

Studies on structural and electrical properties of $\text{Li}_{0.5-0.5x}\text{Co}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ ($0 \leq x \leq 0.6$) spinel ferrite

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ABSTRACT

In the present work, nanocrystalline $\text{Li}_{0.5-0.5x}\text{Co}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ (for $x=0.0, 0.1, 0.2, 0.3, 0.4, 0.5$ and 0.6) ferrite systems were synthesized by solution combustion method. The Rietveld analysis of X-ray result confirms the formation of a single phase spinel cubic crystal structure of the ferrite sample. The lattice constant of the material increases from 8.33 \AA to 8.36 \AA with increasing cobalt content in lithium ferrite. The cation distribution study reveals that the Co–Li ferrite is in the mixed spinel structure of the composition. The DC electrical resistivity result confirms the semiconducting nature and the Curie temperature decreases with increase in Co^{2+} content. The dielectric constant, loss tangent and dielectric loss decrease with frequency and remain constant at higher frequencies are observed, showing the usual dielectric dispersion due to space charge polarization. The impedance spectroscopy analysis of samples reveals the grain interior contribution in the conduction process. The AC conductivity as a function of frequency verifies that the small polarons are responsible for conduction process.

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1. Introduction

The polycrystalline lithium ferrites have very good electric and dielectric properties which are associated with various factors such as the sample preparation methods, substitution of ions, sintering temperature, amount of substitution etc. [1–3]. Cobalt substituted lithium ferrites have been found to be very good substitutes for microwave device application due to their low costs, and low eddy current losses. Ferrites are also appropriate in many device applications due to the low electrical conductivity as compared to that of magnetic materials [4]. For this reason the electrical performance is one of the important parameters of the ferrites which give precise information about the conduction mechanism of the material. The electrical as well as dielectric properties of Li ferrites can be modified by substituting them with different metal ions for device applications [5]. The addition of small concentrations of Co^{2+} ions in lithium ferrites have been found to be relatively useful. The addition of Co^{2+} ions in lithium ferrites is very effective as it opposes the microwave power loss in a device. Gupta et al. [6] has synthesized lithium ferrite by citrate method; they got the lower values of permittivity and permeability in the microwave frequency region. We have reported on the synthesis of high purity, homogeneous and polycrystalline Cobalt substituted Li-ferrite by autocombustion method and

studied there IR absorption spectral analysis as well as physico-chemical properties. The cation distribution of $\text{Li}_{0.5-0.5x}\text{Co}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ ferrites system was reported earlier [7, 8]. Several studies have been reported on effect of additions of cobalt ions in lithium ferrites prepared by solution combustion method. However, only few reports in the literature are available on dielectric performance of cobalt substituted lithium ferrite [1,9]. In order to achieve a high degree of molecular mixing, chemical homogeneity and control of stoichiometry, we have used solution auto combustion method for the synthesis of cobalt substituted lithium ferrites samples. We have carried out Rietveld analysis of XRD results of cobalt substituted lithium ferrite samples. The aim of Rietveld analysis is (i) to characterize the samples in terms of microstructural parameters such as unit cell volume, lattice constant and oxygen position parameters etc., and (ii) to estimate the cations distribution among tetrahedral-A and octahedral-B sites in the spinel lattice. In the present study the electric and dielectric properties of cobalt substituted lithium ferrites are studied as a function of composition and frequency. The obtained results have been compared with those prepared by standard ceramic and precursor methods.

2. Experimental

Samples of $\text{Li}_{0.5-0.5x}\text{Co}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ system (for $x=0.0, 0.1, 0.2, 0.3, 0.4, 0.5$ and 0.6) were prepared by the solution combustion

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synthesis method. In this method calculation of stoichiometry is crucial. It is calculated on the basis of total oxidizing and reducing valencies of oxidizer (O) and fuel (F), which serves as a numerical coefficient so that the equivalence ratio, i.e. Φ_e (O/F), becomes unity and the heat released is at its maximum. A mixture of solution prepared with AR Grade $\text{LiNO}_3 \cdot 3\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ and $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in an appropriate proportion was taken in a beaker. The solution was stirred slowly on magnetic stirrer to obtain a homogeneous mixture and then stoichiometric solution of glycine was added into it to form the redox mixture for the combustion process. The resultant solution was then dehydrated slowly onto a hot plate with continuous stirring until the viscous gel was formed. On further heating, the temperature of the gel increased

and at a certain temperature auto-ignition of the black and fluffy gel took place with the evolution of gases. The resulting powder of all compositions was grinded for 30 min and sintered at the 800°C for 5 h with a heating rate $2^\circ/\text{min}$ in a programmable furnace. The calcined powder then uniaxially pressed into the pellets by using 2% PVA binder and by applying pressure of about 7 ton for 10 min. These pellets were finally sintered at 1100°C for 11 h to reduce porosity and increase density. The samples sintered at 1100°C have further used for studying structural and electrical properties.

The structural properties were studied by a Bruker D2-Phaser X-ray powder diffractometer using $\text{Cu-K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). X-ray diffraction (XRD) patterns were analyzed with the help of FullProf program by employing Rietveld refinement technique. The

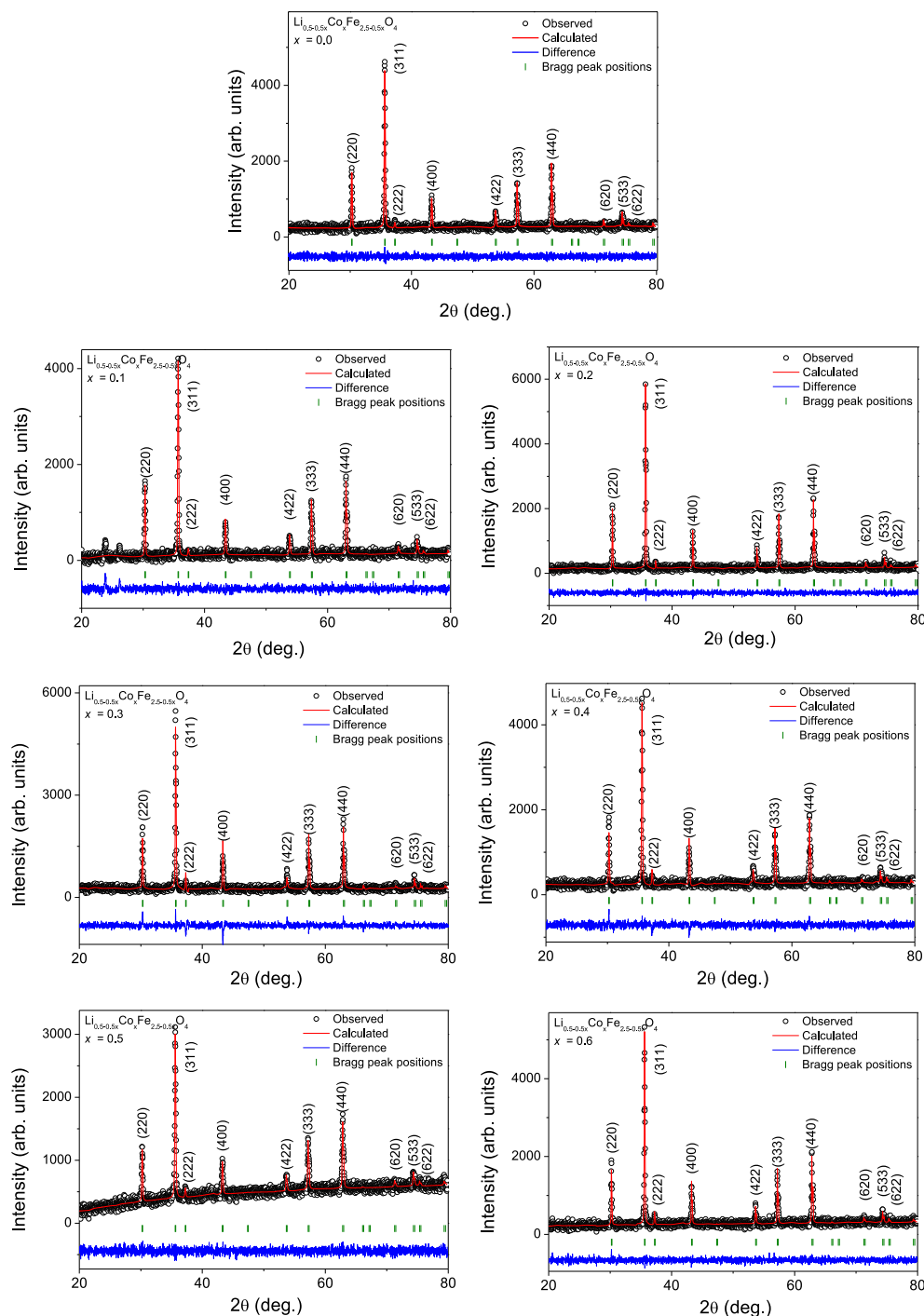


Fig. 1. Rietveld refined X-ray diffraction patterns of $\text{Li}_{0.5-0.5x}\text{Co}_x\text{Fe}_{2.5-0.5x}\text{O}_4$ samples.

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