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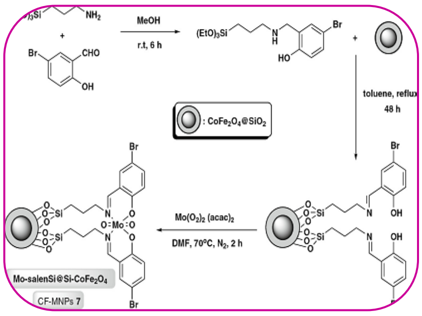
REVIEW OF RESEARCH

EFFECT OF ANNEALING TEMPERATURE ON THE STRUCTURAL PROPERTIES OF COBALT FERRITE NANOPARTICLES



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ABSTRACT

Cobalt ferrite nanoparticles with average particle size of the order of 32 to 48 nm were prepared by sol-gel auto-combustion technique using high purity metal nitrates and L-ascorbic acid as a fuel. X-ray diffraction, scanning electron microscopy and infra-red spectroscopy techniques were employed to characterize the prepared cobalt ferrite nanoparticles. A careful examination of the XRD pattern shows that the intensity of Bragg's reflection increases with increase in annealing temperature. The crystallite size, lattice constant, X-ray density, porosity etc structural parameters were evaluated and their behavior as a function of annealing temperature is reported in this work. The X-ray density decreases with increase in annealing temperature while as the crystalline size increases with increase in temperature. The IR spectra of all the samples were recorded in the wave number of 350 – 1000 cm^{-1} at room temperature.

KEYWORDS: *Crystalline size, X-ray density, infrared spectra.*

INTRODUCTION:

Nanocrystalline materials are of great importance in the recent years from the research and technological application point of view due to their interesting physical and chemical properties which are different than that of bulk counter parts [1, 2]. Nanocrystalline materials exhibit unique crystallo-graphical structure e.g., high surface area and high volume fraction of atoms at interfacial regions. The resulting properties namely super-paramagnetism, super-plasticity, catalatic activity etc are distinctly different from those of their micrometer sized counterparts [3]. The physical and chemical properties are greatly influenced by the synthesis root and various synthesis parameters. Metal oxide nanoparticles in particular spinel ferrites are of current focus because of their interesting, unique optical, electronic, mechanical, structural and magnetic properties and have large number of promising technological applications in high density recording media, ferrofluids, drug delivery, magnetic refrigerators, high frequency devices [4,5] etc.

Cobalt ferrite shows the good magnetostrictive properties, magnetocrystalline anisotropy, high coercivity and moderate saturation magnetization among all the ferrite family [6]. The magnetoelastic properties of Co^{2+} in spinel ferrites are most commonly seen in magnetocrystalline anisotropy and relaxation effects. In general, Co^{2+} ions are stabilized in octahedral sites which give rise to a degenerate or near-degenerate orbital ground state. As a result, the strong spin-lattice interaction that arises from the unquenched orbital angular momentum has served to explain the high spin wave line widths [7]. In the present paper we report the effect of sintering temperature on the structural properties of cobalt ferrite nanoparticles synthesized via sol-gel technique and sintered at three different temperatures.

EXPERIMENTAL:

In the present investigation, cobalt ferrite nanoparticles were prepared by sol-gel auto-combustion method relatively at low temperature using L-Ascorbic acid as a fuel and maintaining metal nitrates to fuel ratio as 1:3. The prepared powder of cobalt ferrite was annealed at different temperatures viz 600°C , 800°C and 1000°C to understand the effect of varying annealing temperature on the structural properties of cobalt ferrite nanoparticles investigated by X-ray diffraction, scanning electron microscope, and infra-red spectroscopy techniques. The X-ray diffraction technique was employed to confirm the phase purity and nano crystalline nature of the prepared cobalt ferrite nanoparticles. The X-ray diffraction pattern was recorded into 2θ range of 20^0-80^0 at room temperature using Cu-Kα radiation. The surface morphological studies were carried out using scanning electron microscopy technique. Infra-red spectra of all the samples were taken at room temperature in the wave number 1000- 350cm^{-1}.

RESULTS AND DISCUSSION:

1. X-ray diffraction:

Figure 1(a, b, c) represents the X-ray diffraction (XRD) pattern of cobalt ferrite samples annealed at temperatures namely CF6, CF8 and CF10 respectively. All the XRD patterns exhibit similar kind of nature except the peak intensity and broadening. The reflections present in the XRD pattern belongs to cubic spinel structure. The analysis of XRD pattern was made through computer program and it indicates that all the samples possess single phase cubic spinel structure with no extra peaks.

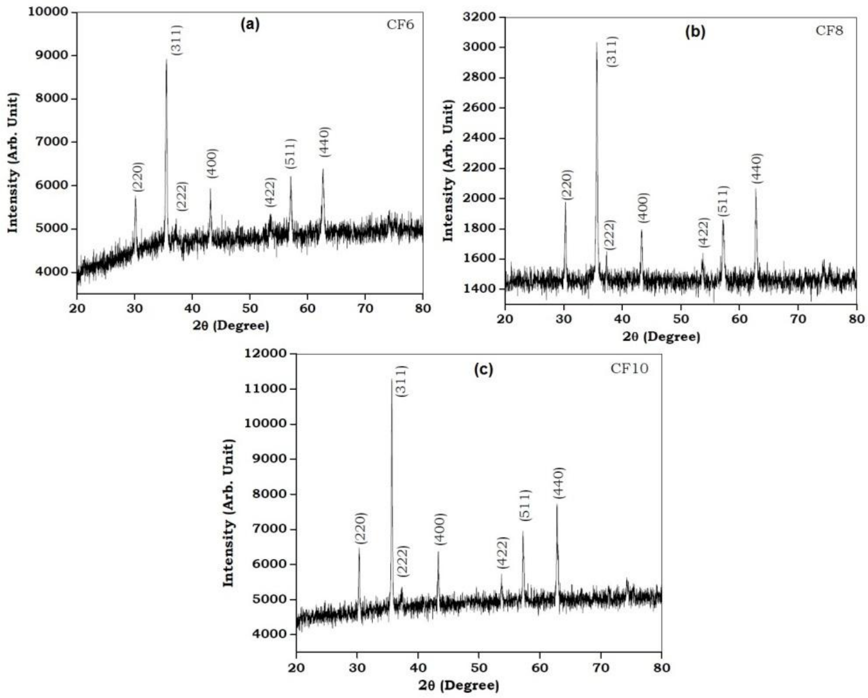

Fig. 1: XRD Patterns of cobalt ferrite nano-particles prepared by sol-gel auto-combustion method using L-Ascorbic Acid as a fuel sintered at a) 600^0C, b) 800^0C and c) 1000^0C

A careful examination of the XRD pattern shows that the intensity of Bragg's reflection increases with increase in annealing temperature. Besides that the broadening of the most intense peak (311) of the

XRD pattern slightly decreases with increasing annealing temperature. The increase in broadening, intensity and sharpness is attributed to increasing annealing temperature. Using XRD data the lattice constant (a) was determined for all the samples (CF6, CF8 and CF10) using the standard relation for cubic symmetry given by

$$a = d\sqrt{N} \quad (1)$$

where, notations have their usual meaning. The values of lattice constant are presented in Table 1. It can be observed from Table that, the lattice constant increases as annealing temperature increases. Similar behavior of lattice constant was reported in the literature [8]. The X-ray density d_x was determined for all the samples under investigation using the following relation,

$$d_X = \frac{ZM}{NV} \quad (2)$$

where, Z = 8 for cubic spinel ferrite, M is molecular weight, N Avogadro's Number and V is volume of the unit cell. The values of X-ray density are presented in Table 6.1. It is found from table that X-ray density decreases with increase in annealing temperature. The decrease in X-ray density is attributed to increasing unit cell volume due to increase in lattice constant. The bulk density (d_B) was determined through Archimedes Principle and their values are reported in the Table 1. It is evident from table that bulk density decreases with increasing temperature. Due to increase in temperature lattice constant increases and hence unit cell volume also increases. The increase in volume overtakes increase in mass and hence bulk density decreases. The porosity (P) of the samples was determined through the values of X-ray density and bulk density. Table 1 shows the values of percentage porosity for all the samples under investigation. The porosity decreases with increase in annealing temperature. The highest porosity of 34% is observed for the sample CF6 may be due to more agglomeration. The lowest density of 29% is observed for the samples CF10 may be due to higher annealing temperature causing the reduction in number of pores. The crystallite size (t) of all the samples was calculated using Scherer's formula [9], for which the most intense peak (311) was considered. The values of crystallite size are presented in Table 1. It is evident from Table 1 that the crystallite size increases as annealing temperature increases. Thus, the increase in annealing temperature results in increase in crystallite size their by affecting the structural properties of the cobalt ferrite.

Table 1: Lattice constant (a), X-ray density (dx), Bulk density (dB), Porosity (P),Crystallite size (t), grain size (G) and absorption bands (υ_1) for cobalt ferrite nanoparticles as function of annealing temperature

Sample	'a' (Å)	'd_X' (gm/cc)	'd_B' (gm/cc)	'P' (%)	't' (nm)	'G' (nm)	'ν_1' (cm^{-1})
CF6	8.357	5.365	3.561	34	33	76.2	566.43
CF8	8.382	5.293	3.654	31	42	80.2	547.79
CF10	8.388	5.281	3.773	29	48	82.3	536.81

2. Scanning Electrron Microscopy:

The microstructure and surface morphology of the present samples was studied using scanning electron microscopy (SEM) technique. Figure 2 shows the SEM images of the samples CF6, CF8 and CF10. The analysis of SEM image shows that the microstructures of the nanoparticles were almost regular in shape and dispersed uniformly. The agglomeration of particles at 600 °C that is for sample CF6 is more as compared to CF8 and CF10. Using the SEM image the grain size (G) was calculated using linear intercept method. Table 1 provides the values of grain size as a function of annealing temperature. It is observed from Table 1 that, the grain size of all the samples is in nanometer range and increases with increasing annealing temperature. Our results on scanning electron microscopy technique are in good agreement with the literature reports [10]. Thus, the nanocrystalline nature of the samples was confirmed through grain size values.

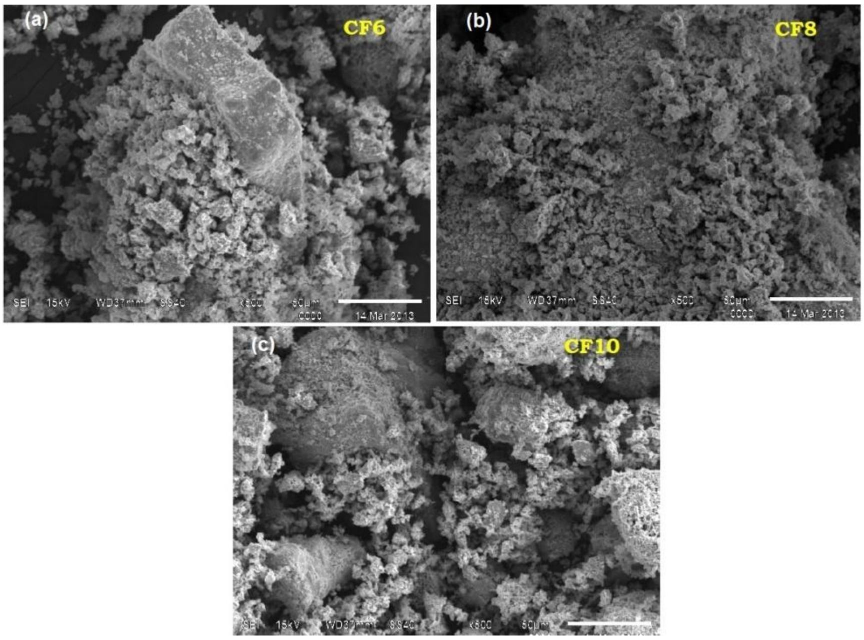

Fig. 2: Scanning electron micrograph (SEM) of cobalt ferrite nano-particles prepared by sol-gel auto-combustion method a) 600^{0}C, b) 800^{0}C and c) 1000^{0}C.

3. Infrared spectra:

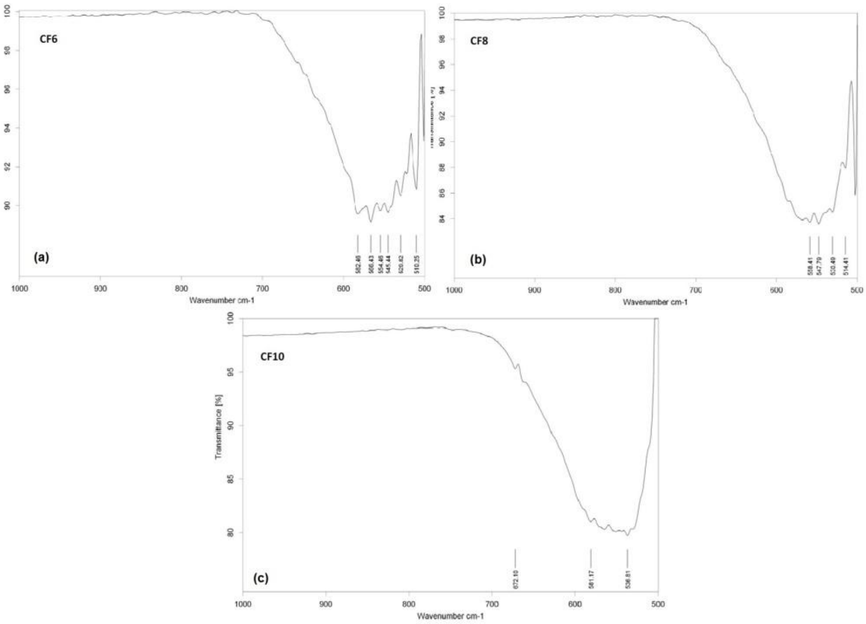

Fig. 3: Infrared spectra of cobalt ferrite nano-particles prepared by sol-gel auto-combustion method using L-ascorbic acid as a fuel annealed at a) 600^{0}C and b) 800^{0}C and c) 1000^{0}C.

The IR spectra of all the samples were recorded in the wave number of 350 – 1000 cm^{-1} at room temperature and is shown in Figure 3 (a,b,c) The spectra show one broad absorption band near 600 cm^{-1}, exhibiting the characteristics feature of spinel ferrite. Actually the spectra should show two bands one near 400 cm^{-1} and other at 600 cm^{-1}. In the present case the spectra range starts from 500 cm^{-1} onwards, hence one absorption band near 400cm^{-1} is invisible. The IR spectra of the present samples are of similar nature to that reported in the literature [11]. The value of high frequency absorption bands υ_1 is presented in Table 1. It is observed from spectra that the absorption band shift towards lower wave number due to increase in annealing temperature.

CONCLUSIONS:

Cobalt ferrite nanoparticles were successfully synthesized using sol-gel auto-combustion method taking L-ascorbic acid as a fuel. The characterization of all the samples annealed at different temperature was carried out using X-ray diffraction, scanning electron microscope and infra-red spectroscope techniques, which confirms the nanocrystalline nature of the studied samples. The lattice constant, crystallite size, grain size increases with increase in annealing temperature.

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