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Synthesis and Characterization of Spray Deposited Lithium Ferrite Thin Film

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ABSTRACT

Nanocrystalline, homogeneous spinel $Li_{0.5}Fe_{2.5}O_4$ thin film was deposited on glass substrate by using spray pyrolysis of metal nitrate aqueous solution. Respective metal nitrates (lithium nitrate) and iron nitrate were used as precursors. Phase composition, crystal structure and morphology of the obtained films were studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and energy dispersive X-ray microanalysis (EDAX). The analysis of XRD data confirms polycrystalline nature and single phase cubic spinel structure of the film. The surface morphology was studied using scanning electron microscopy (SEM) which reveals spherical morphology of the film. The thickness of deposited film was obtained using surface profiler which is found to be below 500 nm, but crystallite size was under 30 nm. The optical transmittance and reflectance measurements were recorded using UV-Vis spectrophotometer. The optical studies reveal that the transition is direct band gap energy.

Keywords: Lithium ferrite, Spray pyrolysis, XRD, SEM, UV-Vis.

INTRODUCTION

Spinel ferrite is an important class of magnetic material with large number of application in various fields [1, 2]. It can be represented by the formula MFe_2O_4 where M stands for metal ions like cobalt, nickel, zinc, copper, manganese etc. Spinel ferrite possesses both electrical and magnetic properties. The high electrical resistivity, low eddy current and dielectric loss, high saturation magnetization, high permeability, high Curie temperature etc. are the remarkable properties of spinel ferrites [3]. These properties are sensitive to method of preparation, nature and type of dopant and distribution of cations at available interstitial sites. In spinel ferrites two interstitial sites are available namely tetrahedral A site and octahedral B site. Spinel ferrites can be studied in different forms viz. bulk, nano and thin film form. Recently, nanocrystalline thin films have become more important from the point of view of their applications.

In the family of spinel ferrite, lithium ferrite is a stable, insulating ferrimagnet that has applications in microwave electronics [4]. It is a close relative of magnetite, which can be represented as [Fe^{3+}] {$Fe^{3+}Fe^{2+}$} O_4, where the square bracket represents the tetrahedral site and the curly bracket represents the octahedral site. Lithium is monovalent, so for $Li_{0.5}Fe_{2.5}O_4$, all the Fe must be in the 3^+ valence state, giving [Fe^{3+}] {$Fe^{3+}_{1.5}$ $Li^{+}_{0.5}$} O_4, assuming the inverse spinel structure. Lithium ferrites are superior to other spinel ferrites at the microwave frequencies due to their characteristic low insertion losses, high spin wave line widths and temperature stabilities of saturation magnetization [5]. Lithium ferrites in thin film configuration are of great interest for emerging microwave applications owing to their high resistivity, low eddy current losses, excellent thermal and chemical stability, and suitable dielectric properties [6, 7].

Nanocrystalline ferrite thin films have applications in high-density magneto-optic recording devices, colour imaging, bioprocessing, ferrofluids and magnetic refrigeration [4, 8, 9]. Apart from these applications, miniaturization in the field of microwave devices employing ferrite components compatible with monolithic microwave integrated circuits (MMICs) is one of the main area for the future growth of ferrite thin film technology.

There are various thin film deposition methods such as pulsed laser deposition [10], spin coating [11], RF sputtering [12], spray pyrolysis [13], chemical bath deposition[14] etc have been employed to deposit nano structured spinel ferrite thin films. A spray pyrolysis deposition (SPD) with a simple apparatus is a versatile technique for producing various thin film materials of a wide range of the composition in air at a relatively low temperature and a high deposition rate. In the present work, lithium ferrite thin films have been prepared by using spray pyrolysis technique. The structural, morphological and optical properties of lithium ferrite thin films have been investigated using standard techniques and results are presented in this paper.

METHODOLOGY

The raw materials of AR grade as lithium nitrate and ferric nitrate [$Fe(NO_3)_3.9H_2O$] were used for deposition of lithium ferrite thin films without further purification. Lithium ferrite thin films were deposited on glass substrate by spray pyrolysis deposition technique. The stoichiometric amount of respective metal nitrates was dissolved in distil and mixed together for spray. Lithium ferrite thin films were prepared by spraying the solution onto previously cleaned glass substrate. Glass substrates mounted on a holder were placed on the surface of a hot plate. A temperature controller was used to hold the preset temperature of 648 K with an accuracy of ± 50 C through a chromel-alumel thermocouple connected to the glass substrate. A prepared solution was atomized in air via pneumatic spray system under an air pressure of 2 kg/cm^2. The atomized droplets were transformed onto the heated glass substrate for 0.5 sec intermittently. The substrate temperature could be reduced under the effect of spray and requires several seconds to recover the preset temperature. The solution spray rate and the distance between nozzle and substrate were kept constant at 0.125 ml/s and 30 cm respectively. The films were annealed at a temperature of 550 °C for 4 hours. The deposited films were characterized by X-ray diffraction method (XRD), a scanning electron microscopy (SEM) and UV-Vis spectrometer to study the structural, morphological and optical aspects of lithium ferrite thin film under investigation.

RESULT AND DISCUSSION

Structural properties

Fig. 1 depicts the X-ray diffraction (XRD) pattern of lithium ferrite thin film. The XRD pattern revealed good crystallinity because of annealing of the film. The lithium ferrite film is oriented along (311) plane. The other planes viz. (210), (310), (311), (421), (422) (511) (440) and (441) also exist in the XRD film which is sharp and intense. The XRD patterns matches well with the characteristics reflections of cubic spinel structure. The analysis of XRD pattern revealed the formation of single phase cubic spinel structured lithium ferrite thin film. The interplanar spacing 'd' values of the film are in good agreement with the standard 'd' value of lithium ferrite. Using the'd' values and corresponding Miller indices, the value of lattice constant of the lithium ferrite thin film was evaluated. The value of lattice constant 'a' is given in Table 1 which is in good agreement with the reported value. The value of 'a' was used to obtain the unit cell volume, X-ray density, bulk density, porosity and same are given in Table 1.

Table 1 Lattice parameter (a), Unit cell volume (V), Crystallite size (t), X-ray density (dx), Bulk density (dB), and Porosity (P) of lithium ferrite thin film

a (Å)	V (cm^3)	d_x (gm/cm^3)	d_B (gm/cm^3)	P (%)	t (nm)
8.3334	578.09	5.3802	3.638	32.3847	18.3

The crystallite size was determined from full width of half maximum (FWHM) of the most intense (310) peak, which was obtained by slow step scanning or on that peak at 0.20 per minutes based on Scherrer's formula Debye-Scherrer formula [15] for crystallite size determination is given by

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \quad \ldots 1$$

where, D is the crystallite size, λ is the wavelength of X-ray, β is the full width at half maximum (FWHM) after correcting the instrument peak broadening (β expressed in radians), θ is the Bragg's angle.

The crystallite size is of the order of 18 nano meter, which indicates the nano-crystalline nature of the film. The value of crystallite size is given in Table 1

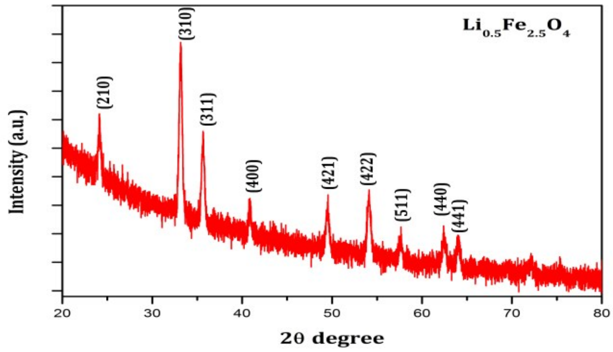

Figure 1: XRD pattern of lithium ferrite thin film

Morphological study

Scanning electron micrograph of lithium ferrite thin film on glass substrate is shown in Fig. 2. The micrograph shows agglomerated particles grown randomly on the substrate. The lithium ferrite thin film has covered the complete substrate surface with some random over growth of particles as evidenced by the micrographs. The grain size measured from SEM is found to be of the order of 14.58 nm, which is in good agreement with the particle size obtained from X-ray diffraction.

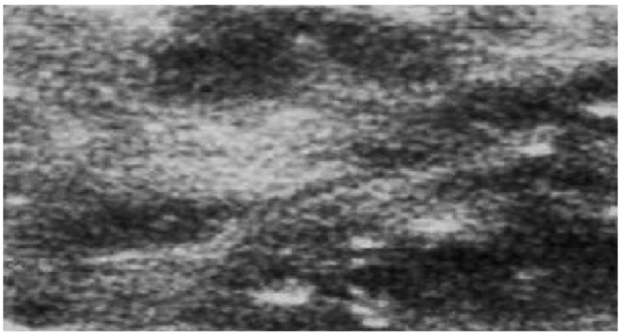

Figure 2: SEM image of lithium ferrite thin film

The chemical composition analysis was carried out on $Li_{0.5}Fe_{2.5}O_4$ thin film sample by energy dispersive X-ray (EDX) analysis. The EDX spectra, confirms the presence of elements as Li, Fe and O in stoichiometric proportion.

Optical study

Optical studies of lithium ferrite thin film were carried out to investigate the band gap energy of the film. The optical absorbance of annealed lithium ferrite thin film was studied in the range 400 - 1000 nm, using UV-Vis spectrophotometer, which has been further used for the energy band gap calculation. The optical characterization of the deposited thin films was used to calculate the band gap by employing both indirect and direct band gap measurement methods. The optical band gap Eg for all the films were estimated from the absorption coefficient (□) using the Tauc relation [16] which is given by

$$\alpha h\upsilon = A\ (h\upsilon - Eg)n$$

where, A is a constant depends on the transition probability, hv is the energy of incident photon and n is an index that characterizes the optical absorption process. It is theoretically equal to 2, 1/2, 3 and 3/2 for allowed indirect, allowed direct, forbidden indirect and forbidden direct electronic transitions respectively depending on the nature of the electronic transition responsible for the reflection. The obtained band gap value for lithium ferrite thin film is 2.37 eV.

CONCLUSION

Nanocrystalline thin film of lithium ferrite has been successively deposited on glass substrate using spray pyrolysis technique. X-Ray diffraction analysis proves that, the film possess simple cubic spinel structure. The morphological studies carried out by SEM analysis shows agglomerated particles grown randomly on the substrate. The EDX analysis confirms the presence of all the elements (Li, Fe and O) in stoichiometric proportions. The optical studies confirmed the band gap value for lithium ferrite thin film is about 2.37 eV.

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