



GD DOPED COBALT FERRITE NANOPARTICLES: SYNTHESIS, STRUCTURAL AND MAGNETIC PROPERTIES

Dr. Karadile
Haribhau
Jagannath

Department Of Physics, Shri Dnyaneshwar Mahavidyalaya, Newasa, Ahilyanagar (MS) India

ABSTRACT Spinel ferrites are magnetic materials that have exceptional structural features. In this study we investigated the influence of Gallium Doping on the structural, electrical and magnetic properties of $Ga_xCoFe_{2-x}O_4$ ($x = 0.00, 0.05, \text{ and } 0.10$). For structural analysis as prepared ferrite powder were characterized by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Magnetic properties. The results confirm the formation of pure cobalt ferrite phase for low Ga^{3+} content. The crystallite sizes and lattice parameters were influenced by the presence of Ga^{3+} , due to their large ionic radii determined a distortion of the spinel phase and cation redistribution between the octahedral and tetrahedral sites. The microstructural study is carried out by using SEM technique and the average grain size is increased with Gd doping. Magnetic analysis as prepared samples was tested with Vibrating Sample Magnetometer (VSM).

KEYWORDS : Spinel ferrite, Synthesis, XRD, SEM and VSM.

INTRODUCTION

Research on magnetic materials, especially spinel ferrites has been fairly active in the last several years because of the wide range of potential applications including digital recording heads, sensors, transformers, telecommunications and computers etc. [1-3]. The structural, micro-structural and optical properties were studied by using xrd, sem-ed and flir. The dc electrical resistivity of spinel is of great importance which provides valuable information regarding conduction mechanism of spinel ferrite. Magnetic properties of ferrites have been widely studied because of their relevance to magnetic recording, biomedical applications, etc. It has been reported that the magnetic properties of the spinel type ferrites can be tuned by effectively substituting various metal ions in the ferrite lattice. Substitution of small amount of other ions can lead to cation redistribution in the octahedral and tetrahedral sites of the spinel structure thereby causing changes in the saturation magnetization and coercivity of the materials. The magnetic properties of pure and gallium substituted cobalt ferrite have been studied in details in order to explore its applications in high density storage, sensors and high frequency applications [4-7]. Spinel structures are generally represented as AB_2O_4 where (A) represents the divalent ion and [B] represents as the trivalent ion. Cobalt ferrite possesses inverse spinel structure and it has been reported that the trivalent ion Fe^{3+} is distributed equally in the tetrahedral and octahedral site [3]. However this type of cation distribution is very sensitive towards the method of synthesis, annealing temperature etc and finding out exact cation distribution for a specific composition requires support from multiple techniques such as IR, XPS and Mossbauer spectroscopy. The substitution of different cations such as Zn, Ga, Cd, etc has been carried out in cobalt ferrite and it is possible to carry out the substitution for both Co or Fe in $CoFe_2O_4$ [8-10]. Resistivity of the prepared sample was studied by using two probe methods. It is found that DC electrical resistivity was found to be increased with increase in gallium content x. Among the magnetic materials, cobalt ferrite, the common composition $CoFe_2O_4$ is of great importance because of its high H_c , moderate M_s , good chemical stability, and high mechanical hardness [11]. Recent studies on the $CoFe_2O_4$ nanomaterials have attracted interest due to the size-dependence of the magnetic properties. For example, M_s is smaller than that of the bulk one about 80 emu/g due to the disordered canting spins on the surfaces [12-15]. H_c reaches a maximum value as the crystallite size is near the single-domain size, which is about 30-40 nm [16,17] and if the crystallite size is smaller than 10 nm, the H_c of the single-domain $CoFe_2O_4$ nanoparticles may decrease to zero at room temperature, i.e., the super paramagnetism occurs [18,19]. Since Co is mainly responsible for anisotropy in cobalt ferrite, we have carried out direct substitution of nonmagnetic Ag for Fe in cobalt ferrite in order to investigate the effect on magnetic behavior. Ag substitution is likely to affect the cation distribution and thereby significantly alter the magnetic properties. In the latest several years, many synthesis technologies such as auto combustion [14,19,20], sol-gel [21,22], high-energy ball milling [23], thermal decomposition methods [24,25] and hydrothermal method [11,12,26-28] have been developed to prepare the single-domain $CoFe_2O_4$ nanoparticles. Among all these techniques, the sol gel

technique has been widely used due to the effective size-control and the easy synthesis steps.

Experimental Synthesis

Ag substituted cobalt ferrite ($CoAg_xFe_{2-x}O_4$, ($x = 0.00, 0.05, \text{ and } 0.10$)) samples of the nanocrystalline material were prepared using sol-gel auto combustion technique. All AR grade chemicals such as cobalt nitrate ($Co(NO_3)_2 \cdot 6H_2O$), Ferric nitrate ($Fe_2(NO_3)_6 \cdot 9H_2O$), Gallium nitrate ($Ga(NO_3)_3 \cdot 6H_2O$) are used without any further purification. Citric acid ($C_6H_8O_7$) was used as fuel for the synthesis. The metal nitrates to fuel (citric acid) ratio was taken as 1: 3. The pH of the reacting solution was maintained 7 using stepwise addition of Ammonia solution. The temperature was maintained around 80 °C and the solution was heated with constant magnetic stirring for 4 hours till the solution was converted into brown gel. After this, the stirring was stopped and temperature of gel was increased up to 120 °C. After a while the gel was self ignited and turned in to ash, which is then cooled to room temperature and grinded nearly for half an hour using agate mortar pestle. The as synthesized powder is sintered at 600 °C for 4 hrs, again grinded for 30 min. to ensure complete spinel and then used for further investigations of structural electrical and magnetic properties. The nanoparticles were then pressed with KBr Press into discs.

Characterizations:

The XRD patterns were recorded at room temperature in the 2 θ range of 20° to 80° using Cu-K α radiation ($\lambda = 1.54056 \text{ \AA}$) in a scanning rate of 1° per minute at a step size of 0.02°. The phase purity of the compounds was studied using the X-ray diffraction. The surface morphologies of these samples were observed using a Scanning emission electron microscope. FEI Quanta FEG 200 scanning electron microscope (SEM) equipped with energy dispersive spectroscopy (EDS) is used to examine the micro structural features such as grain size and porosity. The magnetization and coercivity was calculated by using Vibrating Sample Magnetometer (Lakeshore VSM 7410S).

RESULTS AND DISCUSSION

Powder X-ray Diffraction Study: Structural Analysis

The X-ray diffraction (XRD) patterns were recorded for the finely grained samples of the $Ga_xCoFe_{2-x}O_4$ system with $x = 0.00, 0.05, \text{ and } 0.10$ are shown in Fig.1. The XRD pattern shows the reflections corresponding to (2 2 0), (3 1 1), (4 0 0), (3 2 2), (4 2 2), (5 1 1) and (4 4 0), which are allowed peaks of the cubic spinel structure. The plane (3 1 1) is most intense whereas others are relatively low intense. The analysis of XRD pattern revealed the formation of single phase cubic spinel structure. The increase in lattice constant is attributed to the fact that, the Fe^{3+} (0.74 Å) ions of smaller ionic radii are replaced by Ga^{3+} (0.84 Å) ions of larger ionic radii. Our results on the variation of lattice constant with Ag substitution are fairly agreed well with those reported in the literature [30-32]. The variation of lattice constant with gallium substitution is shown in table 1. The values of lattice constant for $x = 0.00, 0.05 \text{ and } 0.10$. i.e. for $CoFe_2O_4$ and $CoGa_xFe_{2-x}O_4$ are in good agreement with the reported value of lattice constant [33]. The most intense peak (3 1 1) was considered for the determination of the full

width at half maxima (FWHM). The values of crystallite size are given in Table 1. The interplanar spacing d is calculated by using the Bragg law of XRD, as: $n\lambda = 2d \sin\theta$, where, (n) is the order of diffraction, (λ) is the wavelength of the X-ray employed is equals to 1.54056Å for the Cu- α , source and (θ) is Bragg's angle. The crystallite size (t) of the Co-Ga ferrite nanoparticles was calculated from the main peak (311) in the XRD patterns using the Debye-Scherrer's equation (35),

$$t = k\lambda/\beta \cos\theta$$

where, k is Scherer's constant and is equals to 0.89 for spinel ferrite, (β) is the full width at half maximum (FWHM) of the peak (311) recorded in XRD pattern, taken in radians. The X-ray density (d_x) of the ferrite nanoparticles was calculated using the equation given by, $d_x = 8M/a^3N_A$, Where, (M) is the molecular weight of the composition, (N_A) is the Avogadro number and (a) is the lattice constant. A semi-empirical relationship using to estimate the surface area of these nanoparticles by the following equation (36), $SSA = 6000/d_x$ (cm^2/g). L_A and L_B are determined from the following relations (37): $L_A = 0.25a\sqrt{3}$, $L_B = 0.25a\sqrt{2}$. Where, (L_A) and (L_B) denoted to the hopping length for A and B sites, respectively. All X-ray characteristic parameters of the $\text{CoGa}_x\text{Fe}_{2-x}\text{O}_4$ spinel ferrite nanoparticles sintering at temperature 600°C, are shown in table 1.

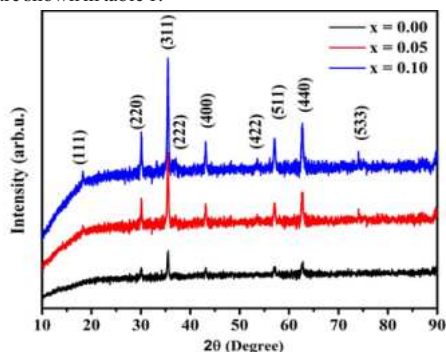


Fig.1. XRD patterns of Ga Doped $\text{CoFe}_{2-x}\text{O}_4$ ($x=0.00, 0.05$ and 0.10)

Table 1: X-ray parameters; t (crystallite size), a (lattice constant), d_x (x-ray density), L_A , L_B (hopping length for tetrahedral and octahedral sites respectively), Porosity (%), Cell Volume (Å^3), SSA (Specific Surface Area) for Co-Ga ferrites Calcinated at Temp. 600 °C.

Sample s	2θ(deg.) Peak (311)	t nm	a Å	d_x (g/cm^3)	L_A (Å)	L_B (Å)	(%)	V (Å^3)	SSA (m^2/g)
$x=0.00$	35.48	14.3 4	8.38 0	5.27 65	3.63 3	2.96 6	24.7 23	590.69 4	52.783
$x=0.05$	35.48	14.4 5	8.38 8	5.29 65	3.63 2	2.96 5	24.7 23	590.70 4	108.06 6
$x=0.10$	35.46	14.5 6	8.39 0	5.30 74	3.63 31	2.96 6	24.7 23	590.72 9	81.141

3.2 Surface Morphology

The scanning electron microscopy studies were undertaken for the samples $\text{Ga}_x\text{CoFe}_{2-x}\text{O}_4$ ($x = 0.00, 0.05$ and 0.10) and images are shown in Fig. 3. The SEM images clearly indicate the well grown grains having spherical nature and showing agglomeration, similar observations are made by [40]. It can be seen that the grain size and shape are significantly affected by the substitution of Gd. The morphology of grains is in spherical and shows the decreasing trend with increasing copper concentration in the synthesized ferrites. The average grains sizes of the ferrites are in the range of 4–6 μm . The average particles size is smaller than 100 nm for all the samples and it is slightly larger than crystallite size determined by XRD.

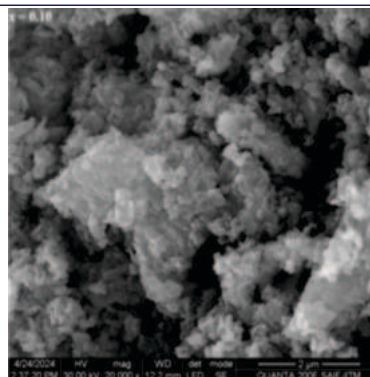
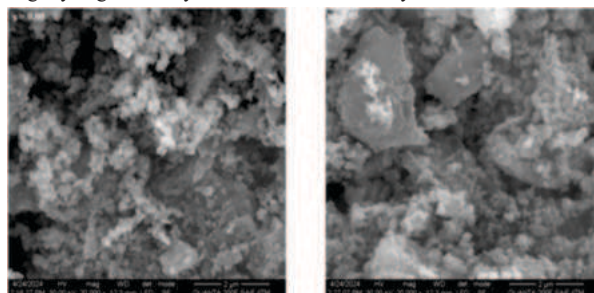


Fig. 2. SEM images of $\text{CoGa}_x\text{Fe}_{2-x}\text{O}_4$ ($x=0.00, 0.05$ and 0.10).

3.3 Magnetic Properties

In order to study magnetic nature of the material samples hysteresis measurements were carried for all the prepared material samples. The hysteresis loops for all samples are shown in fig. 6. The coercivity, saturation magnetization, magnetic remanence are shown in table 4. From table 4, it is observed that with an increase in the concentration of x (i.e., Ga^{3+} ion at Fe^{3+} -site there was a decrease in the saturation magnetization values. This decrease in the saturation magnetization is attributed to the substitution of low-magnetic Ga^{3+} ion at Fe^{3+} -site which reduces the AB and BB magnetic interactions. Further, increase in Ga^{3+} ion concentration, the saturation magnetization found to decrease. Further, the lower magnetization values for $x = 0.100$ was due to non-magnetic dopant concentration increases. The magnetic field dependence of magnetization (M-H) curves recorded at room temperature for all the compositions is shown in figure 6. The value of saturation magnetization for pure cobalt ferrite was observed to be 18.97 emu/gm which is in agreement with previous reports [12]. Continuous fall in the saturation magnetization was observed with M_s Values of 18.97 to 10.12 emu/gm respectively. For $x = 0.000$ to $x = 0.100$ which subsequently decreased for further Ga doping. The composition $x = 0.000$ exhibited higher value of saturation magnetization as compared $x = 0.100$ sample. The substitution of non-magnetic Ga^{3+} ions has been reported to exhibit similar changes in magnetization in previous literature report and it has been assigned to the site distribution of Ga^{3+} ion in the spinel structure [12-16]. Such a behavior can be explained based on the fact that the Ga^{3+} ions in spinel structure have a strong preference for the tetrahedral site. So increasing the Ga content will push the Fe^{3+} ions present on the tetrahedral site to the octahedral sites. Since Ga^{3+} ions have no net magnetic moment associated with them it will reduce the total moment associated with the A-site. However at higher concentrations Ga ions may also be force to occupy the octahedral sites which can result in a decrease of magnetization. The Coercivity of the samples was found to decrease with increasing Ga content which is an indication of decreasing anisotropy of cobalt ferrite with increase of Ga content.

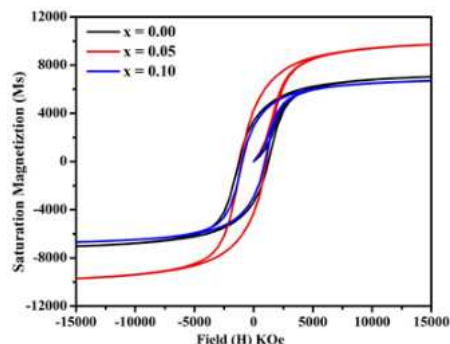


Fig.3. Vibrating sample Magnetometer of Gd doped CoFe_2O_4 nanoparticles.

Table.2. Saturation Magnetization (M_s), Remnant Magnetization (M_r), Coercivity (H_c), Squareness Ratio (M_r/M_s), Mol. W., Bohr Magnetron (nB) and Anisotropy Constant (K) for Gd doped CoFe_2O_4 .

(x)	(M_s) (emu/g)	(M_r) (emu/g)	(H_c) (KOe)	(M_r/M_s) s)	Mol. W.	(nB) (μB)	(K) (erg/cm^3)
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x = 0.00	18.97	9.07	1.244	0.4781	234.620	0.796	24.58196
				234	8	913	
x = 0.05	11.38	4.96	0.94	0.4358	235.314	0.479	11.14292
				524	7	477	
x = 0.10	10.12	4.47	1.19	0.4416	236.008	0.427	12.54458
				996	6	647	

CONCLUSION:-

Ga_xCoFe_{2-x}O₄ (x = 0.000, 0.050 and 0.100) ferrite samples were synthesized successfully by sol-gel auto combustion technique. The structural, electrical and magnetic properties of Ga substituted cobalt ferrites prepared by sol-gel auto combustion technique were studied. XRD, SEM, FTIR Structural analysis confirmed the formation of spinel phase with no impurities formed. DC electrical resistivity measurements reveal that as gallium is substituted into cobalt ferrite, the resistivity increases. The electrical resistivity decreases with increasing temperature, as predicted by the Arrhenius equation. The changes in magnetic properties could be explained based on the cation distribution of Ga ions in spinel lattice. The decreased anisotropy makes these materials promising candidates for exploring magnetostrictive behavior.

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