



Microstructural, magnetic and electric properties of mixed Cs–Zn ferrites prepared by solution combustion method

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ABSTRACT

Nanosized zinc substituted ferrites with composition $\text{Cs}_{0.5-x/2}\text{Zn}_x\text{Mn}_{0.05}\text{Fe}_{2.45-x/2}\text{O}_4$ ($x = 0-0.5$) were prepared by solution combustion route. The ferrites obtained have been characterized by powder XRD, Mössbauer spectroscopy and Transmission Electron Microscope (TEM). Magnetic and electrical properties have also been studied. Powder X-ray diffraction analysis shows the formation of single phase cubic spinel structure. The saturation magnetization (M_s) initially exhibits an upward trend followed by a regular decrease with increasing diamagnetic Zn content. Curie temperature shows a downward trend with Zn content. The Mössbauer spectra display transition from ferrimagnetic to super-paramagnetic phase with increasing 'x' value. The temperature dependence resistivity shows regular decrease with temperature reflecting semiconductor behaviour of the ferrite samples. The permittivity (ϵ') and tangent loss ($\tan \delta$) measured at room temperature as a function of frequency shows the expected ferrite behaviour. TEM studies indicate the formation of nanosized ferrite particles. These results demonstrate promising features of Cs–Zn ferrites in microwave applications.

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1. Introduction

The ability to prepare nanostructures with defined morphologies and sizes in large scale is an essential requirement for applications in nanomaterials. As a result, extensive efforts have been devoted to develop synthetic capabilities to produce nanomaterials with tailored magnetic and electrical properties. The present-day microwave industry demands high-performance mixed ferrite materials capable of operating at high frequencies [1,2]. The mixed ferrites are the ferrimagnetic oxide materials exhibiting high resistivity, permeability and low eddy current losses. These novel materials are extensively used in radio, TV, radar, audio–video and digital recording, bubble devices, memory cores of computer and microwave devices [3–5]. An outstanding quantity of theoretical and experimental work has been carried out by engineers and physicists to understand the microstructural, electrical, dielectric and magnetic properties of spinel ferrites suitable for high-frequency applications [6–9]. Out of various existing spinel ferrites, alkali metal based ferrites have gained interest particularly for high-frequency telecommunication devices and in other high-frequency applications like circulator and filters because

of their high resistivity and hence low eddy current losses, high saturation magnetization and low-cost fabrication. Although several methods have been developed for the synthesis of mixed ferrites [10–13], solution combustion route has the advantage of obtaining nanosized and pure ferrites at lower temperature and in shorter time as compared to other conventional methods [14]. Several investigations have been reported on the synthesis and characterization of mixed lithium, sodium, potassium and rubidium ferrites [15,16] but nothing seems to be reported in literature on respective mixed Cs–Zn ferrites. In this paper, we therefore report the synthesis, characterization and magnetic/electric properties of nanosized Cs–Zn ferrites prepared by novel solution combustion method.

2. Materials used and method

The starting materials, such as caesium nitrate (99%, Spectrochem, AR), manganese nitrate (98.5%, Alfa Aesar), iron (III) nitrate (99.9%, Agro Organics), zinc nitrate (99.9%, Merck) and ethylene glycol (99.5%, Merck) were weighed in stoichiometric proportions. Aqueous solutions of respective metal nitrates prepared by using deionized water were mixed followed by the addition of ethylene glycol in desired molar ratio dropwise with vigorous stirring and the reaction mixture was combusted in muffle furnace at 600 °C for 30 mins. The powdered product i.e. $\text{Cs}_{0.5-x/2}\text{Zn}_x\text{Mn}_{0.05}\text{Fe}_{2.45-x/2}\text{O}_4$ ($x = 0-0.5$)

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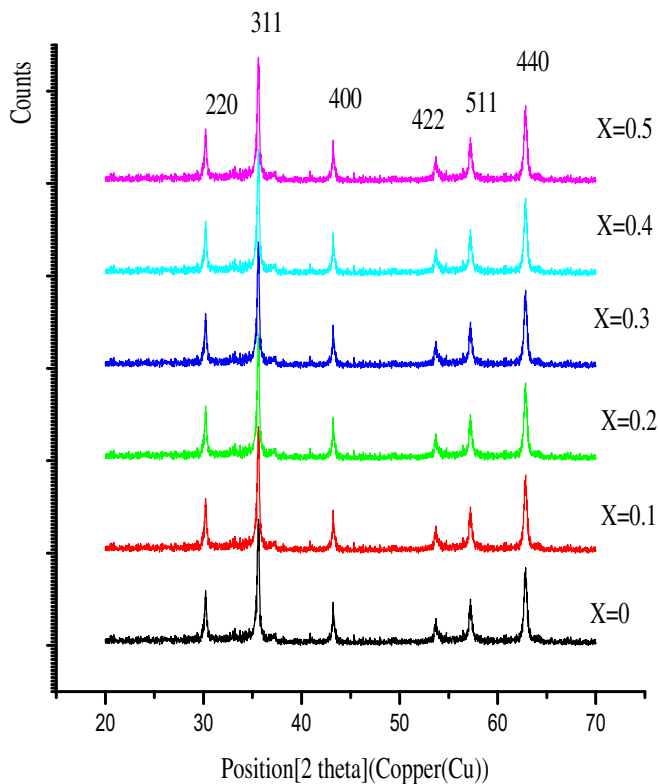


Fig. 1. X-ray powder diffraction pattern for $\text{Cs}_{0.5-x/2}\text{Zn}_x\text{Mn}_{0.05}\text{Fe}_{2.45-x/2}\text{O}_4$ with x varies from 0 to 0.5.

obtained was stored in a desiccator. Ethylene glycol used in this method acts as a fuel (capping agent) for the combustion synthesis of ferrites.

2.1. Instrumentation

X-ray investigations on the powders obtained were carried out by X-ray powder diffractometer (Rigaku made diffractometer, RINK 2000) using a $\text{Cu K}\alpha$ radiation ($\lambda = 1.54059 \text{ \AA}$) in a wide range of Bragg angles 2θ ($20^\circ \leq 2\theta \leq 80^\circ$) with step size of 0.0170 and scan step time of 20.0286 s^{-1} . The size and shape of ferrite particles were analysed by transmission electron microscope (TEM, Hitachi H-7500). The elemental analysis of the samples was performed using the EDXRF spectrometer. The exciter source consisted of a 3 kW long-fine-focus Mo-anode X-ray diffraction tube along with a 4 kW X-ray generator procured from PanAnalytic, The Netherlands. A Si (Li) detector ($100 \text{ mm}^2 \times 5 \text{ mm}$, $8 \mu\text{m}$ Be window, $\text{FWHM} = 180 \text{ eV}$ at $\text{Mn K}\alpha$ X-rays, Canberra, US) in the horizontal configuration coupled with a PC based multichannel analyser was used to collect the fluorescent X-ray spectra. Spectra were taken using the setup with the X-ray tube operating voltage 38 kV and a combination of the selective absorbers based on the ^{30}Zn , ^{35}Br and ^{38}Sr elements (K-shell jump ratios ~ 7) in the incident beam. Infrared studies were carried out on Varian 660, FTIR system after preparing pellets with KBr.

^{57}Fe Mössbauer spectra were recorded on Wissel (Germany), Mössbauer spectrometer. A ^{57}Co (Rh) γ -ray source was employed and the velocity scale was calibrated relative to ^{57}Fe in Rh matrix. Mössbauer spectral analysis software WinNormos for Igor Pro has been used for the quantitative evaluation of the spectra. Isomer shift values were reported with respect to pure metallic iron absorber. Curie temperature for the Cs–Zn ferrite samples was

Table 1

Variation of various XRD parameters with composition 'x' for $\text{Cs}_{0.5-x/2}\text{Zn}_x\text{Mn}_{0.05}\text{Fe}_{2.45-x/2}\text{O}_4$.

Composition (x)	Molecular weight	Density (d_{XRD}) g/cm^3	Density (d_{Exp}) g/cm^3	Porosity (%)	Lattice parameter 'a'
0	270.45	6.0974	5.7019	6.45	8.3845
0.1	267.54	6.0180	5.6659	5.89	8.3909
0.2	264.63	5.9335	5.6158	5.35	8.3999
0.3	261.72	5.8477	5.5543	5.03	8.4097
0.4	258.51	5.7644	5.5298	4.06	8.4186
0.5	255.90	5.6698	5.4103	4.58	8.4333

determined by using a simple experimental setup based on gravity effect in the laboratory.

Saturation magnetization values were measured by using Vibrating Sample Magnetometer (Lake Shore's new 7400 series). The electric properties (dielectric constant and tangent loss) were measured with 8714ET precision LCR metre. Resistivity of the samples was measured by using two probe Kethley high sensitive resistivity metre.

3. Results and discussion

3.1. Microstructural analysis

Fig. 1 shows the X-ray diffraction patterns for different compositions of $\text{Cs}_{0.5-x/2}\text{Zn}_x\text{Mn}_{0.05}\text{Fe}_{2.45-x/2}\text{O}_4$. The diffraction peaks (220), (311), (400), (422), (511) and (440) reveal the existence of single phase cubic spinel ferrites and are comparable to those reported for respective lithium ferrites [5]. Table 1 and Fig. 2 (a) shows the variation of lattice constant as a function of 'x'. The value of lattice constant 'a' increases with increasing Zn content (x) in the composition. The lattice constant 'a' can be calculated theoretically by the following relation.

$$a = \left(8/3\sqrt{3}\right) \left[(r_A + r_O) + \sqrt{3}(r_B + r_A) \right]$$

where r_O is the radius of oxygen ion, r_A and r_B are the ionic radii of tetrahedral (A) and octahedral (B) site respectively. This relation clearly indicates that there exists a correlation between the ionic radii and the lattice constant. This is attributed to the substitution of larger Zn^{2+} cation (0.083 nm) for smaller Fe^{3+} cation (0.067 nm). Caesium ferrite being inverse spinel, have all the Cs^+ ions in octahedral position along with half of the Fe^{3+} ions and remaining Fe^{3+} ions occupy tetrahedral site. The addition of Zn^{2+} ions which have strong affinity for tetrahedral site, only Fe^{3+} ions present at tetrahedral site get replaced resulting in an increase in lattice parameter.

The theoretical or X-ray density (d_{XRD}) of the various compositions of Cs–Zn series has been calculated by using the relationship [17]:

$$d_{\text{XRD}} = 8M/Na^3$$

where M is Molecular weight of the ferrite, N is Avogadro's number and 'a' is lattice constant obtained from the different XRD patterns. In Fig. 2 (b) the theoretical/X-ray density (d_{XRD}) and experimental density shows a regular decrease with increasing 'x' value due to a decrease in molecular weight of the ferrite. The magnitude of observed and calculated densities have been found to be comparable. Both parameters show a downward trend with increasing magnitude of 'x'. However, the X-ray density for any given composition is higher than that of the experimental density and this difference is primarily due to the porosity of the material.

The percentage porosity for all the compositions was calculated by using the equation:

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