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Study of structural and magnetic properties of Nd doped zinc ferrites



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1. Introduction

Nanophase ferrite are a class of materials and have been extensively investigated due to their potential applications in non-resonant devices, radio frequency circuits, high quality filters, road antennas, transformer cores, read/write heads for high speed digital tapes [1–3] and also in gas sensing [4]. The physical properties of nanophase ferrites depend on the method of synthesis, doping of cations in the host crystal structure, size and morphology of the particles [5-6]. Synthesis and characterization of new magnetic materials have received considerable attention, especially in the studies of nanocomposite materials with tailored electric and magnetic properties. The magnetic properties of ferrites can be tailored by controlling the different types and amount of metal ions substitution [7]. Introduction of rare earth atoms magically modifies magnetic properties of ferrites [8]. For example, doping the parent spinel ferrite with rare earth ions leads to structural disorder and lattice strain, thereby increasing the electrical and magnetic parameters [9]. In particular, an appreciable decrease in saturation magnetization (25 emu/g) was noticed with increasing Gd ion content [10]. Recently, neodymium ions have been introduced in copper zinc ferrites with the objective of improving the magnetic performance of the materials [11]. Several synthesis methods have been used to synthesize nanophase magnetic materials and each synthesis method has its own merits and demerits [12–15]. Among various methods, the co-precipitation technique is one of the efficient methods because control over the surface morphology, structure and

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ABSTRACT

 $ZnFe_{2-x}Nd_xO_4$ (where x = 0.012, 0.025, 0.050, 0.075, and 0.1) nanocrystalline ferrites were synthesized by the co-precipitation method followed by the heat treatment at 800 °C for one hour. The morphological and structural properties of the mixed system were characterized by XRD, FTIR, TEM techniques. Infrared spectra show two strong absorption bands in the frequency range $400-600 \text{ cm}^{-1}$ which were respectively attributed to tetrahedral and octahedral sites of the spinel ferrite. The effect of Nd content on crystallites size, lattice parameter and X-ray density of the material has been discussed. The crystallites size of the mixed system was found in the range of 16.8–29.5 nm. Magnetic properties of the material were investigated by the VSM technique. VSM results suggest that the energy of the mixed ferrite, in an external field, is proportional to its crystallite size and the number of magnetic particles in a single magnetic domain. No significant coercivities were recorded which indicates the super paramagnetic behavior of synthesized nanophase mixed ferrites.

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chemical composition can be attained by carefully monitoring the preparation parameters. The advantage of this method includes processing at lower temperature, simpler synthetic steps and the ease of compositional control. In the co-precipitation method, particles are produced in the range of few nanometers to hundred nanometers, as we can easily control the size of particles by changing the hydrolysis time, pH of solution, concentration of precursors etc. Nanophase zinc ferrite (ZnFe₂O₄) is a hard spinel with high saturation magnetization, coercive field, mechanical hardness and high magneto crystalline anisotropy. There are total 64 tetrahedral and 32 octahedral interstitial sites available within each unit cell [16], in which zinc ions occupy tetrahedral sites (A-sites) while iron and manganese ions occupy both tetrahedral sites (A-sites) and octahedral sites (B-sites) [17]. This special structure allow introduction of different metallic ions to change the magnetic, electronic and other properties considerably [18,19]. In present study, synthesis and characterization Nd doped zinc ferrites. To synthesize Nd–Zn ferrite sample with different Nd contents, the coprecipitation technique is used and samples were annealed at 800 °C. Effect of doing concentration on structural and magnetic properties of $ZnFe_{2-x}Nd_xO_4$ (x is content in percentage) nanocrystalline have been discussed.

2. Experimental details

2.1. Preparation of samples

Nanocrystalline samples of neodymium doped zinc ferrite with composition $\text{ZnFe}_{2-x}\text{Nd}_x\text{O}_4$ (where x=0.012, 0.025, 0.050, 0.075, and 0.1) were synthesized by the co-precipitation method. High purity salts Zn(NO₃)₂ · 6H₂O, Nd(NO₃)₃ · 5H₂O and Fe(NO₃) · 9H₂O were taken as starting materials. Aqueous solutions were prepared and mixed in respective stoichiometric composition. The prepared solution was heated at 80 °C to make it homogeneous. Precipitation was accomplished with constant stirring and adding sodium hydroxide solution drop by drop to maintain the pH of solution at about 12. The precipitated particles were then washed several times with double distilled water to remove the salt residues and other impurities. The precipitated particles were then dried at 100 °C to obtain the powders. The formation of nano-phase ferrite is a two step process: the first one is conversion of metal salts into hydroxides (co-precipitation step) and the second is transformation of hydroxides into nanoferrites (fertilization step). The samples were further annealed at 800 °C (1 h) to improve the crystalline properties of the $ZnFe_{2-x}Nd_xO_4$.

2.2. Characterization of samples

Complementary methods were used to characterize annealed $ZnFe_{2-x}Nd_xO_4$ samples. In order to determine the crystallite size and lattice constant, XRD patterns of samples were recorded by an X-ray diffractometer (Philips PW/1710) with Cu filter using monochromatic CuK α radiation of wavelength 1.548 Å at 50 KV and 40 mA, in the scanning range $2\theta \sim 10-80^{\circ}$. To investigate the particle size distribution and morphology of the samples, TEM micrographs were recorded using transmission electron microscope (Hitachi-H7500) at 100 KV. For this purpose, samples were dispersed in ethyl alcohol in an ultrasonic bath and then pipetted on a carbon-coated copper grid. To study the compositional characteristics from optical excitation of vibrational modes, infrared spectra were recorded in the range 4000–400 cm⁻¹ using Fourier transform infrared (FTIR) spectrometer (Perkin-Elmer 1600). For FTIR characterization, pellets were obtained by homogeneously mixing the samples with KBr powder and then pressing. The saturation magnetization and hysteresis parameters of the samples were measured at room temperature using high field loop tracer operating at 230 V, 50 Hz.

3. Results and discussion

3.1. XRD study

Fig. 1 reveals the XRD patterns of annealed $ZnFe_{2-x}Nd_xO_4$ samples. For all five Nd doped samples the crystallite size (D_{D-S}) has been obtained by the well known Debye–Sherrer relation [20]

$$D_{D-S} = k\lambda / \beta_{hkl} \cos \theta_{hkl}, \tag{1}$$

In order to determine crystallite size and lattice strain simultaneously in the sample, we have used the Williamson–Hall (W–H) equation

$$\beta_{hkl} \cos \theta_{hkl} = \frac{k\lambda}{D_{W-H}} + 4\varepsilon \sin\theta hkl,$$
 (2)

λ is the wavelength of X-ray used, *θ* is the Bragg angle, and *β* is FWHM of the diffraction peak. D_{W-H} is the average crystallite size from W–H plot, *ε* is the lattice strain. Check cell code was run for Nd³⁺ content x=0.012 and found that plane corresponding to diffraction peaks at $2\theta \sim 29.89^{\circ}$, 35.31°, 56.63°, and 62.19° could be assigned to the Miller indices (220), (311), (511) and (440), respectively. These indices are of cubic zinc ferrite structure (JCPDS-22-1012). Introducing an appropriate amount of Nd³⁺ ions (x=0.025) into Zinc ferrite, an additional peak at $2\theta=32.51^{\circ}$ was appeared which indicates the presence of the secondary phase. Using this single peak it is very difficult to identify the structure of the secondary phase, however comparing this peak with JCPD card no. 82-2421 data and it may be identified as NdFeO₃



Fig. 1. XRD patterns of Nd³⁺ doped zinc ferrites at x=0.012, 0.025, 0.050, 0.075 and 0.1.

phase. For x = 0.05, the value of peak intensity at $2\theta = 32.51^{\circ}$ (122) is almost the same to the characteristic peak of ferrite at 2θ =34.31° (311). This result indicates that by introducing Nd³⁺ (x=0.05) in pure zinc ferrite sample using the co-precipitation technique and followed by annealing at 800 °C, a coexisted phase of pure zinc ferrite (primary phase) with the orthorhombic phase of NdFeO₃ (secondary phase) can be obtained. Further, Fig. 1 reveals that the peak intensity of orthorhombic phase becomes stronger with the increase of Nd^{3+} ion content up to x=0.1. Results suggest that when concentration of Nd³⁺ ion is very less in zinc ferrite samples, the tetrahedral sites of cubic structure are occupied by zinc ions (r=0.6 Å) whereas the octahedral sites by iron ions. Further, substitution of Fe³⁺ (r=0.87 Å) by Nd^{3+} (r=1.16 Å) determines a gradual change in the crystallite structure from cubic to orthorhombic one. The values of crystallites size obtained using the Debye-Scherrer and Williamson-Hall (W-H) equations are given in Table 1. It is noticed that size of zinc ferrite nanocrystallites increases almost linearly with increasing Nd³⁺ content. Fig. 2 shows W-H plot in which the values of strain are estimated from slope of the lines and given in table 1. The positive slopes of W-H plot manifest tensile strain present in the annealed samples and the value of crystallite size increases with increase in Nd³⁺ concentration. The increase crystallite size induces a lattice strain in the crystal structure. This may be explained on the basis of defects present in the parent ZnFe₂O₄, which allows the absorption of lattice strain caused by the higher dopant amounts of Nd³⁺ ions. Further, effect of Nd³⁺ content on lattice parameter is also investigated by using well known formula:

$$a = \lambda \sqrt{(h^2 + k^2 + l^2)/2} \sin \theta, \tag{3}$$

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