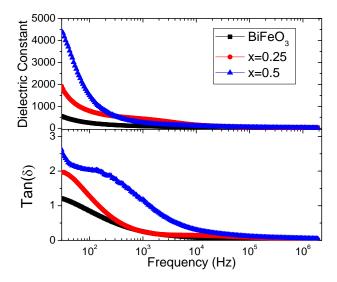
Sample	Sextet	IS (mm/s)	QS (mm/s)	LWD (mm/s)	HMF (T)	AREA (%)
x =0.00	1	0.37	0.03	0.36	49.6	52
	2	0.30	0.02	0.34	49.4	48
x = 1.00	1	0.35	0.02	0.33	50.3	65
	2	0.19	-0.02	0.29	49.6	35
x = 0.25	1	0.40	0.06	0.32	49.7	50
	2	0.20	0.01	0.32	49.6	45
x =0.50	1	0.39	0.03	0.33	49.9	57
	2	0.19	0.00	0.30	49.7	43

Mössbauer spectroscopic studies

<sup>57</sup>Fe Mössbauer spectroscopy is one of the most efficient tools to investigate the local magnetic behavior and oxidation state of the iron atoms in the matrix. The

Mössbauer spectra for (1-x) BiFeO<sub>3</sub>– x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x= 0.00, 1.00, 0.25 and 0.5) nano-composites are shown in **Fig. 4a** to **4d** respectively. The black dots represent the experimentally recorded data points whereas the blue lines are the least square fit of the spectrum. The parameters extracted from the best fit data are given in **Table 1**.

There is an error of about  $\pm 0.02$  mm/s in isomer shift, quadruple splitting and linewidth whereas the error associated with hyperfine field and area ratio is about ±0.1 T and  $\pm 2\%$  respectively. The spectrum for BiFeO<sub>3</sub> could be fitted with two sextets (have shown in the figure by red and green color) of isomer shift ranging from 0.30 to 0.37 mm/s, nearly zero QS and nearly equal magnetic hyperfine field of 49.4 to 49.6 Tesla. No other iron containing impurity phases have been found. Fig. 4b represents the Mössbauer spectrum of the Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> in the spinal cubic structure which can also be fitted with two sextets of isomer shifts 0.35 and 0.19 mm/s and hyperfine magnetic field of 50.3 and 49.6 T respectively. These values are similar to those reported in [30]. The hyperfine values of BiFeO<sub>3</sub> and Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> nanoparticles are close to each other. However, the percentage of transmission for the case of BiFeO<sub>3</sub> nanoparticles (less than 2%) is much less than that of Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (more than 10%). The Mössbauer spectra of two composite materials with x=0.25 and x=0.5 (shown in Fig. 4c and d) should have all the four sextets coming from both the ferroelectric and ferromagnetic phases. But the parameters of these two phases are close to each other and so the spectrum for the two composites could be well fitted with two sextets with isomer shifts 0.40 and 0.20 mm/s with acceptable line width values (0.30-0.33 mm/s). The isomer shift which is a measure of the oxidation state of Fe-atoms is found to be 0.19-0.40 mm/s. These values indicate that Fe-atoms are in Fe<sup>3+</sup> states in all the samples but have two different kinds of surroundings.



**Fig. 5**. Dielectric constant ( $\epsilon$ ') and loss tangent (Tan( $\delta$ )) as a function of frequency of (1-x) BiFeO<sub>3-x</sub> Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x= 0.00, 0.25 and 0.5) nanocomposite measured at 300K.

#### Dielectric studies

The variation of dielectric constant ( $\epsilon$ ') and loss tangent (Tan( $\delta$ )) of the two composites along with BiFeO<sub>3</sub>

nanoparticles are plotted in Fig. 5 as a function of frequency in the range 30Hz- 2MHz. One can immediately see that all the samples have a very high value of dielectric constant in low frequency region which is about one order of magnetite higher than reported in other BiFeO<sub>3</sub>-based composites like ZnFe<sub>2</sub>O<sub>4</sub>–BiFeO<sub>3</sub> [21], MnFe<sub>2</sub>O<sub>4</sub>–BiFeO<sub>3</sub> [31] and CuFe<sub>2</sub>O<sub>4</sub>–BiFeO<sub>3</sub> [32]. However similar kind of dielectric dispersion with large value of dielectric constant in low frequency range has been found in  $\text{Li}_{0.5}\text{Co}_{0.75}\text{Fe}_2\text{O}_4-$ BaTiO<sub>3</sub> composites [33]. Such a value of the dielectric constant at low frequency and the low value ( $\approx 20$ ) at high frequency is a signature of large Maxwell-Wagner type dielectric dispersion [34, 35]. The inhomogeneity in the microstructure (grain structure or grain boundary) in these composites of ferroelectric and ferromagnetic phase can give rise to space charge polarization which can in turn result into such high value of dielectric constant at low frequencies.

## Conclusion

Nano-composite of (1-x) BiFeO<sub>3</sub>–x Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub> (x=0.25 and 0.5) with average crystallite size of about 45 nm have been successfully prepared. X-ray diffraction data shows that nanoparticles are single phase in nature and crystallize in the same structure as the bulk compound. Magnetization and coercive field increases with increasing ferromagnetic phase as measured by VSM. Room temperature Mössbauer spectrum also confirmed that the increase in magnetization is intrinsic and not resulting from any impurity phases. The dielectric constant is increased to high values  $10^3$ - $10^4$  by making composite with Li<sub>0.5</sub>Fe<sub>2.5</sub>O<sub>4</sub>.

# Acknowledgements

This work has been supported by the project Volubilis AI n°: MA/11/248 and by CNRST/CNRS cooperation (Project chimie 1009). We are grateful to the "Association Marocaine des Chimistes Théoriciens" (AMCT) for its pertinent help concerning the programs.

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