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Investigation on structural and magnetic properties of cobalt doped magnesium ferrite nanoparticles

J. Balavijayalakshmi[1*], T. Sudha[1], K. Karthika[1]

[1]Department of Physics, PSGR Krishnammal College for Women, Coimbatore, Tamilnadu, India.

Abstract: Nanosized spinel ferrites have attracted considerable attention for their interesting structural, magnetic and electrical properties. The physical and chemical properties of nanomaterials have been enhanced because of their surface-to-volume ratio. To search for new good gas-sensing materials and the new properties of conventional materials has become an active research field. Magnesium ferrite is a most versatile ferrite, due to its high resistivity and low eddy currents. Also the technological importance of cobalt ferrite has motivated several studies on the synthesis as well as the physical properties of this material. Hence an attempt is made to synthesize cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) by co-precipitation method. Magnesium chloride [$MgCl_2.6H_2O$], Cobaltous Chloride [$CoCl_2.6H_2O$], anhydrous Ferric Chloride [$FeCl_3$] along with sodium hydroxide [NaOH] is used as raw materials. Magnesium-Cobalt ferrite sample annealed at 600°C are subjected to X-ray diffraction to calculate the average nano-crystalline size using Debye – Scherrer formula and is found to be 2-4 nm. The FT-IR spectrum of the sample is recorded and the characteristic absorption bands are observed at 578 cm^{-1} and 406 cm^{-1} corresponding to tetrahedral and octahedral sites respectively. The morphological analysis of the sample is studied using Scanning Electron Microscope (SEM). The magnetic properties of the cobalt doped magnesium ferrite nano particles are studied using Vibrating Sample Magnetometer (VSM) and the magnetic properties are found to be improved due to cobalt doping.
Keywords: Co-precipitation method, nanosize, Debye – Scherrer, FT-IR, SEM, vsm.

Introduction

MFe_2O_4 ferrospinels (where M = Mg^{2+},Ni^{2+},$Co^{2+}$and Zn^{2+}, etc.) constitute a very important group of magnetic materials as they have a wide range of applications in devices used in telecommunication systems, computer memories, transmitting microwaves, heat transfer, etc. Among the ferro spinels, magnesium ferrite ($MgFe_2O_4$) is a partially inverse spinel and its degree of inversion is sensitive to the thermal history of the sample, microstructure and preparative parameters. It has been shown that shape of nanoparticles strongly influences the magnetic properties and the coercive force of needle-shaped particles is generally higher than that of their isometric counterparts[1-2]. Furthermore, it has been shown that specific surface area of needle-shaped nanoparticles is greater than lamellar or rod-shaped particles[3]. In the field of nano-magnetism this surface area plays very important role in governing magnetic properties. The electrical resistivity of ferrites increases with increase in doping amount. The curie temperature, magnetic moment, electrical resistivity and lattice constant are found to be affected by substitution in the spinel lattice and are due to the formation of secondary phase on grain boundaries[4-5]. In case of cobalt ferrite, the incorporation of cobalt ions results in an increase in coercivity, which is due to the coupling of the spins of cobalt and iron ions[6-7]. Also, the physical and chemical properties of ferrites are dependent upon factors such as annealing temperature, annealing time, rate of heating and rate of cooling, etc.[8]. Various methods such as ceramic method[9], sol–gel method[10-11], hydrothermal method[12], citrate method[13] and combustion method[14] are used for preparation of spinel ferrites. The present work deals with the synthesis of cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) using chemical co-precipitation and study of their structural and magnetic properties.

Experimental

Cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) are prepared by co-precipitation method. The desired composition is obtained by taking stoichiometric amounts of magnesium chloride [$MgCl_2.6H_2O$], Cobaltous Chloride [$CoCl_2.6H_2O$] and anhydrous ferric chloride [$FeCl_3$] dissolved in distilled water. The neutralization is carried out with sodium hydroxide solution and the reaction temperature is maintained at 60°C. The pH of the solution is maintained at 8 and stirred for 2hrs. The precipitate is thoroughly washed with distilled water until it is free from impurities. The product is dried at a temperature of 100°C to remove the water contents. The dried powder is mixed homogeneously and annealed at 600°C.

The synthesized sample is subjected to X-ray diffraction to calculate the average particle size using Debye – Scherrer formula. The FT-IR spectrum of this sample is recorded to ensure the presence of the metallic compounds. The magnetic properties of the cobalt doped magnesium ferrite nano particles is studied using Vibrating Sample Magnetometer (VSM).The morphological analysis of the sample is carried out using Scanning Electron Microscope (SEM).

Results and discussion

FT-IR Spectral Analysis

Infrared absorption spectrum in the range of 4000-400 cm^{-1} is recorded at room temperature by using SHIMADZU using KBr pellet method. The spectrum transmittance (%) against wave number is used for interpretation of the results. The FT-IR spectrum of the investigated cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) is shown in Figure 1.

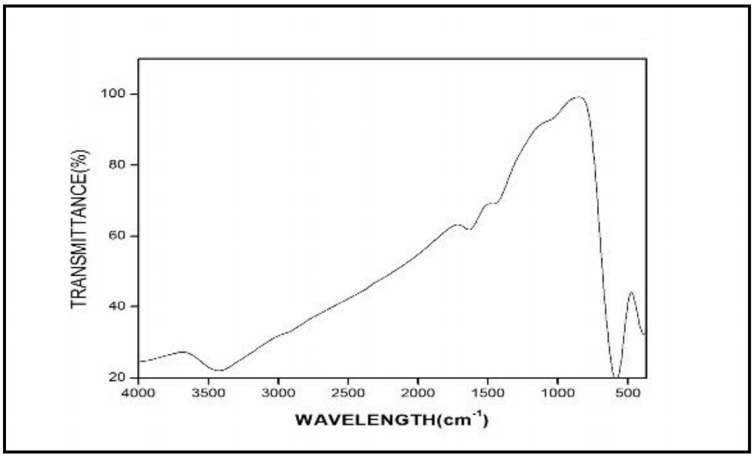

Figure 1. FT-IR spectrum of Cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) annealed at 600°C

The two main broad metal–oxygen bands are seen in the infrared spectrum of all spinels, especially ferrites. The higher absorption band observed in the range 578 cm^{-1}, is caused by the stretching vibrations of the tetrahedral metal–oxygen bond and the lowest band observed in the range 406cm^{-1}, is caused by the metal–oxygen vibrations in the octahedral sites[15-16]. The absorption band around 3560 cm^{-1} indicates the presence of hydroxyl group. The absorption peaks at 1435 cm^{-1}, 1627 cm^{-1} and 3520 cm^{-1} due to traces of adsorbed or atmospheric CO_2 [17].

3.2 Structural Analysis

X-ray diffraction pattern of cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) annealed at 600°C is shown in Figure 2. The data are collected in a 2θ range from 10° to 80° at a step size of 0.02 using Shimadzu 6000 X-ray diffractometer equipped with Cu-kα radiation of a wavelength of λ=1.5406 Å. All the main peaks are indexed with reference to the standard pattern and found to be (220), (311), (400), (442), (511) and (440)[18]. The well-defined (311) peak appears to be more intense.

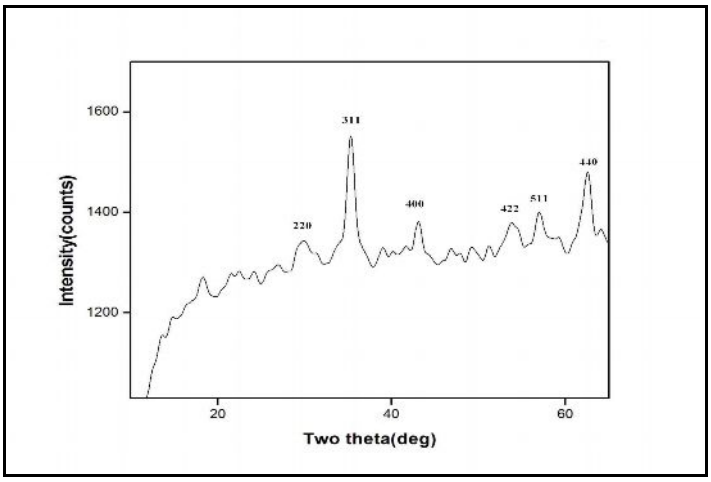

Figure 2. XRD spectrum of Cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) annealed at 600°C

All the diffractograms showed the characteristic reflections of the spinel phase. The average particle size (D) is calculated using the Scherrer formula[19]

$$D=0.9\lambda/\beta\cos\theta$$

Where λ is X-ray wavelength, β is the angular line width at half maximum intensity and θ is the Bragg's angle. The average crystallite size from X-ray technique is found to be 2-4 nm.

SEM Analysis

Figure 3 shows the surface morphology of cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) annealed at 600°C at different magnifications. From the scanning electron micrographs, it is clear that the method of synthesis of cobalt doped magnesium ferrite has resulted in uniformly distributed granular like ferrite nanoparticles.

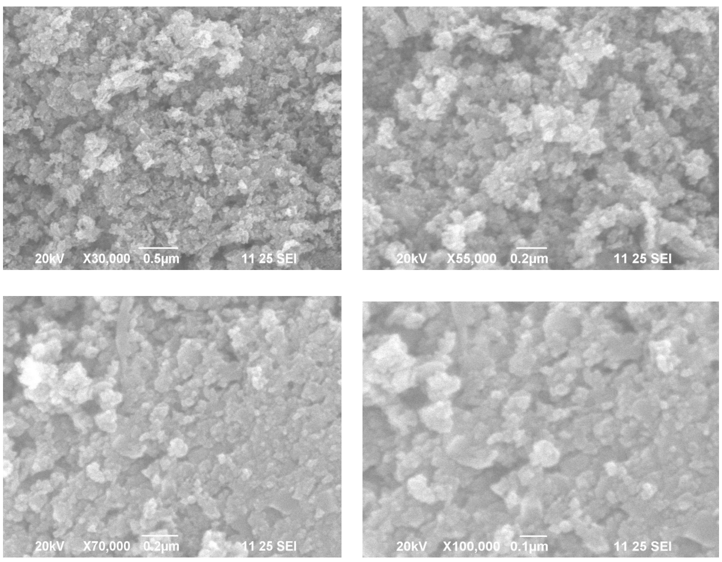

Figure 3. SEM micrographs of $Mg_{0.6}Co_{0.4}Fe_2O_4$ nanoparticles annealed at 600°C

Magnetic Measurements using VSM

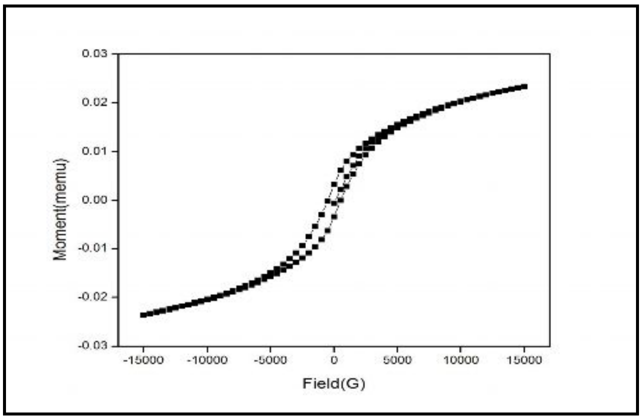

Figure 4. VSM measurements of $Mg_{0.6}Co_{0.4}Fe_2O_4$ nanoparticles annealed at 600°C

The magnetic measurements of cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) are performed using the VSM technique and the results of magnetic hysteresis at room temperature are shown in Figure 4. The value of saturation magnetization as well as coercivity and remanence are found to be 23.497E-3 emu , 486G, 3.3811 E-3 emu respectively. Smit and Wijn[20] and Kulkarni and Joshi[21] have reported M_S value for bulk particle of $MgFe_2O_4$ as 27 emu/g. Therefore the decrease in saturation magnetization can be attributed to the effect of nanoregime on it[22]. The difference in the value of M_S can be explained may be due to the change in the concentration of cation distribution. But it is found that the doping of cobalt improved the coercivity and magnetic properties of magnesium ferrite nanoparticles compared to pure magnesium ferrite nanoparticles.

Conclusion

Cobalt doped magnesium ferrite nanoparticles ($Mg_{0.6}Co_{0.4}Fe_2O_4$) annealed at 600°C are prepared by the co-precipitation method. The FT-IR spectra show main absorption bands around 578 cm^{-1} and 406 cm^{-1} corresponding to the vibration modes of the tetrahedral and octahedral sites respectively. XRD pattern reveals that the synthesized ferrites consist of nano crystalline particles with size in the range from 2 to 4nm. The SEM micrographs show uniformly distributed granular like structure. The magnetic properties of the synthesized nanoparticles are found to be improved because of cobalt doping. The synthesized materials can be tested for gas sensing properties.

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