Synthesis & Morphological Characterization of Zn-Al Substituted Lithium Cubic Ferrite.

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Abstract:

Lithium ferrites substituted with Zn, Al having general chemical formula $Li_{0.5(1-x)}Zn_xFe_{2.5-y}Al_{y-0.5x}O_4$ have been prepared with x=0.1 ; y= 0.2,0.4,0.6,0.8 by sol gel Method. X-Ray Diffraction analysis confirms the spinel structure of sample. The lattice parameter, X-ray density, bulk density, grain size, porosity etc. have been determined for each composition. The Fourier Transform Infra -Red (FT-IR) spectra analysis in the range $400 – 4000$ cm^{-1} show the characteristic features of synthesized ferrite powder.

Key Words: Sol Gel Method, XRD, FT-IR etc.

1.   Introduction:

The derivatives formed due to substitution in Lithium ferrite are substantial materials for microwave applications because of its low cost excellent temperature performance, hysteresis loops [1]. Several series of compositions of lithium ferrites covering a wide range of properties have become available commercially. Lithium Ferrite one of the most versatile magnetic material for multilayer chip inductor (MLCI) applications and surface mount devices (SMDs) due to their high electrical resistivity , low sintering temperature and high permeability [2]. There are many reports in the literature on the effect of non-magnetic and magnetic cation substitution on various properties of lithium ferrite [3-6]. In present communication structural properties like lattice constant, X- ray density, bulk density, porosity, grain size etc. have been reported on the basis of X- ray diffraction (XRD). Infra red spectroscopy used to determine the local symmetry in crystalline [7] also ordering phenomenon in spinel [8-9]. As such it is necessary to study the effect of Zn, Al substitution on infra red spectra of lithium ferrite in present work.

2.   Experimental:

The Ferrite powders were synthesized through nitrate–citrate sol gel method to achieve homogeneous mixing of chemical constituents. Analytical grade of lithium nitrate (LiNO3), zinc nitrate (Zn(NO3)2.6H2O), aluminum nitrate [Al(NO3)3.9H2O], ferric nitrate (Fe(NO3)3.9H2O) were used to prepare Li0.5(1-x)ZnxFe2.5-yAly-0.5xO4 ferrite with x=0.1 ; y= 0.2,0.4,0.6,0.8 compositions. Metal nitrate dissolved in deionized water with continuous stirring till clear solution obtained. Metal nitrate solution standardized, mixed with citric acid (C6H8O7.H2O) solution in 1:1 stichometric molar ratio. The PH of solution was adjusted to 7 using ammonia. The solution was first heated at 80 0C to convert sol into desired gel. The gel, then was

formed, incinerated at 500 0C to form a fluffy ash powder. The as-burnt powder sintered at 800 0C for 4 hr. The ferrite powders were characterized for X- ray diffraction (Bruker AXS D8 Advance), Fourier transform infra-red spectroscopy (FT-IR) (Thermo Nicolet Avatar 370).

3. Results and Discussion:

3.1 X-Ray Diffraction: X-ray diffraction (XRD) pattern of each composition shown in fig. 1, corresponds to well defined FCC phase, confirm the spinel structure. The Lattice Parameter calculated considering (311) plane as standard [10] by using the following equation:

$$a = d (h2+k2+l2) \, \tfrac{1}{2}$$

It can be observed from fig. 2 that the unit cell parameter decreases with increasing y, obeying the Vigard's Law [11]. The linear decrease in lattice parameter is due to the replacement of larger Fe^{+3} (0.64 Å) by smaller Al^{+3} ions with ionic radius 0.51 Å. The peaks are also shifted towards higher 2θ with substitution. The Bulk Density (d) of a specimen in pallet form determined by precise values of weight measured through electronic balance machine and volume measured by digital vernier caliper. The X-ray density (dx) for different compositions was calculated using the relation [12]:

$$dx = \frac{ZM}{Na^3}$$

Where Z is the Number of molecules per unit cell (Z=8) of spinel lattice, M is molecular weight of the ferrite sample, N is the Avogadro's number and 'a' is the lattice constant of the ferrite. The X-ray density and bulk density is found to decrease with increase of y. The porosity (P) was calculated using the relation [12]:

$$P = \left(1 - \frac{d}{d_x}\right) \times 100\%$$

The effect of composition 'y' on the porosity is shown in the fig. 3. It is observed that the porosity (%P) increases with 'y' composition. The particle size was calculated using the well known Debye- Scherer formula [10]:

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

Where D is particle size or grain size, λ is the wavelength of X-ray radiation, θ is the Bragg's angle, β is the Full Width Half Maxima (FWHM). The data for average particle size calculated for each sample which is in the range 20 - 23 nm. The values of lattice parameter, X-ray density, bulk density, porosity, grain size etc are tabulated in Table 1.

3.2 IR Spectral Analysis: The room temperature IR spectra recorded in the range 400 – 4000 cm -1 are shown in fig.4. The spectra show two main absorption bands below 1000 cm-1 as a common feature of all spinel ferrites. The high frequency band ν1 in the range 593-600 cm -1 while the low frequency band ν2 in the range 471-485 cm-1 [13]. The highest one ν1 corresponds to intrinsic stretching vibrations of the metal at tetrahedral site (Td) M tetra ↔ O whereas the lowest ν2 band correspond to octahedral metal stretching (Oh) M octa ↔ O [14]. The details of absorption frequency for different compositions are given in Table 2. According to Puri and Varshney [15] Zn^{+2} shows a strong preference to tetrahedral A- site and Li^{1+}, Al^{3+} shows a strong preference to octahedral B-site [16]. Due to replacement of Li^{1+}, Al^{3+} ions at octahedral site in the ferrite lattice affects the $Fe^{3+}-O^{2-}$ stretching vibration. This may be the reason for the observed change in ν1 band

positions. The presence of absorbed water is featured by the band in range 3430-3440 cm-1 and 1620- 1630 cm-1 which is assigned to O-H stretching and H-O-H bending modes of vibration [17-18]. This band attributes to the presence of water crystallization in the sample. It was reported earlier that the presence of water plays an important role in the formation of ferrites and their stabilization [19]. The band at 2330-2360 cm-1 corresponds to traces of adsorbed or atmospheric CO_2 [20].

4.Conclusion:

Increasing Al concentration leads to decrease in Lattice Parameter, X-Ray Density, Porosity and Grain Size. X- Ray Diffraction Analysis data indicates that the unit cell volume decreases with Al content. The IR spectra at room temperature of investigated samples showed a shifting in the band position v1 to higher frequency with an increasing Al content. As3

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Table 1 : Lattice Parameter, X-Ray Density, Bulk Density, Porosity (% P), Grain Size (D) of $Li_{0.5(1-x)}Zn_xFe_{2.5-y}Al_{y-0.5x}O_4$.

Comp	Absorption (cm-1)	
y	v1	v2
0.2	593	471
0.4	594	474
0.6	596	480
0.8	600	485

Table 2: IR Absorption Band of $Li_{0.5(1-x)}Zn_xFe_{2.5-y}Al_{y-0.5x}O_4$.

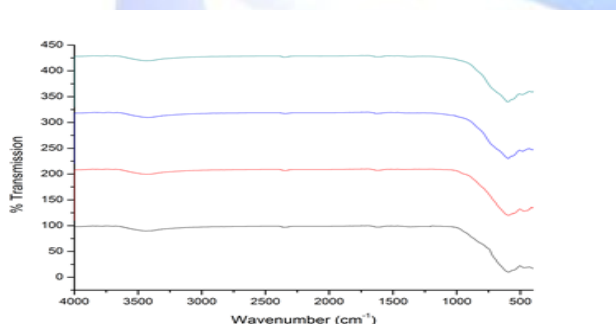| Comp Y	Lattice Parameter	Cell Volume	X- Ray Density d_x (g/cm3)	Bulk Density	Porosity (%P)	Grain Size D nm
0.2	8.2849	568.67	4.813	4.118	14.44	21.14
0.4	8.2674	565.08	4.708	4.102	12.87	22.89
0.6	8.2229	556	4.647	4.116	11.42	20.39
0.8	8.2592	563.4	4.451	3.981	10.56	22.71

Fig. 4: IR Absorption Spectra of $Li_{0.5(1-x)}Zn_xFe_{2.5-y}Al_{y-0.5x}O_4$.
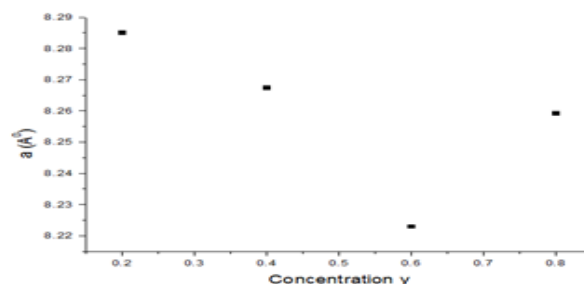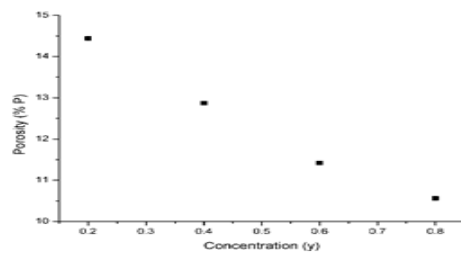

Fig. 2: Lattice Parameter a as a function of compositional parameter 'y'.
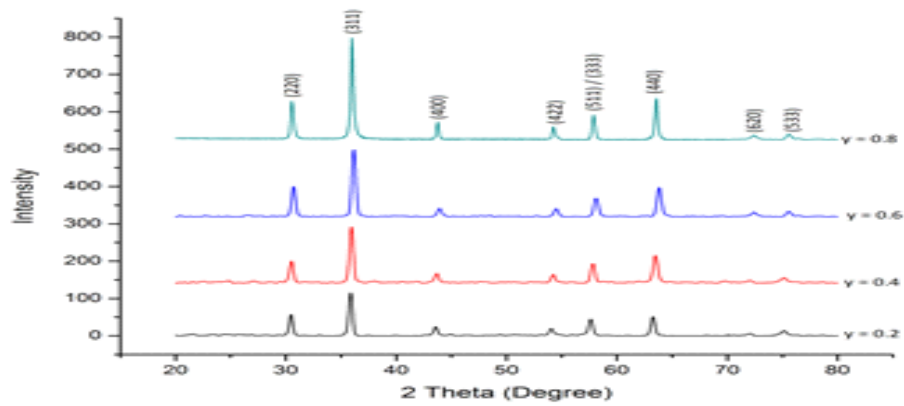
Fig 3: The effect of 'y' concentration on the variation of

Fig. 1: X- ray diffraction pattern of $Li_{0.5(1-x)}Zn_xFe_{2.5-y}Al_{y-0.5x}O_4$.