

Synthesis and Characterization of $Zn_{0.5-x/2} Mg_x Fe_{2(2.5-x/2)} O_4$ Nanocrystalline ferrite Prepared by Auto-Combustion Method

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Abstract

Ferrites having chemical formula, $Zn_{0.5-x/2} Mg_x Fe_{2(2.5-x/2)} O_4$ with $x = 0, 0.1$ and 0.2 have been prepared by auto-combustion route. XRD confirms the absence of extra line formation the single phase of the ferrite samples. The patterns indicate well-defined peaks of crystalline FCC phase which confirm spinel cubic structure formation for the samples. Average crystallite size from Scherer Formula is found to be nano range. Infrared spectroscopy detects tetrahedral and octahedral metal ion complexes. Microstructure studied using Scanning Electron Microscope (SEM) gives dense like structure.

Keywords: Spinel ferrites; XRD; Activation energy; Drift mobility; jump length;

Introduction

Spinel nanocrystalline ferrites are one of the best magnetic semiconductors which have a great importance in telecommunication, microwave and electronics engineering. They are used in variety of applications ranging from simple function devices, like small permanent magnets, to sophisticated device used for the electronic industry [1,2]. Some interesting applications of these materials are found in computer peripherals, telecommunication equipments, permanent magnets, electronic and microwave devices, magnetic media used in computers, recording devices and magnetic cards. The electrical and magnetic properties are the most important properties of ferrites, which depend on the processing conditions, sintering temperature, chemical composition and the amount of the additives [3]. High electrical resistance and lower saturation magnetization (M_s) are the characteristics properties of soft ferrites which are not found in traditional soft ferromagnetic materials like silicon, steels or perm alloys [4].

In recent years, nanocrystalline ferrite materials are becoming a subject of intense research because of their unique properties, from the fundamental and the application point of view [5]. Ferrite with a spinel structure which is formed by a nearly closepacked FCC array of anions with holes partly filled by the cations can be represented by the formula $M Fe_2 O_4$ [6], where M represents divalent metallic ions located in M interstitial (tetrahedral) sites and Fe metallic ions located in (octahedral) sites. Due to the large electronegativity of oxygen, the ionic type of bonds prevails in almost all oxide spinels [7]. Soft spinel ferrite ($M Fe_2 O_4$, $M = Co, Ni, Zn, Mn, etc.$) nanoparticles have been intensively investigated due to their remarkable magnetic and electrical properties and wide practical applications in ferrofluids technology magnetic drug delivery, magnetic high-density information storage etc [8, 9, 10].

The aim of our present work is to optimize a composition of the Zn-Mg based ferrite to achieve high-performance soft magnetic materials prepared using such a simple and low-cost method will be more promising in high-frequency applications and industrialization. Hence in the present paper, we have

specially focused on the effect of Mg substitution in Zn on micro-structural, electrical properties were studied and co-related in light of literature in fundamental point of view.

Experimental

Ferrite prepared by ceramic method involves high-temperature to complete the solid-state reaction between the constituent oxides or carbonates. The particles obtained by this method are rather large and non-uniform in size. These non-uniform particles, on compacting result in the formation of voids or low density. In order to overcome these difficulties arising due to ceramic method, many researchers have chosen wet chemical method like co-precipitation, hydrothermal processing, auto-combustion etc for production of homogeneous, fine and reproducible ferrites at low temperature [11]. Hence in the present case, we have successfully synthesized Mg substituted Zn ferrite using auto-combustion technique. The details about the synthesis parameters and structural analysis are reported in our previous paper [12].

Results and Discussion

XRD Study

The X-ray diffraction patterns for the present samples with general formula $Zn_{0.5-x/2} Mg_x Fe_{2(2.5-x/2)} O_4$ with $x = 0, 0.1$ and 0.2 are shown in Fig.3.1 It can be seen that all samples exhibit cubic spinel structure. The absence of extra line confirms the single phase formation of the ferrite samples. The peaks were indexed by comparing the interplanar distance with JCPDS data (JCPDS card no .48-0489,48-0492,77-0013) corresponding to Zn-Mg ferrite. The patterns indicate well-defined peaks of crystalline FCC phase which confirm spinel cubic structure formation for the samples. No additional impurity reflections were observed ensuring the phase purity. The crystallite sizes of the studied samples calculated using Debye – Scherrer formula [13] are listed in Table-3.1.

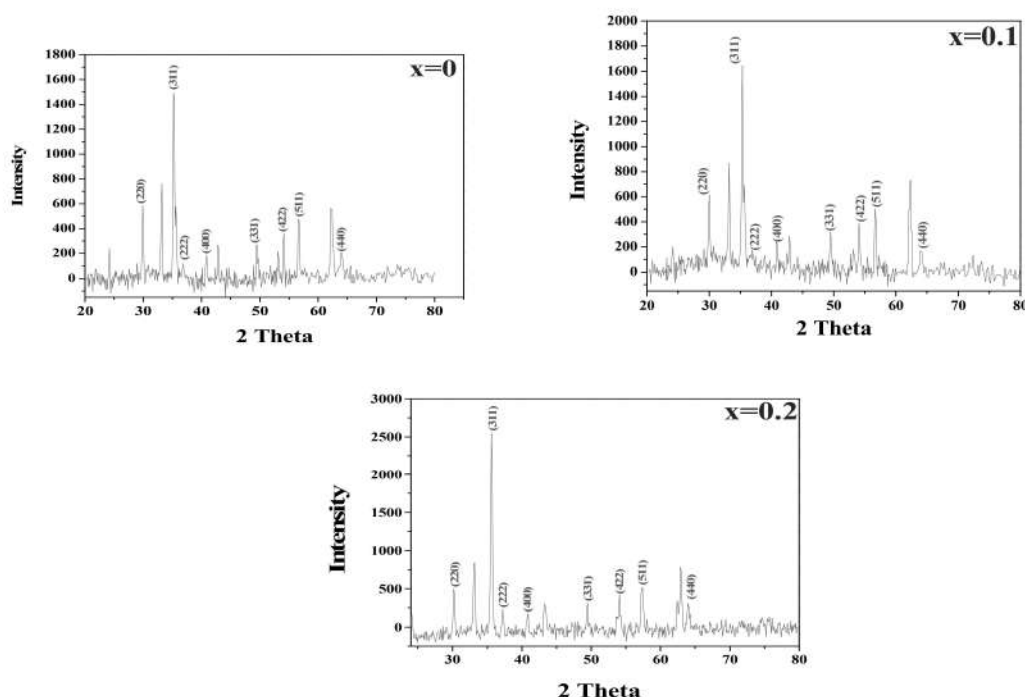


Fig 3.1 shows of the sample $x=0, 0.1$ and 0.2 of $Zn_{0.5-x/2} Mg_x Fe_{2(2.5-x/2)} O$ ferrites.

Table 3.2 Crystalline size of the sample $x=0, 0.1$ and 0.2 of $Zn_{0.5-x/2}Mg_xFe_{2(2.5-x/2)}O$ ferrites.

IR	Mg-content x	Lattice parameter A°	Crystallite size from Scherrer Formula 'nm'
	0	8.3924	28
	0.1	8.4071	20
	0.2	8.3799	34

Study

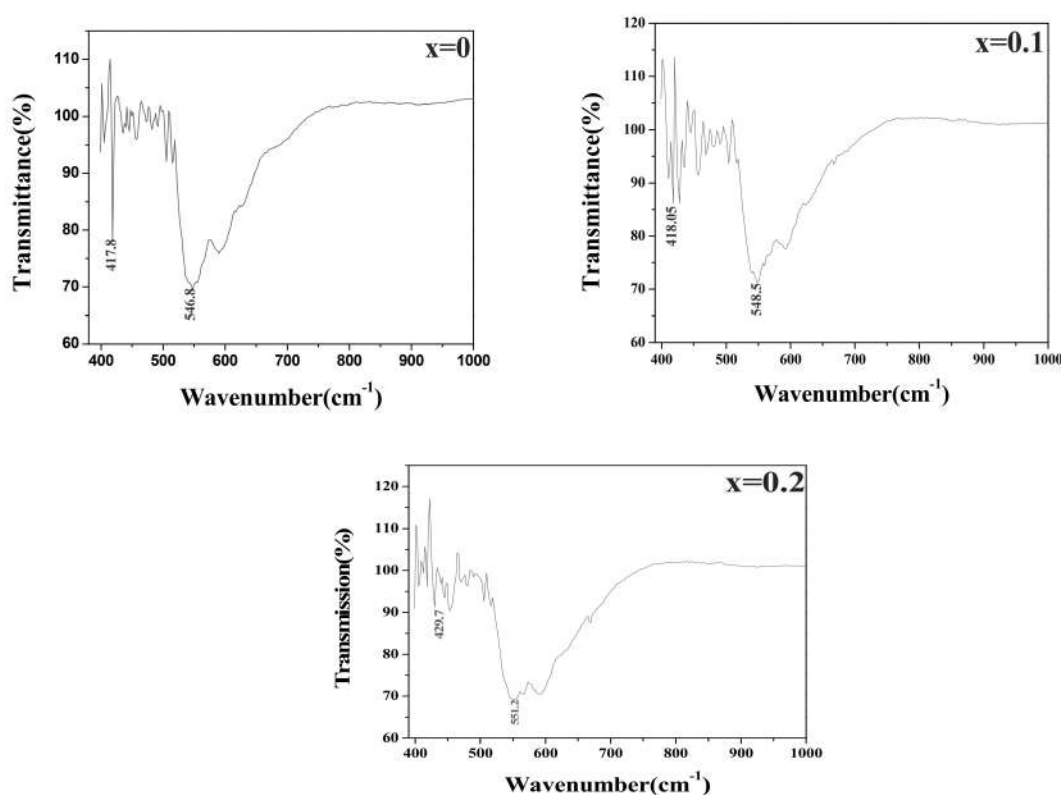


Fig. 3.2. Shows IR spectra of the sample $x=0, 0.1$ and 0.2 of $Zn_{0.5-x/2}Mg_xFe_{2(2.5-x/2)}O$ ferrites

Fig. 3.2 shows IR spectra for the sample $x=0, 0.1$ and 0.2 of $Zn_{0.5-x/2}Mg_xFe_{2(2.5-x/2)}O$ ferrites for three typical samples $x=0, x=0.1$ and $x=0.2$. IR spectra of the Zn-Mg ferrite samples recorded between 400 cm^{-1} and 1000 cm^{-1} . The spectra elucidate the positions of cations in the crystal structure with oxygen ions and their vibrational modes, which represents the various ordering positions and structural properties for the different compositions. In ferrites, the metal cations are situated in two different sub-lattices, namely tetrahedral (A-sites) and octahedral (B-sites) according to the geometric configuration of the oxygen ion nearest neighbors. The band ν_1 around 600 cm^{-1} is attributed to stretching vibrations of tetrahedral complexes and ν_2 around 400 cm^{-1} cm to that of octahedral complexes. It is noticed that by

increasing the copper content, the frequency ν_1 is shifted to lower frequencies, while the position of ν_2 slightly changed. It is also obvious that the peak intensity changes with increasing Mg content. The difference in the band position is expected because of the difference in $\text{Fe}^{3+}\text{-O}_2^-$ distance for the octahedral and tetrahedral sites. It was found that the Fe–O distance of A-sites (0.189 nm) is smaller than that of the B-sites (0.199 nm) [14]. This has been interpreted by the more covalent bonding of Fe^{3+} ions at the A site. Ni^{2+} and Cu^{2+} ions show a strong preference for B-sites due to favorable fit of charge distribution, while Fe^{3+} is distributed in both the octahedral and tetrahedral sites [14, 15]. So the $\text{Fe}^{3+}\text{-O}^{2-}$ stretching vibration is affected by the replacement of Mg^{2+} by Zn^{2+} ions at octahedral sites in the ferrite lattice, which have a larger ionic radius and higher atomic weight than Fe^{3+} . Due to charge imbalance, the oxygen ion is likely to shift towards the Fe^{3+} ions, making the force constant between Fe^{3+} and O^{2-} is greater. Hence, an increase in the bond stretching frequencies was expected in the inverse ferrite [14]. The intensity ratio is a function of the change in dipole moment with the inter-nuclear distance. This value represents the contribution of the ionic Fe–O bond in the lattice. So the observed decrease in the peak intensity with increasing Mg content is expected due to the perturbation occurring in the Fe–O bands by the substitution of Mg^{2+} by Zn^{2+} .

SEM Study

The SEM micrographs of the samples are shown. The average grain diameter was measured by counting the number of grain boundaries intercepted by a measured length of a random straight line drawn on the graph i.e. by line intercept method.

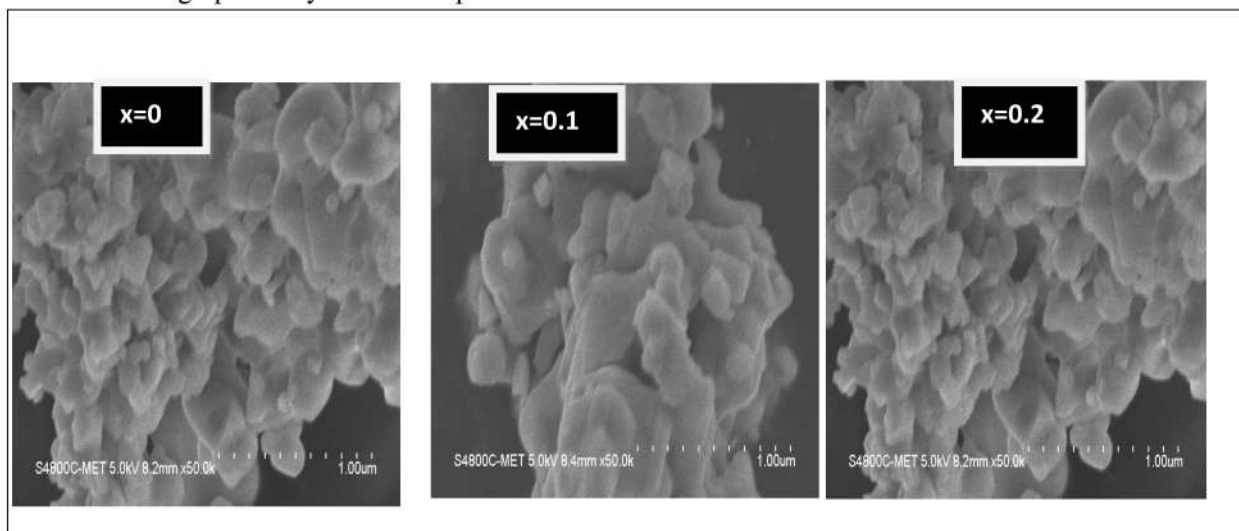


Fig. 3.3 Shows the SEM micrographs of sample $x=0$, 0.1 and 0.2 of $\text{Zn}_{0.5-x/2}\text{Mg}_x\text{Fe}_2(2.5-x/2)\text{O}$ ferrites

Fig 3.3 shows the SEM micrographs of the samples with $x=0$, 0.1 , and 0.2 . It is observed that majority of grains are spherical in shape. The SEM micrograph for $x = 0$ shows well dispersed grains with identical shape and size. It is interesting to note that, as the amount of Mg^{2+} content increases, the nature of microstructure gets modulated. In the case of $x = 0.1$ the grains are non identical in size and shape whereas for the case of $x = 0.2$ the grains are agglomerated. Agglomeration is understood to increase with sintering temperature and hence some degree of agglomeration at higher sintering temperature appears unavoidable [16].

Conclusions

The auto-combustion method is found to be convenient for the synthesis of nanocrystalline Mg–Zn ferrites. X-ray diffraction patterns correspond to well defined FCC phase and confirm the spinel structure. No additional impurity reflections were observed ensuring the phase purity. The formation of single-phase cubic spinel ferrites was confirmed by IR patterns. The IR spectra exhibit two major bands in the range i.e. The high frequency band ν_1 is in the range 600 cm^{-1} and the lower frequency band ν_2 is in the range 400 cm^{-1} . The absorption band ν_2 is caused by the stretching vibration of tetrahedral metal-oxygen bond, and the octahedral sites. we are deals with the micro structural analysis of ferrites. The morphological features were observed using Scanning Electron Microscope . It is a powerful technique to a magnification 10,000 and the grain size was calculated using the line intercept method. It is also observed that as the amount of Mg²⁺ content increases, The agglomeration is due to high sintering temperature.

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