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European Journal of Applied Engineering and
Scientific Research, 2012, 1 (4):173-178
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Synthesis and characterization of cobalt substituted Li-Ni-Zn nano ferrites by chemical route

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ABSTRACT

The cobalt substituted Li-Ni-Zn ferrites with the general formula $Li_{0.5}Ni_{0.15}Co_{0.1}Zn_{0.5}Fe_2O_4$ were prepared using auto combustion method. Structural characterization of the samples was carried out using X-ray powder diffraction technique. The lattice parameter of the sample was found to be 8.376\AA . The surface morphology of the ferrites was studied by Scanning Electron Microscopy (SEM) gives nanosized grains. Nanocrystalline nature of ferrites was also confirmed by Transmission Electron Microscopy (TEM). IR absorption spectra show two prominent bands characteristics of A and B site vibrations.

Keywords: Nanoferrite, X-ray diffraction, Infrared Spectroscopy, Microstructure

INTRODUCTION

Ferrite materials technology has now reached a very advanced stage, in which the design engineers control the properties to a large extent, to suit the particular device. The mixed valence ferrites have attracted large scientific interest in the recent years because of their outstanding magneto-transport properties [1]. The selection of ferrite material for specific applications depends mainly on the basic constituents of the composition as well as on the preparation conditions [2]. During the preparation of polycrystalline ferrites slightly reducing conditions, the divalent iron ions formed in the bulk of the material lead to high conductivity grains separated by ultra- thin insulating layers, so that the samples behave as inhomogeneous dielectric materials.

Amongst ferrites, Li and Li-substituted ferrites have achieved greater importance in applications because of their square hysteresis loop such as in memories applications and microwave components [3]. To improve the electrical and magnetic properties of Li-ferrites, divalent, trivalent and tetravalent ion substitutions were studied earlier [4]. Cobalt ferrite, with its high magneto crystalline anisotropy and remarkable stability, is one of the most promising candidates for biological application that require magnetic nanoparticles, ranging from recording media to photomagnetism [5]. Magnetic resonance imaging (MRI) and magnetic fluid hyperthermia (MFH) [6-8].

A recent literature survey on the preparation and characterization of various spinel nanocrystalline ferrites is discussed here in brief. Gul et al. [9], have found that as Zn concentration increases in $Co_{1-x}Zn_xFe_2O_4$; the iron ions migrate from A to B sites to accommodate the increased number of Zn^{2+} ions on the A sites. Bhavikatti et al. [10]

Cobalt is invariably doped in various ferrites for the rapid relaxation of the lattice. Thus we expect that, Co substitution in Li-Ni-Zn ferrites may improve their microstructural, morphological and other properties of ferrites. In the present work our prime aim is to achieve the successful synthesis of these ferrites by Pramanik method [11] and to study the possible effects of Co substitution on the microstructural aspects of Li-Ni-Zn ferrites.

Ferrite particles in nanoscale can be produced by soft chemical methods, such as co-precipitation, sol-gel, hydrothermal synthesis etc. The Pramanik method has been chosen in the present case as the conditions of synthesis in this method are highly controllable and further it provides homogeneous powder. Chemical synthesis routes play a crucial role in designing the final product and also they are better and less cumbersome for the production of fine grained mixed oxide products. The use of chemistry in the preparation of materials can avoid three major problems-diffusion, impurities and agglomeration. The reason being that the fine grained powders allow for shorter diffusion distances and improved homogeneity. The chemical precursor used can be easily refined to increase the purity, and careful control of solvent removal from the precursor will lead to the production of crushable agglomerates.

MATERIALS AND METHODS

The actual method of sample preparation consists of two steps. In the first, the stoichiometric amounts of AR Grade individual nitrates viz. nickel nitrate [Ni (NO₃)₂.6H₂O], zinc nitrate [Zn (NO₃)₂.6H₂O], cobalt nitrate [Co (NO₃)₂.6H₂O], lithium nitrate [LiNO₃] and iron nitrate [Fe (NO₃)₂.9H₂O] are mixed together with 10% aqueous solution of polyvinyl alcohol (PVA) and aqueous solution of sucrose (2 mole per metal ion).

The resulting solution is heated slowly at 200°C for 3h in air to form the viscous liquid. The evaporation is accompanied by evolution of brown fumes of nitrogen dioxide (NO₂) due to decomposition mixture of nitrates. This distributes the cations throughout the polymer structure so that precipitation is prevented during the evaporation process. In the second step is precursor is evaporated completely to produce the fluffy carbonaceous pyrolysed mass. The dry mass is calcined at 400°C for 6 hr. The calcined powder is granulated using PVA as a binder and were uniaxially pressed at a pressure of 5 ton/cm² for 5 min to produce pellet specimen having 10mm diameter and 3mm thickness. The specimen are finally sintered at 600°C for 6 hr in air [12] for densification.

Confirmation of single phase formation of the samples was carried out using Philips x-ray diffractometer (Philips Model PW 3710) operated at 40 KV and 30 mA ($\lambda=1.542\text{\AA}$). The 2θ angle was varied from 20° to 80°. The microstructure of the sample was recorded using scanning electron microscope (SEM Model Hitachi S3400N).

The nanoparticle morphologies were investigated by using transmission electron microscopy (TEM Model Philips "CM 200 super twin STEM"). The IR spectra of the sample were recorded using PERKIN EMLER IR Spectrometer (Spectrum 100) in KBr medium, in the wave number range 300 to 800 cm⁻¹.

RESULTS AND DISCUSSION

3.1 X-ray diffraction analysis:

The XRD patterns of the sample Li_{0.5}Ni_{0.15}Co_{0.1}Zn_{0.5}Fe₂O₄ shown in Fig. 1. The patterns indicate well-defined peaks of crystalline fcc phase which confirm spinel cubic structure of the sample. No any additional peaks corresponding to impurity phase were observed. The crystallite size (t) of the samples was calculated using Scherrer formula [13]. The particle size of a crystal, 'D' is calculated from the full width at half maximum of the XRD peaks at 2θ , using the Scherrer formula

$$D = 0.9 \lambda / \beta (\cos \theta)$$

Where λ , is the x-ray wave length and β is the FWHM in radians.

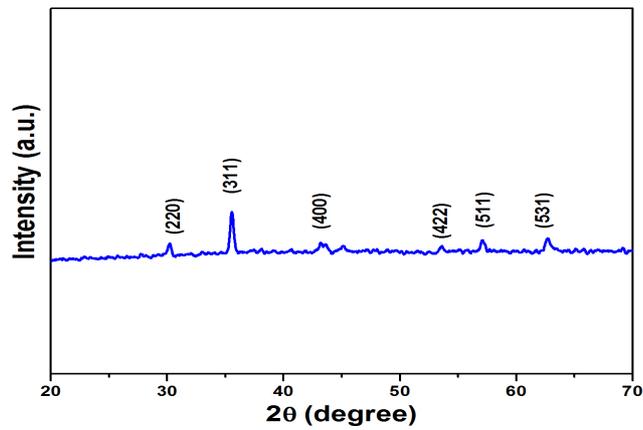


Fig. 1. X-ray diffraction pattern of $\text{Li}_{0.5}\text{Ni}_{0.15}\text{Co}_{0.1}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$

For cubic spinel structure the interplaner distance 'd', the lattice constant 'a' and the Miller indices (hkl) of reflecting planes are related by the equation

$$d = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

The data on lattice constant, crystallite size x-ray density 8.376 (\AA), 23 nm and 5.123 g/cm^3 .

3.2. Surface morphology by TEM and SEM



Fig. 2 Transmission Electron Microscopy of $\text{Li}_{0.5}\text{Ni}_{0.15}\text{Co}_{0.1}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$

The Transmission Electron Microscopy was used to record the size of the ferrite particles. Typical TEM images of the synthesized powder captured at different magnifications of the sample Fig 2, which confirm their nano crystalline nature.

Most of the particles appear spherical in shape however some elongated particles are also present. More agglomerated particles of low polydispersity in size than separated particles are present in the images. Agglomeration appears unavoidable due the absence of surfactant. The average particle size is found to be around 30 nm. The size of the particles as observed by TEM is consistent with the average particle size of 17 to 24 nm as estimated from XRD analysis.

The samples for TEM analysis were dispersed in propanol with the aid of ultrasound, and then applied to a copper grid, where they were allowed to dry and were later viewed on TEM. The selected area diffraction (SAD) patterns and bright field images for samples were also obtained. Fig. 3 shows the SAD patterns for the sample which one can clearly see the diffraction rings. This studies indicates the polycrystalline nature of the ferrite as well as the uniformity of particles in both morphology and size distribution [14, 15].

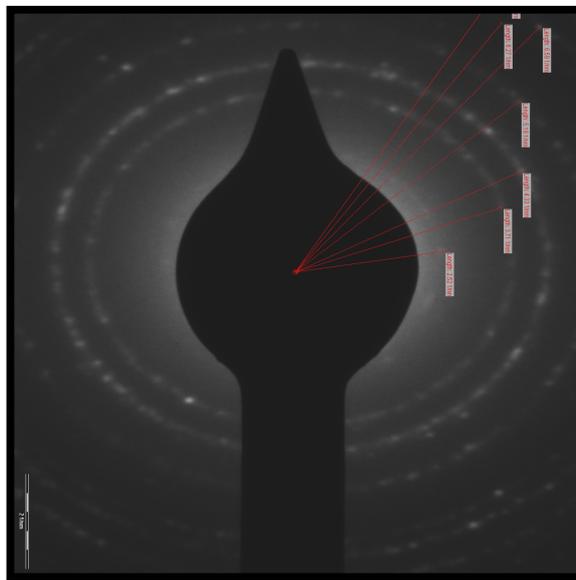


Fig 3 SAD patterns for the s $\text{Li}_{0.5}\text{Ni}_{0.15}\text{Co}_{0.1}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ample

Fig. 4 shows SEM micrographs of the samples. It is observed that the majority of the grains are spherical in shape. The Mechanical behavior and physical properties of materials are strongly influenced by their microstructure.

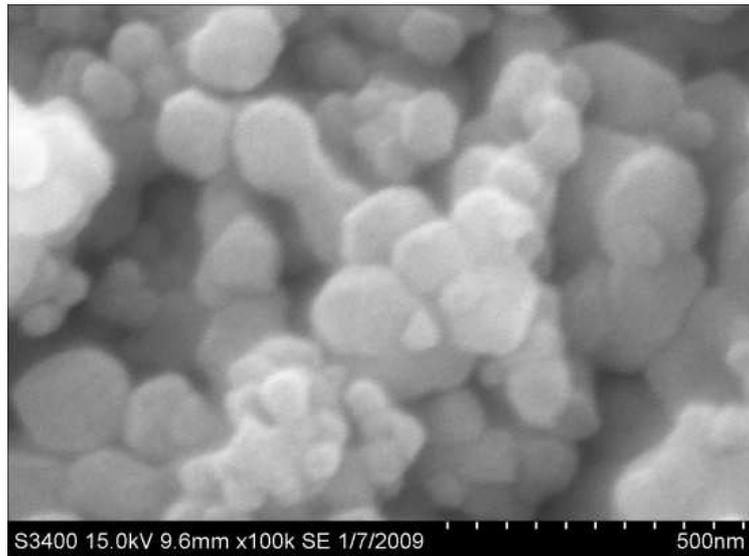


Fig.4.SEM micrographs of the samples

Thus microstructure studies are essential in order to understand the relationship between the processing parameter and the behavior of the materials used in practical applications. The average grain size is found to 103 nm.

3.3. FTIR Studies

Fig.5 shows the IR spectra of the prepared samples recorded in the range of 1000-300 cm^{-1} .

The spectrum elucidates the position of the ions in the crystal structure and their vibration modes, which represents the various ordering positions on the structural properties of the synthesized compound.

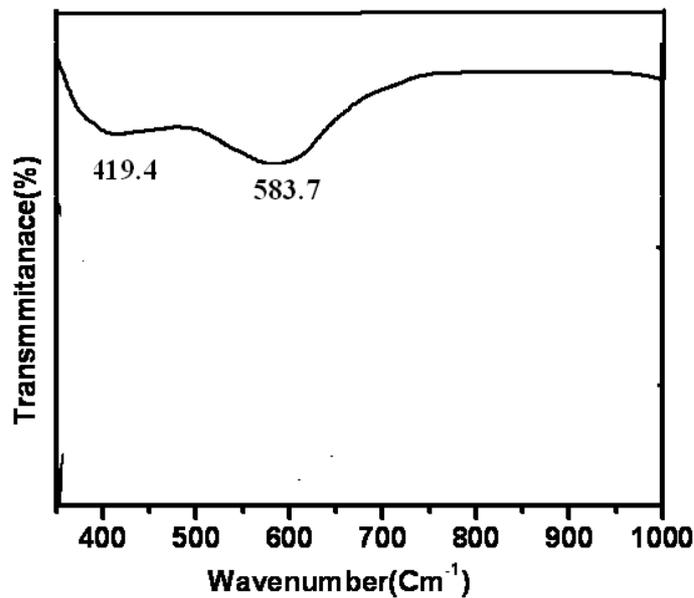


Fig 5 The FTIR spectra of sample

The absorption bands for the synthesized ferrite are in the expected range. It is well known fact that the normal and inverse cubic spinels have four absorption bands signifying (ν_1 , ν_2 , ν_3 , and ν_4). It is noticeable that the main absorption bands (ν_1 , and ν_2) have appeared in all series of the investigated ferrite system. These two bands are common in almost all spinel ferrites. They are located around 600 and 400 cm^{-1} , respectively. In the present work, the absorption band ν_1 around 400 cm^{-1} is attributed to vibrations of octahedral complexes whereas the absorption band ν_2 around 600 cm^{-1} is attributed to those of tetrahedral complexes. The value of ν_2 are higher than those of ν_1 indicating that the normal mode of vibrations of the tetrahedral complexes is higher than that of the corresponding octahedral site. The variation in the band positions is due to the difference in the Fe^{3+} - O^{2-} distances for the octahedral and tetrahedral complexes [11].

CONCLUSION

The $\text{Li}_{0.5}\text{Ni}_{0.15}\text{Co}_{0.1}\text{Zn}_{0.5}\text{Fe}_2\text{O}_4$ ferrite was prepared by Pramanik method. The formation of ferrite phase was confirmed by XRD studies. The particle size obtained from XRD data is found to be around 23 nm which in close agreement with the average particle size of 30 nm obtained from TEM studies. The main absorption bands of spinel ferrite have appeared through IR absorption spectra recorded in the range of 1000-300 cm^{-1} . The analysis of IR spectra indicates the presence of splitting in the absorption band owing to change in cobalt concentration.

Acknowledgements

The authors are thankful to UGC WRO, Pune for financial assistance, IIT, Mumbai and USIC, Shivaji University, Kolhapur for extending experimental facilities.

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