

Influence of Annealing Temperature on $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ Nano-Ferrite



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Abstract

In this paper, we have reported the effect of annealing temperature on structural properties of $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ nano-ferrites prepared via self-combustion method without using any external oxidizing agents to change the pH of the precursors. The self-combustion method offered homogenous particle size over the composition at a lower temperature. X-ray diffraction (XRD) studies show that particle size was increased from 15nm to 59nm with increasing annealing temperature for Co-Mg nano-ferrites. The spinel ferrite functional group formation was identified by the analysis of FTIR spectroscopy data. Raman spectroscopy was used to study the structural stability of nano-ferrite.

Keywords: X-ray diffraction; Raman spectroscopy; Nano-magnetic properties; Dielectric constant

Introduction

In recent decay, there is a great interest has been taken on the experimental and theoretical research on the nano-ferrites due to their unique and unusual physical and chemical properties which is useful in various technological applications such as switching circuits, high density magnetic storage, microwave based instruments, telecommunication equipment, magnetic fluids, gas sensors, etc. [1-5]. It is established that structural, electric and magnetic properties of nano-ferrite depend on the synthesis method, pH, type and nature of mixing, sintering temperature, cation distribution between tetrahedral and octahedral sites. Although plentiful reports are available on the structural, magnetic and electric response of Mg-doped cobalt ferrites, but work related to systematic study of the effect of annealing temperature on $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ ferrites prepared via self-combustion method is still lacking. Therefore, in the present paper, we have endeavored to investigate the effect of annealing temperature on structural properties of $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ nano-ferrite.

Experimental

$\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ nano-ferrite was synthesized by the self-combustion method using a mixture of metal nitrates with citric acid. The molar ratio of nitrates to citric acid was 1:1. The stoichiometric amount of metal nitrates and citric acid were dissolved in 50ml deionized water separately, mixed and stirred

to get a homogenous solution. The homogenous solution was continually stirred with a hot plate at 100 °C to get the liquid gel. The liquid gel was heated on a hot plate until it burnt out completely in the powder form. The obtained powder was grounded and sintered at different temperature i.e. 200, 400, 600, 800 and 1000 °C for 6h. The phase formation and structural properties were carried out using Rigaku MiniFlex X-ray Diffractometer with Cu-K α radiation. The XRD patterns were measured over the 2 θ range from 20-80° with a step size of 0.02°. Fourier transform infrared (FTIR) spectra were recorded in the frequency range 400-4000cm⁻¹ using PerkinElmer spectrometer. The Raman spectra of these ferrites were observed using STR-500 Confocal micro-Raman spectrometer.

Results and Discussion

The XRD pattern of $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$ nano-ferrites annealed at different temperature i.e. 200, 400, 600, 800 and 1000 °C are shown in Figure 1. XRD pattern analysis shows that all the ferrites have single phase cubic spinel structure without any impurity peak belonging to the space group Fd3m. All the characteristic peaks are in good agreement with the standard JCPDS card No 22-1086. Insets of Figure 1 show that 2 θ shifts towards the lower angle side as increasing the annealing temperature for both the ferrites. As annealing temperature increases the intensity of (311) peak become higher while peak broadening becomes narrower,

which indicates an increase in the crystallinity and particle size. The average particle size (D) of these ferrites is calculated using the Debye-Scherrer's formula. The specific surface area was calculated assuming all the particles in spherical form using the Sauter formula. The crystallite size, specific surface area,

lattice constant, X-ray density and micro strain of all the samples are listed in Table 1. The variation in crystalline size and X-ray density as a function of annealed temperature are shown in Figure 2. The XRD results indicate the formation of crystallinity with increasing the calcination temperature.

Table 1: Variation in XRD parameters with temperature.

Parameters	Temperature (°C)				
	200	400	600	800	1000
d (Å)	2.5221	2.5235	2.5245	2.5259	2.5276
a (Å)	8.365	8.3695	8.3727	8.3773	8.3832
D (nm)	15	17	31	47	59
Strain $\times 10^{-3}$	2.4	2.11	1.18	0.77	0.62
density	4.8534	4.8455	4.84	4.8321	4.8218

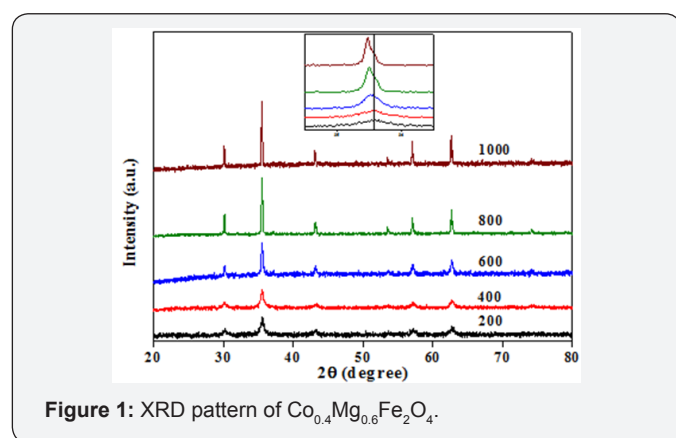


Figure 1: XRD pattern of $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$.

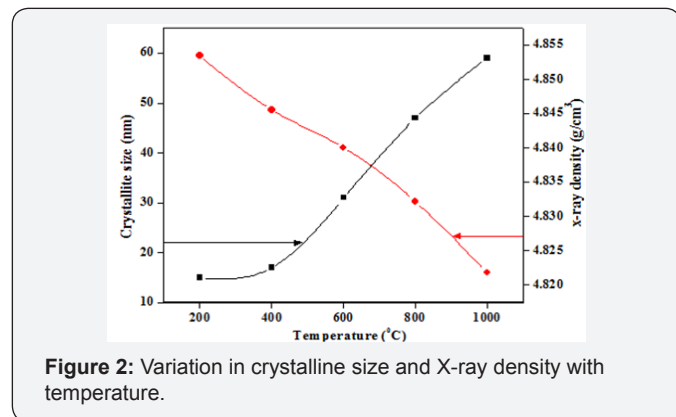


Figure 2: Variation in crystalline size and X-ray density with temperature.

The formation of cubic phase spinel structure in cobalt ferrites was confirmed by FTIR spectra. (Figure 3a) shows the IR spectra in the wave number range of 4000-400 cm^{-1} . The FTIR spectrum shows that in ferrites, the metal ions occupy two different interstitial sites in the lattice; one is at the tetrahedral site while the other is at the octahedral site according to the configuration of the oxygen nearest neighbors in fcc structure [6]. The two principle absorption band in the range of 600-400 cm^{-1} are observed in IR spectra where the high-frequency band observed between 580-578 cm^{-1} for all samples has been attributed to stretching vibrations of tetrahedral metal-oxygen

bonding while the lower frequency band observed in between the frequency range 424-407 cm^{-1} due to the octahedral metal-oxygen vibrations. A very weak band near 1380 cm^{-1} is found due to the nitrate group which remained as a residue in the sample while bands observed near 1600 cm^{-1} correspond to the stretching and bending modes of H-O-H due to the moisture or absorbed water during sample preparation process.

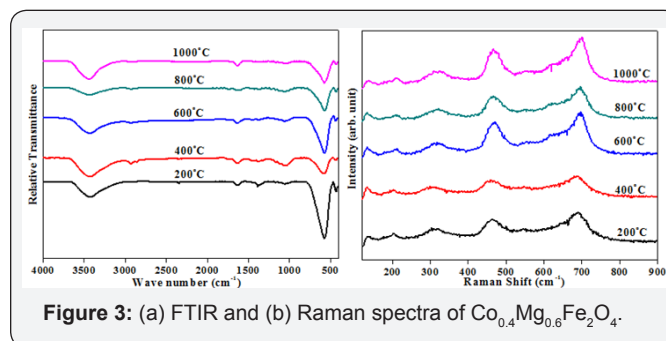


Figure 3: (a) FTIR and (b) Raman spectra of $\text{Co}_{0.4}\text{Mg}_{0.6}\text{Fe}_2\text{O}_4$.

The Raman spectroscopy technique is a powerful and sensitive tool to gain insight into the vibrational energy states and structural information of the compound. Figure 3(b) shows the Raman spectra in the wave number range of 180-900 cm^{-1} . Factor group analysis predicts that a cubic phase inverse spinel structure with space group $Fd-3m$ having five Raman active modes namely $A_{1g} + E_g + 3T_{2g}$. In these active modes, A_{1g} mode is due to the symmetric stretching of metal oxygen (Co-O and Fe-O) bonds along the tetrahedral site, while the E_g mode is a result of the symmetric bending of an oxygen atom with the metal ions [7,8]. The $T_{2g}(2)$ modes rise due to the asymmetric stretching of metal-oxygen bonds (Co/Fe-O) along the octahedral site, while $T_{2g}(3)$ modes are due to asymmetric bending of metal-oxygen bonds of the octahedral site. The $T_{2g}(1)$ mode is attributed due to the translational movement of the metal ion at the tetrahedral site together with four oxygen atoms. The active modes of Raman spectra in between the region 660-720 cm^{-1} is indicative of tetrahedral site, while modes in between 460-640 cm^{-1} are indicative of the octahedral site [8]. The ionic radii of cobalt and iron are different which occur a considerable difference in

Co/Fe-O bond length. This distribution in bond length creates a doublet like peak where one peak corresponds to the cobalt ions at octahedral site, while another peak represents the occupancies of cobalt ions both octahedral as well as tetrahedral sites. This doublet like behavior of Raman spectra shows the mixed spinel structure of cobalt ferrites, which is a confirmation of XRD and FTIR results. Thus it is found that structure remains identical, while XRD parameters alteration with increasing annealing temperature.

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