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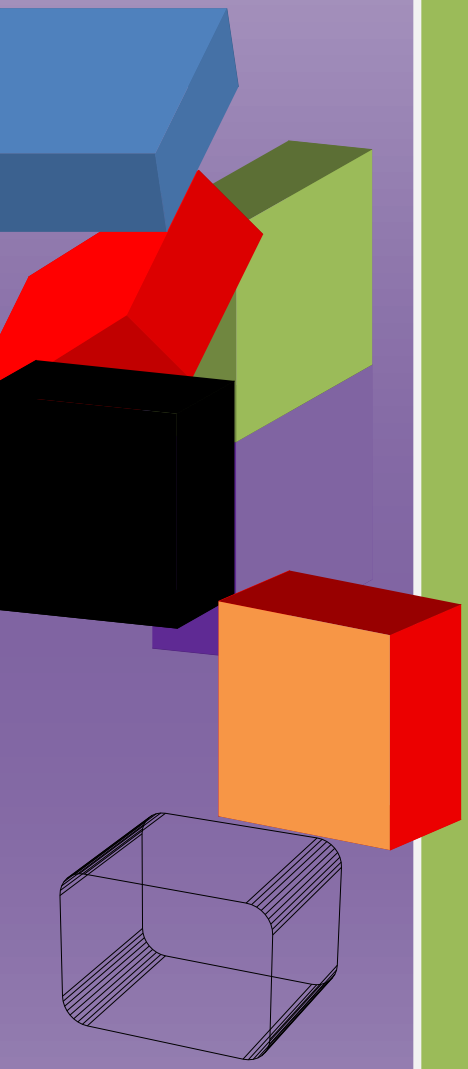

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STRUCTURAL, ELECTRICAL AND THERMOELECTRIC POWER MEASUREMENT STUDIES OF POLYOL ROUTE SYNTHESIZED $LI_{0.5}FE_{2.5}O_4$

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Abstract

Lithium ferrite powder was synthesized by a novel polyol method. The X-ray diffraction results indicate that the synthesized nanocrystalline lithium ferrite have only spinel structure without the presence of other phase impurities. FT-IR data indicates two strong bands observed in spectrum which is well matched with spinel structure. The morphology and particle size of the spinel ferrite powder, as revealed by SEM analysis. It shows that the $Li_{0.5}Fe_{2.5}O_4$ sample contain uniform and homogenous particles. The electrical behavior clearly indicates that the present ferrite have semiconductor like nature. Thermoelectric power measurement also confirms n-type semiconducting nature of lithium ferrite. A room temperature magnetization result shows a ferromagnetic behavior of the lithium ferrite. The present work describes the structural, electrical and magnetic properties of lithium ferrite.

Keywords: *lithium ferrite, Polyol synthesis, SEM, Magnetization*

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1.0 Introduction

Spinel ferrite nanoparticles are being extensively investigated in recent years because of their remarkable electric and magnetic properties having wide practical applications in magnetic recording, electronics, catalysis, information storage system, ferrofluid technology, magnetocaloric refrigeration and medical diagnosis [1-4]. Among the spinels, lithium ferrite is a candidate of particular interest due to its high saturation magnetization, high coercivity, strong anisotropy and excellent chemical stability. It is well known that most of the physical and chemical properties of ferrites depend strongly on the size, shape, composition and microstructure of the particles which are sensitive to the preparation methodology and preparative parameters used in their synthesis [5-6].

In our previous research work, $Li_{0.5}Fe_{2.5}O_4$ is an inverse spinel ferrite in which Li^+ ions occupies the octahedral (B sites) and Fe^{3+} occupies the tetrahedral (A sites) of the spinel lattice[7]. Lithium and substituted lithium ferrites are useful for microwave devices such as isolators, circulars, gyrators, phase shifters, cathode materials and memory cores owing to their high Curie temperature, high resistivity, low eddy current losses, high saturation magnetization and hysteresis loop properties, which offer performance advantage over other spinel structures [8–11]. Due to various applications our research group [12-15] studied the structural, electrical and magnetic properties of chromium and manganese substituted Li-ferrites synthesized by the sol-gel method.

Several synthesis methods such as forced hydrolysis, co-precipitation, combustion reaction and sonochemical have been suggested for nanocrystalline lithium ferrite preparation. Among various methods for synthesizing ferrites, the polyol method stands out as an alternative and highly promising method. Polyol method is a low temperature synthesis technique that offers a unique mechanism. The powder characteristics such as crystallite size, surface area, size distribution and nature of agglomeration are dependent on the nature of the fuel and fuel-to-oxidizer ratio. Among the various control parameters

in a polyol process, fuel plays an important role in determining the morphology, phase, and particulate properties of the final product.

In the present study, lithium ferrite nanoparticles were synthesized from a polyol method. This method does not require the addition of any other chemicals to the solution, and it has the advantages of simplicity, a low cost, a lack of by-product effluents, and an environmentally friendly operation. In the present research work, preparation of nanosized lithium ferrite by polyol route has been discussed, along with various characterization techniques, viz. XRD, SEM, FT-IR, TEP and VSM etc.

2.0 Experimental details

2.1 Synthesis

Lithium ferrite has been synthesized by a polyol-mediated route. High purity (AR grade) Ferric Nitrate, Lithium nitrate were used as raw materials. The stiochiometric amounts of individual metal nitrates were dissolved in doubly distilled deionized water to get a clear, transparent solution. The solution ($25cm^3$) was mixed with $25cm^3$ of ethylene glycol and refluxed at 100 ◦C for 1 h. Sodium hydroxide (molar ratio, Sodium /cation = 2.5) was dissolved in $25cm^3$ of water and mixed with $25cm^3$ ethylene glycol and this solution was added to the clear solution containing the precursor ions in water and ethylene glycol mixture. This mixture was then refluxed at 180 ◦C for 4 h to get the precipitate of the oxide. The precipitate obtained was separated by centrifugation, washed with acetone and ethyl alcohol followed by drying in an oven at 90 ◦C for 5 h. After drying, all samples were calcined in air at 500 ◦C for 4 h.

2.4 Characterization

The phase formation of the sintered samples was confirmed by x-ray diffraction studies. (Philips PW-1710 X-ray diffractometer with CuKα radiation λ=1.57058Å). The

FTIR spectra were recorded using Perkin Elmer FTIR in KBr pellets. Grain size was measured using a Scanning electron microscope (SEM).Two probe technique was employed to measure the D.C. resistivity of the sample in the temperature range of room temperature to 723 K. Silver paste was applied to both the surfaces of the pellets for good ohmic contacts. Magnetic study was carried out by using B-H loop traces technique. Magnetic measurements of all the compositions were carried out by using a high field vibrating sample magnetometer. The measurements were done at room temperature. Saturation magnetization (Ms), coercive field (Hc) and remenant magnetization (Mr) of the samples were studied from the hysteresis loops of respective curves.

3.0 Results and discussion

3.1 X-ray diffraction study

XRD pattern of newly polyol route synthesized lithium ferrite are shown in **Fig. 1.** The XRD patterns clearly indicate that the prepared samples contain cubic spinel structure. The crystallite size of sintered ferrites was calculated from the full width at half maxima of the most intense (311) peak by using Scherrer's formula.

$$t = 0.9\lambda / \beta \cos\theta$$

Where, symbols have their usual meaning.

The crystallite size of this sample is 22 nm indicates that sample is nanocrystalline range. The X-ray density was calculated according to the formula

$$dx = 8M/Na^3$$

where, N = Avagadros number (6.023 X 10^{23} atom/mole)

M = Molecular weight, and

a = lattice constant which was calculated from the X-ray diffraction pattern.

The results of lattice constant, crystallite size and X-ray density are as given in **Table 1.**

3.2 Scanning Electron Microscopy and Energy dispersion x-ray analysis

The morphology and composition of the lithium ferrite has been determined using the Scanning Electron Microscope (SEM). SEM image of the lithium ferrite powder was shown in the **Fig. 2.** The SEM image indicates that sample shows grains are uniform and homogeneous in nature.

3.3 Infrared Absorption Spectrum

FTIR spectra were recorded to confirm the formation of nanocrystalline lithium ferrite which is shown in **Fig. 3.** The high frequency band 550 cm^{-1} is due to the vibration of the tetrahedral M· · ·O bond and the low frequency band 370 cm^{-1} is due to the vibration of the octahedral M· · ·O bond in the crystal lattices of the lithium ferrite sample. The vibrational frequencies depend on cation mass, cation–oxygen distance and the bonding force.

3.4. Electrical resistivity study

The temperature dependence electrical resistivity of lithium ferrite sample was obtained in the range of 10^6 - 10^{10} Ωcm. **Fig. 4.** show the plots of log ρ vs 1000/T for the sample which are almost linear without any break indicating their semiconducting nature. The relationship $\rho = \rho_o \exp \Delta Ea/kT$ is found to be obeyed for composition. It is seen from the figure that the value of log ρ resistivity decreases with increasing temperature for lithium ferrite sample. The decrease in resistivity with an increase in temperature is because of increase in drift mobility of charge carriers. The observed behavior clearly indicates that the present ferrite have semiconductor-like behavior. The resistivity is due to the presence of Fe^{2+} ions. The decrease in resistivity arises due to the mobility of the extra electron, which comes from Fe^{2+} through the crystal lattice. The movement is described by a hopping mechanism, in which the charge carriers jump from one ionic site to the next [16]. The conduction is thus assumed to occur according to a hopping

mechanism from Fe^{2+} to Fe^{3+} ions on B-sites. The decrease of the electrical resistivity with increasing temperature may be related to the increase of the drift mobility of thermally activated charge carriers (electron and hole) according to hopping conduction mechanism.

3.5. Thermoelectric power measurement

The composition variation of seebeck coefficient as a function temperature as shown in **Fig.5.** All the samples are n-type semiconductor. The conduction mechanism in n-type specimens is predominantly due to the electrons from Fe^{2+} to Fe^{3+} ions.

3.6. Magnetic study

A magnetic property of lithium ferrite sample was carried out by using vibrating sample magnetometer (VSM) and it is depicted in **Fig. 6.** This sample shows a typical S-type shape in M–H curve, though the coercive force is 190.49Oe. The saturation and remanent magnetization were 51.98 and 13.62emu/gm respectively. The magnetic data for the sample reveals that, it is ferrimagnetic in nature. Thus as the particle size decreases and the magnetization increases.

4.0 Conclusions

We have succeeded in synthesizing spinel lithium ferrite nanoparticles by a polyol method. The SEM image indicates that sample shows grains are uniform and homogeneous in nature. The resistivity is decreasing with increasing temperature for the samples indicate that lithium ferrite have semiconductor-like behavior. Thermoelectric power measurement also confirms N-type semiconducting nature. The magnetic studies showed that the saturation magnetization and remanent magnetization of the lithium ferrite nanoparticles indicates sample is strongly ferrimagnetic in nature. This simple,

cost-effective, and environmentally friendly method that produces no by-product effluents can be used to synthesize pure crystalline spinel lithium ferrite nanoparticles.

References

[1] S. Sun Adv. Mater. 18 (2006) 393–403.

[2] L. Suber and D. Peddis, Magnetic Nanomaterials (2010) vol. 4 (New York: Wiley)

[3] P. Tartaj, M. Del Puerto Morales, S. Veintemillas-Verdaguer, T. Gonzales Carreno C. J. Serna, J. Phys. D: Appl. Phys. 36 R (2003) 182–97

[4] L. Zhao, H. Yang, L. Yu, Y. Cui, X. Zhao, B. Zou, S. Feng, J. Magn. Magn. Mater. 301 (2006) 445–51.

[5] E.K. Mooney, J.A. Nelson, M.J. Wagner, Chem. Mater. 16 (2004) 3155–3161.

[6] D.R. Cornejo, A. Medina-Boudri, H.R. Bertorello, J. Matutes-Aquino, J. Magn. Magn. Mater. 242 (2002) 194-196.

[7] P.P.Hankare, R.P. Patil, U.B. Sankpal, S.D. Jadhav, P.D. Lokhande, K.M. Jadhav, R. Sasikala, J. of Solid State Chemistry, 182,(2009) 3217.

[8] H.M.Widatallah, C. Johnson, F.Berry, M.Pekala, Solid State Commun.120 (2001)171.

[9] G.M.Argentina, P.D.Baba, IEEE Trans.Microwave Theor.Technol. 22(1974)652.

[10] J.S. Baijal, S. Phanjoubam, D.Kothari,C.Prakash, P.Kishan, Solid State Commun. 83 (1992) 679.

[11] Y.P. Fu, C.S. Hsu, Solid State Commun.134 (2005) 201.

[12] P.P. Hankare, R.P. Patil, U.B. Sankpal, S.D. Jadhav, I.S. Mulla, K.M. Jadhav, B.K. Chougule, J. Magn. Magn. Mater. 321 (2009)3270.

[13] R.P. Patil, B.V. Jadhav, P.P. Hankare, Results in Physics, 3 (2013) 214–218.

[14] P.P. Hankare, R.P.Patil, U.B.Sankpal, K.M.Garadkar, R.Sasikala,

A.K.Tripathi , I.S.Mulla J. of Magnetism and Magnetic Materials, 322 (2010) 2629–2633.

[15] R.P.Patil, P.P.Hankare, K.M.Garadkar, R.Sasikala, J. of Alloys and Compounds, 523 (2012) 66–71.

[16] R.P.Patil, B.V. Jadhav, P.P. Hankare, Results in Physics, 3(2013)330.

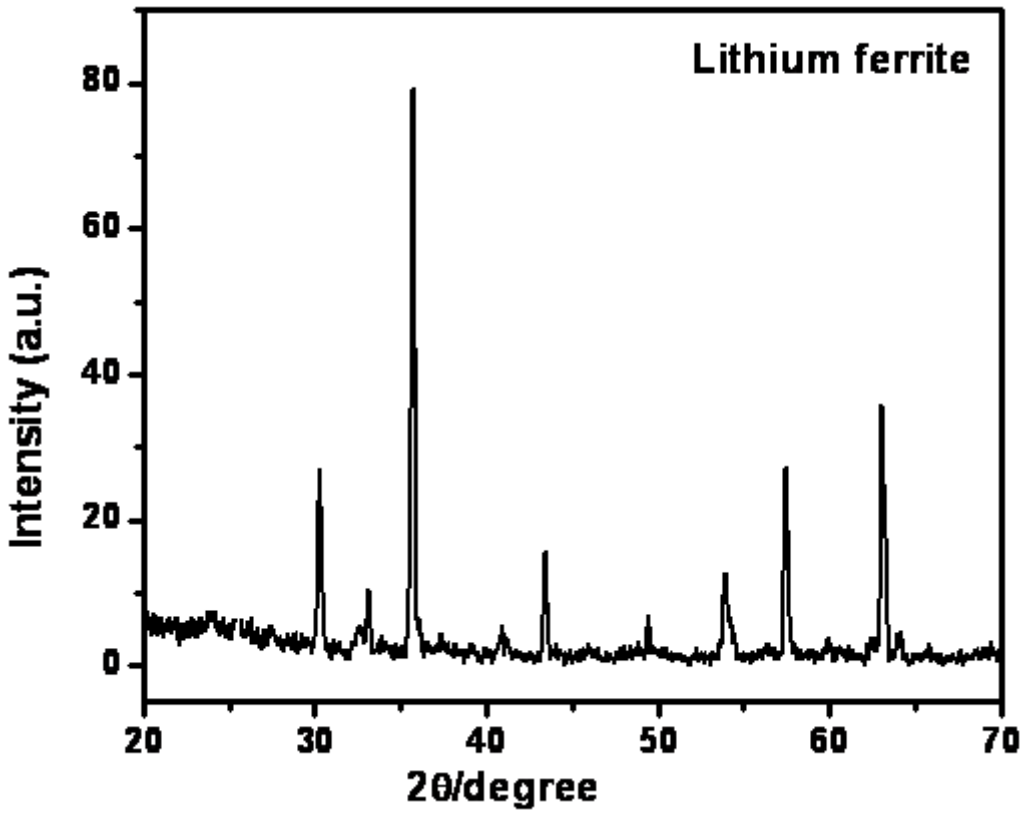

Fig.1. X-ray diffraction pattern of $Li_{0.5}Fe_{2.5}O_4$

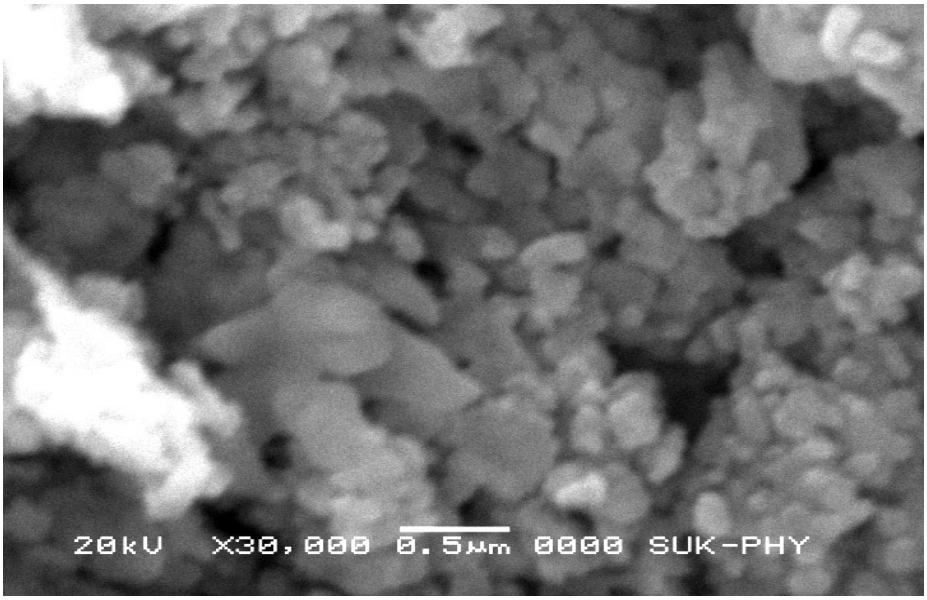

Fig.2.SEM Micrograph of $Li_{0.5}Fe_{2.5}O_4$

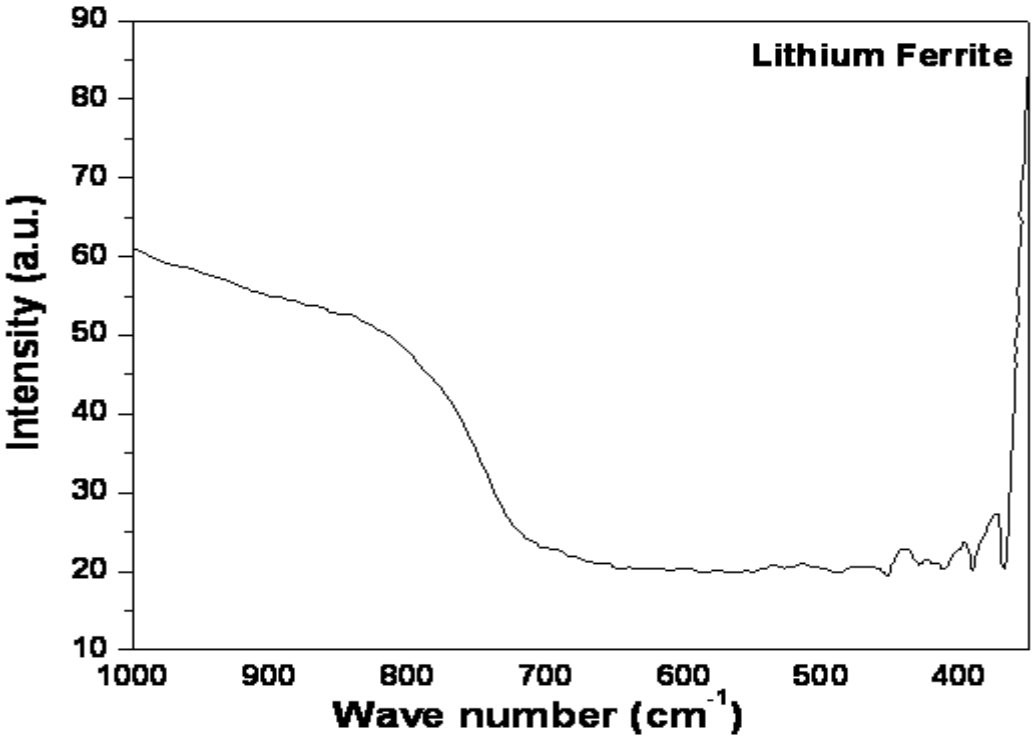

Fig.3. FT-IR spectrum of $Li_{0.5}Fe_{2.5}O_4$

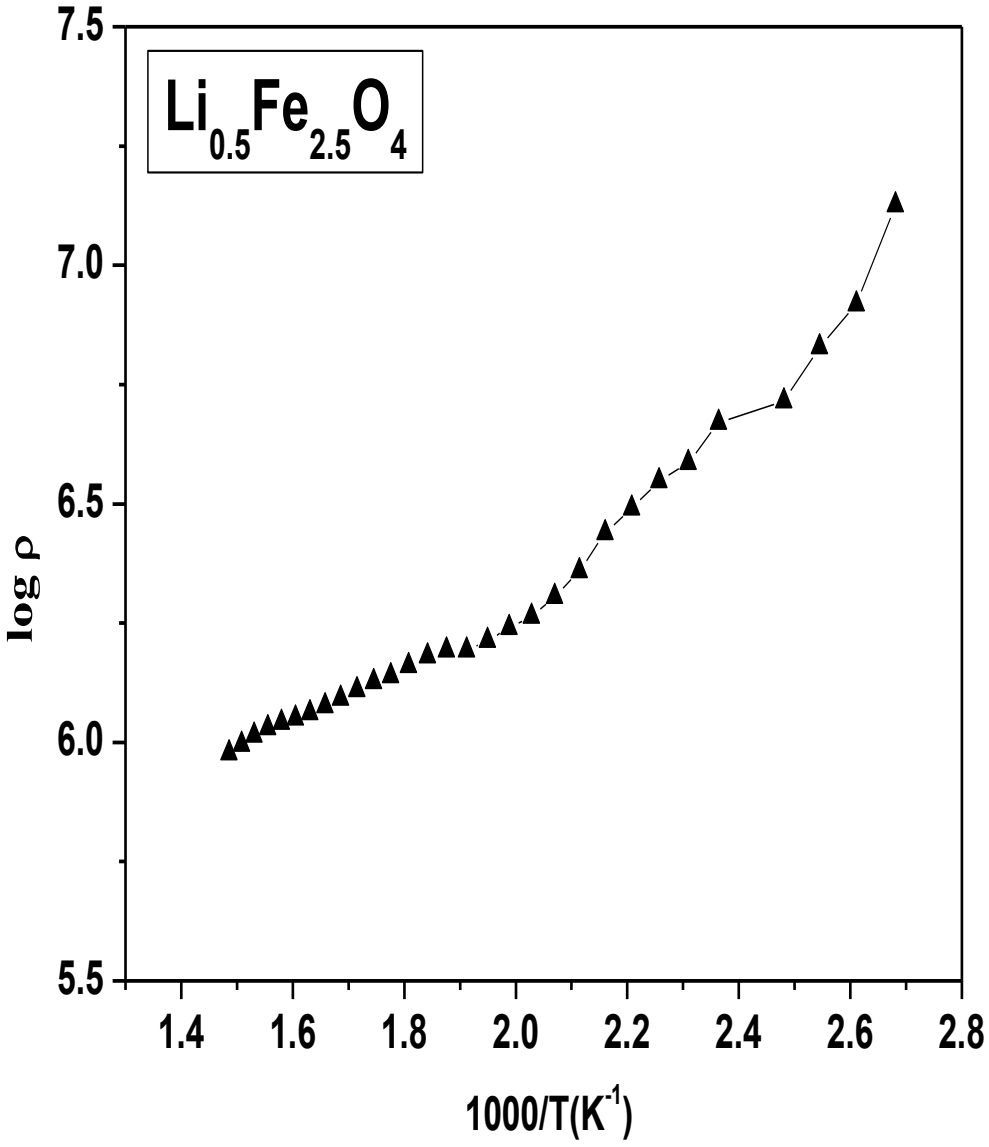

Fig. 4. Electrical resistivity study of $Li_{0.5}Fe_{2.5}O_4$

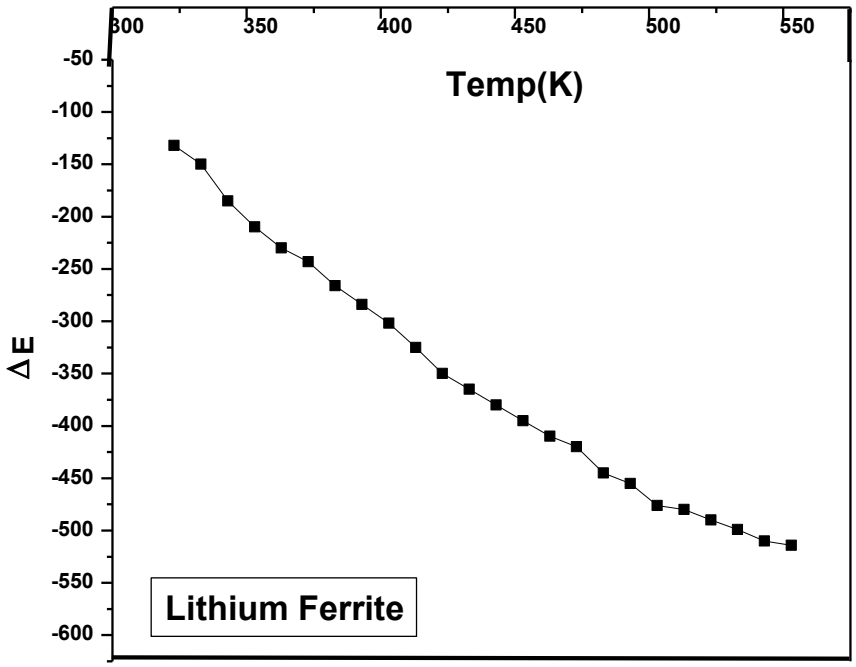

Fig.5. TEP study of $Li_{0.5}Fe_{2.5}O_4$

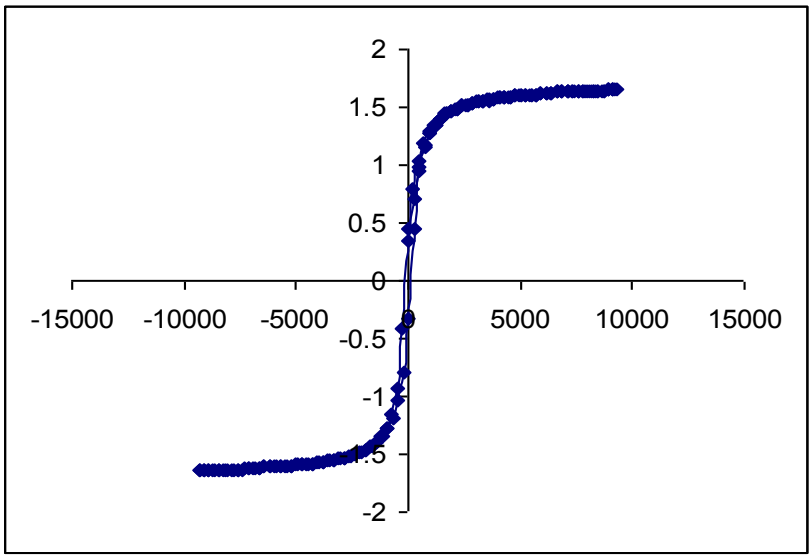

Fig.6. Vibrating sample magnetometer data (VSM) for $Li_{0.5}Fe_{2.5}O_4$

Table.1. Lattice constant, crystallite size & x-ray density for $Li_{0.5}Fe_{2.5}O_4$ sample

Composition	Lattice constant (a) Å	Crystallite size (t) nm	X-ray density(dx) gm/cm^3
$Li_{0.5}Fe_{2.5}O_4$	8.31	22.0	4.1