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Synthesis and Studies of Titanium Doped Lithium Zinc Ferrites

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Titanium doped Lithium Zinc ferrites were synthesized by Combustion synthesis method using Polyethylene glycol of molecular weight 6000 as fuel. XRD powder patterns indicate single phase cubic spinel structure formation for all the compositions. The nano size of the particles were obtained. The Variation of lattice constant with the concentration of Titanium and The variation of X-ray density and (bulk) with concentration of Titanium was studied.

1. Introduction

During the past few decades, new developments in the use of material science in advanced technological applications have attracted considerable attention. In addition to the discovery of superconductors, the use of heat resistant tiles in the space shuttle, components in high temperature engine, electromagnetic interference suppression as well as radio frequency interference suppression of noise. Inelectronic devices, ferrite cores etc. has generated considerable interest in these fields. The increasing use of materials in advanced technological applications has resulted in a demand for materials, suitable for the specific application. Ferrites represent an important category of materials which is largely used due to their numerous practical applications like magnetic components, phase shifters, microwave devices etc. To most people ferrites are completely unknown, yet they touch the lives of everyone, every day and contribute significantly to improve their Lifestyle. Lithium and substituted lithium ferrites is among them, gaming Importance because of their interesting properties and technological importance.

Lithium and substituted lithium ferrites form an important class of ferrites which have been extensively studied because of their beneficial properties suitable for various applications.

Generally, ferrites are prepared by the ceramic process involving high temperature solid state reactions between the constituent oxides/ carbonates. Several workers used to lower the sintering temperature as it is desirable to suppress lithium and oxygen loss during firing[18-21]. J.H. Hsu et a1[22]and Z. Yue et a1 [23]found that additives enhance densification;

however, Xiwei Qi et a1[24], Simonet and Hermosin[20], Van and Hook[25]reported that the additives form glassy phase at grain boundaries which have detrimental effect on various properties. Therefore, low temperature sintered ferrites without an additive has turned out to be a hot topic of research. This has been found to be achieved by using wet chemical method of synthesis which gives active ultrafine powders. Among the wet chemical methods, the citrate precursor method is gaining much importance as it is simple, economic and produces good results [26-29].

Cannas et a [30]studied the citrate precursor synthesis method and found that it is very sensitive to certain parameters involved in the synthesis process. It can produce nanoparticles, which in the last few years has been reported to display enhanced properties. B.S. Randhawa et al made investigation on ferrite nanoparticles observing that they are formed at low temperature and in short time. Moreover, ferrites obtained by the wet chemical method have probability of lesser defect due to impurities, as ball milling is avoided [31]. This has great beneficial Influence on the magnetic properties. A.K. Singh et a1[32]studied the dielectric properties of Mn substituted Ni-Zn ferrites prepared by citrate precursor method and observed lower value of dielectric constant and dielectric loss as compared to those obtained in ferrites prepared by ceramic method.

The chemical method being very sensitive to the various parameters involved, extra care is needed while synthesis and if carefully controlled, it can give extraordinary good properties.

2. SYNTHESIS TECHNIQUES:

Titanium doped Lithium Zinc ferrite having general formula $Li_{0.5(0.4+x)}Zn_{0.6}Ti_xFe_{(2.2-1.5x)}O_4$ where x = 0.1, 0.15, 0.2 and 0.25 were prepared by Combustion synthesis method. The precursors Li2CO3, ZnO, TiO2 and Fe2O3 all AR grade are taken in stoichiometric ratio. The amount of precursors taken for the synthesis of 10gm of the samples were listed in table 2.1. These precursors are well ground in agate mortar and pestle for 1 hr. Polyethylene Glycol (PEG) of molecular weight 6000 which acts as fuel for Combustion synthesis is mixed with the precursor mixture in the precursor to PEG ratio 1:2 in weight. This mixture was further ground for 2 hr to get fine powder. Precursor and PEG mixture was taken in to Silica crucible and heated at the rate of 3°C per minute up to 450°C in High Temperature Furnace and kept at 450°C for 1hr.

The PEG ignites on heating ~420°C, thus, undergoing combustion resulting in formation of fine nano-particle product. Apparently, nucleation of the LiZnTi ferrite occurs as a result of the heat generated due to high exothermicity of the decomposition of PEG in the precursor. Further decomposition of the precursor is self-sustained resulting in complete decomposition giving LiZnTi ferrites product.

The samples $Li_{0.5(0.4+x)}Zn_{0.6}Ti_xFe_{(2.2-1.5x)}O_4$ with x = 0.1, 0.15, 0.2 and 0.25 were labelled as LZTF1, LZTF15, LZTF20 and LZTF25 respectively.

X-ray diffraction (XRD)

The final ferrite product has been characterized by XRD and the physical properties of these samples have been studied.

The crystal-lite size can also be calculated using the angle in the Debye

Scherrer equation given by,

$$D_{hkl} = \frac{0.89\,\lambda}{\beta\cos\theta}$$

Where λ is the incident wavelength of CuKα radiation of XRD, is the full- width at half-maximum and θ is the diffraction angle.

Another important physical parameter of spinel ferrite, is the theoretical or X-ray density which is calculated using the value of lattice parameter in the formula,

$$d_{xrd} = \frac{8M}{Na^3}$$

where 'M' is the molecular weight of the sample, N is the Avogadro's no. $=6.023x10^{23}$ unit and 'a' is the lattice constant.

Thus the use of X-ray technique helps in phase identification, determination of lattice parameter, crystallite size, densification etc.

Density and porosity

In ceramics high density usually near theoretical density and fine grain size are desirable. However, it is not an easy task and there is always a slight deviationin the values obtained from experiment.

For the density measurement pellets of all LiZnTi ferrite samples of 10mm diameter are made using Hydraulic press by applying pressure of 6-7 ton and these pellets were sintered at 500°C for densification.

3. RESULTS AND DISCUSSION: .

. Data obtained from analytical methods and the instrumental techniques used 'in the present investigations are presented below. Interpretations from these results are also discussed.

3.1 X-ray diffraction analysis:

X-ray diffraction studies were carried out to examine the structure as well as to determine interplanar distances and the lattice constants. The samples of LiZnTi ferrite prepared were analyzed for its crystal structure by powder X-ray diffraction method using reflection angles in the range from 20^0 to 80^0 .XRD powder patterns for all the samples, indicate single phase cubic spinel structure formation for all the compositions of $Li_{0.5(0.4+x)}Zn_{0.6}Ti_xFe_{(2.2-1.5x)}O_4$ where x = 0.1, 0.15, 0.2 and 0.25. X-ray spectra of these samples are given in Fig. 3.1

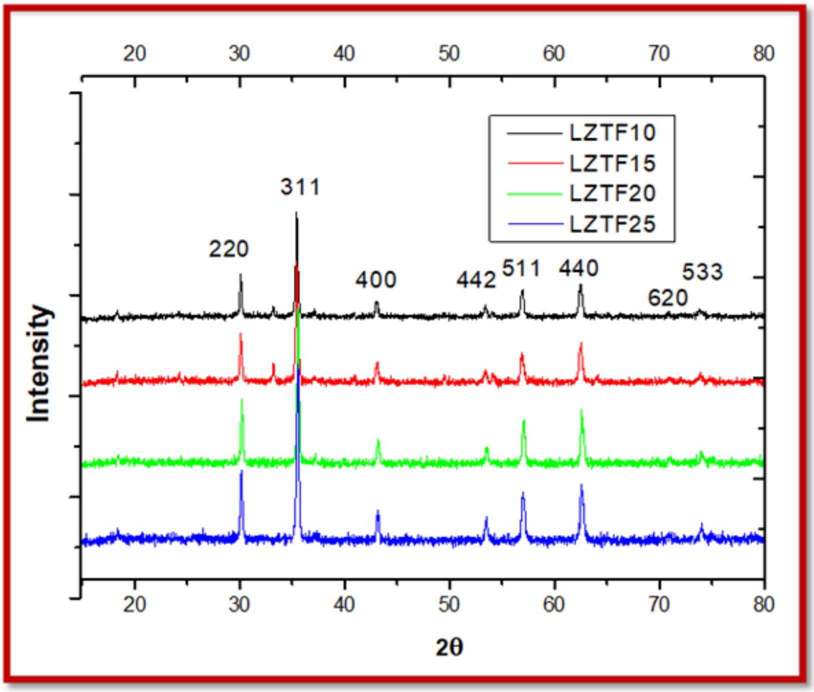

Fig. 3.1. X-ray diffraction patterns of $Li_{0.5(0.4+x)}Zn_{0.6}Ti_xFe_{(2.2-1.5x)}O_4$ samples.

Lattice Constant 'a' from X-ray diffraction data:

The values of lattice constants 'a' are calculated by using Scherer formula.The LiZnTiferrite samples show an increase in the lattice constant value as the concentration (x) of Titanium was increased.

Table 3.1. Calculated lattice constant for LiZnTi ferrites.

Samples	Lattice parameter (a) nm
LZTF10	0.8239
LZTF15	0.8373
LZTF20	0.8387
LZTF25	0.8447

The particle size is an important parameter in the ferrite materials with regard to their applications. The particle size values calculated by the Scherer formula show that the size of LiZnTi ferrite particles formed were in the nano-meter range. The values are given in Table 3.2.

Table3.2 . Particle size of LiZnTi ferrites.

So No.	Samples	Particle size nm
1	LZTF10	23.76
2	LZTF15	21.32
3	LZTF20	25.21
4	LZTF25	22.48

X-ray density:

X-ray densities for the samples have been calculated as explained in Chapter2 by using equation 2.5.The X-ray density decreases gradually from 5.32 to 4.83. The X-ray densities depend on molecular weight and the lattice constant of the ferrite sample.The X-ray densities are found to decrease with increase in the value of 'x', since lattice constant is inversely proportional to the X-ray density.

Table 3.3. X-Ray Densities forLiZnTi ferrite samples.

So No.	Samples	X-ray density gm/cc
1	LZTF10	5.32
2	LZTF15	5.03
3	LZTF20	4.97
4	LZTF25	4.83

Mass or bulk density:

The bulk densities ofLiZnTi ferrite samples (sintered at 500°C) increase with increase in Ti content. Fig 3.3 show the variation of bulk densities with the concentration of Ti. The bulk density of the samples is found to be highly dense as compared to the x-ray density values.

Table 3.4 : Bulk Densities of theLiZnTi ferrite samples.

So No.	Samples	Bulk density gm/cc
1	LZTF10	5.53
2	LZTF15	5.30
3	LZTF20	5.25
4	LZTF25	4.97

4. Conclusion

Titanium doped Lithium Zinc ferrites having general formula, $Zn_{0.6}Ti_xFe_{(2.2-1.5x)}O_4$ where x = 0.1, 0.15, 0.2 and 0.25 were synthesized by Combustion synthesis method at 450°C using Polyethylene glycol of molecular weight 6000 as fuel. XRD powder patterns for all the samples, indicate single phase cubic spinel structure formation for all the compositions. Thenano size particles ranging from 21 to 25nm were obtained. The LiZnTi ferrite samples show an increase in the lattice constant value as the concentration (x) of Titanium increased. The X-ray density decreases from 5.32 to 4.83, similar variation in density (bulk) was observed by experimental determination using Archimede's principle.Thedensities are found to decrease with increase in the value of 'x', since lattice constant is inversely proportional to the density.

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