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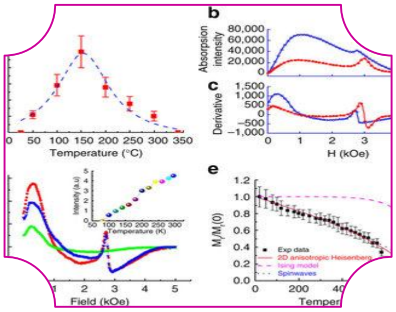
COBALT FERRITE NANOPARTICLES: INVESTIGATIONS ON MAGNETIC AND ELECTRIC PROPERTIES AT DIFFERENT ANNEALING TEMPERATURES



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ABSTRACT

Nanoparticles of cobalt ferrite were obtained by using L-ascorbic acid and annealed at three different temperatures 600^0*C, 800*^{0}*C and 1000*^{0}*C in order to study the effect of sintering temperature on magnetic and electrical properties. The magnetic properties such as saturation magnetization, coercivity and remnant magnetization were studied at room temperature by using hysteresis curves. The saturation magnetization decreases with increase in sintering temperature while as the magneton number shows increasing trend. Two probe technique was used to study the electrical properties of cobalt ferrite as a function of temperature within the temperature range 300K to 850K. The dielectric constant shows maximum value at high frequency and minimum values at low frequency. The decrease in dielectric constant is exponential in nature.*

KEYWORDS: *sintering temperature, magnetization, dielectric constant.*

INTRODUCTION:

In the family of spinel ferrite, cobalt ferrite ($CoFe_2O_4$) with inverse spinel structure has been widely studied [1, 2] as they display attractive magnetic properties as well as electrical and other properties making them useful in wide range of applications. Cobalt ferrite possess high Curie temperature, high magnetocrystalline anisotropy, high permeability, high coercivity, high saturation magnetization, good magnetic mechanical properties, excellent chemical stability etc and therefore is subject of interest of researchers. The compositional and micro-structural properties are sensitive to the preparation method used for their synthesis. The magnetic nanoparticles of cobalt ferrite cab be obtained by variety of methods including sol-gel [3], hydrothermal [4], co-precipitation [5], microemulsion [6] etc. The magnetic nanoparticles with a higher surface area to volume ratio provide higher sensitivity, better targeting and improvement of the colloidal stability of the nanostructures [7]. Sol-gel auto-combustion synthesis method is an easy and convenient

method for obtaining nanoparticles of spinel ferrite. Sol-gel technique offer enhanced controlled over homogeneity, elemental composition and powder morphology. Like synthesis methods, synthesis parameters and synthesis conditions also strongly influences the physical and chemical properties of spinel ferrite nanoparticles. In order to obtain materials with desired properties, it is necessary to obtain high density powder with small and uniform grain size and controlled stoichiometry. This can be achieved by using sol-gel synthesis and annealing the obtained as prepared powder at suitable temperature.

In the literature, there are reports on the effect of fuel additives [8], chelating agent [9], and aging time [10] on structural, microstructural and magnetic properties of cobalt ferrite nanoparticles. The influence of heat treatment on cobalt ferrite ceramic powder was reported by Juliana B. Silva et.al [11]. The effect of variation of annealing temperature on the structure, morphology, magnetic, electric and dielectric properties of cobalt ferrite nanoparticles is not systematically reported in the literature to our knowledge.

EXPERIMENTAL:

The nano-powders of cobalt ferrite were synthesized by well known sol-gel method using metal nitrates of respective ions and L-Ascorbic acid, as a fuel. Amounts of Co $(NO_3)_2$, Fe $(NO_3)_3$ with molar ratio Co^{2+}/Fe^{3+} = ½ were dissolved completely in de-ionized water. The aqueous solution containing Co^{2+} and Fe^{3+} ions was poured intoL-ascorbic acid with the total cations/L-Ascorbic acid molar ratio 1:3 and the initial pH of the solution was measured. To increase the pH up to 7, ammonia hydroxide in aqueous form was added to the mixed solution drop by drop. The mixture was stirred using magnetic stirrer and evaporated at 80^0C to form a gel. The temperature of the gel was further increased to 110^0C for 1-2 hours. The gel burns rapidly and turned into brown loose powder. The obtained powder was annealed at 600^0C, 800^0C and 1000^0C and was used for further characterization. Cobalt ferrite nano-particles synthesized by sol-gel auto-combustion method at 600^0C, 800^0C and 1000^0C were named as CF6, CF8 and CF10 respectively.

The magnetic measurements were recorded at room temperature using pulse field hysteresis loop technique. The DC electrical resistivity measurements were carried out in the temperature range 300-600K using two probe techniques. A silver paste was applied on the surfaces of pellet to ensure the good ohmic contact. The dielectric constant measurements were carried out at room temperature as a function of frequency (100Hz – 1MHz) using LCR-Q meter (Model 4192, HP make).

RESULTS AND DISCUSSION:

1. Magnetic properties:

The magnetic properties tested by pulse field hysteresis loop technique at room temperature for different produced cobalt ferrite annealed samples show strong influence of annealing temperature. The magnetization (M) versus applied magnetic field (H) that is M-H plots for all the samples are shown in Figure 1. The plot indicates the normal hysteresis loop and is used to evaluate the values of saturation magnetization (M_S), remenance magnetization (M_r) and coercivity (H_C). Table 1 illustrates the values of all these magnetic parameters for different annealing temperature. It can be noticed from Table 1 that the saturation magnetization increases whereas coercivity and remanence magnetization decreases with increasing annealing temperature. The increase in annealing temperature increases the crystallite size of the samples, which results in increasing the saturation magnetization and decreasing remanance magnetization and coercivity.

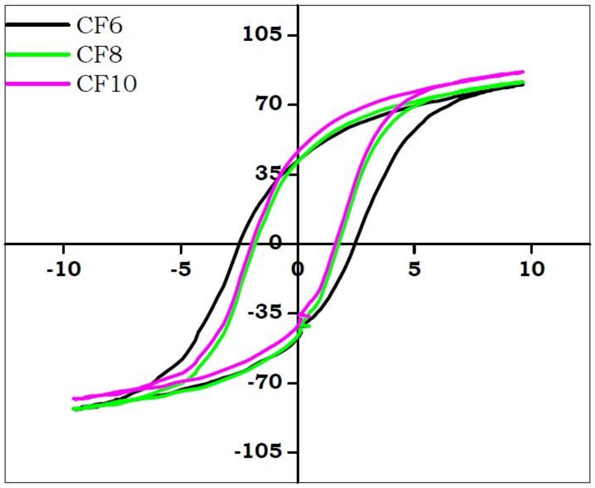

Fig.1:M-H plots of cobalt ferrite nano-particles prepared by sol-gel auto-combustion technique.

The magneton number (n_B) (the saturation magnetization per formula unit in μ_B) was calculated for all the samples using the following relation.

$$n_B^A = \frac{M_S \times \text{Molecular weight}}{5585} \qquad (1)$$

The observed values of magneton number are listed in Table 1 as a function of annealing temperature. It can be observed from table that the magneton number increases with increasing annealing temperature. Similar results of magnetic properties are reported in the literature [12, 13].

Table 1: Saturation magnetization (M_S), Remenance Magnetization (M_r), Coercivity (H_C) and Magneton number (n_B), Room temperature resistivity (ρ_0) and Activation energies (ΔE), for cobalt ferrite prepared by sol-gel auto-combustion technique as function of annealing temperature.

Sample	M_S (emu/gm)	M_r (emu/gm)	M_r/M_S	H_C	n_B	$\rho_0\times10^9$ (ohm-m)	ΔE (eV)
CF6	80.21	69.27	0.864	2444	3.37	1.70	0.806
CF8	81.62	68.37	0.838	1069	3.42	0.65	0.769
CF10	86.22	66.22	0.768	998	3.62	0.50	0.662

2. *D.C. Electrical Resistivity:*

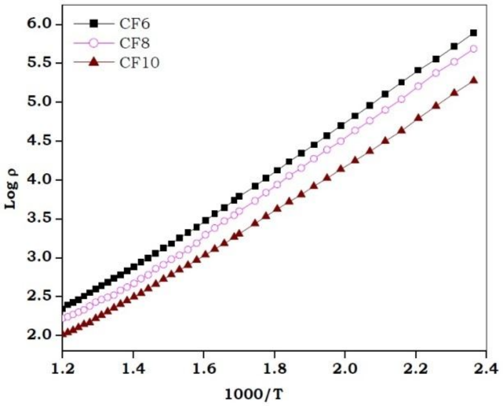

Fig. 2: Variation of DC electrical resistivity with temperature of cobalt ferrite nanoparticles prepared by using sol-gel auto-combustion technique

The d.c. electrical resistivity of all the samples was obtained using two probe techniques in the temperature range 300K to 850K. The plot of logarithm of resistivity versus reciprocal of temperature for all the samples is shown in Figure 2. It is evident from figure that as temperature increases resistivity decreases exhibiting the semiconducting behavior of the samples. The resistivity plot shows anomalous behavior. The resistivity at room temperature decreases with increase in annealing temperature decreases as noticed from the Table 1. The activation energy was calculated using resistivity plots and the Arrhenius relation [14]. The values of activation energy are presented in Table 1. It is observed from the values of activation energy that as annealing temperature increases activation energy decreases.

3. *Dielectric Constant (ε'):*

The dielectric constant (ε') and dielectric loss tangent (tan δ) for the present samples were studied as a function of frequency. Figure 3shows the frequency dependence of the dielectric constant at room temperature for the studied samples. The Figure depicts that all the samples exhibit dielectric dispersion. At low frequency the dielectric constant shows maximum value and at high frequency the dielectric constant shows minimum value. The decrease in dielectric constant is exponential in nature. The dielectric behavior of the present samples can be explained on the basis of Koop's model [15] and Maxwell's-Wagner polarization theory [16, 17].

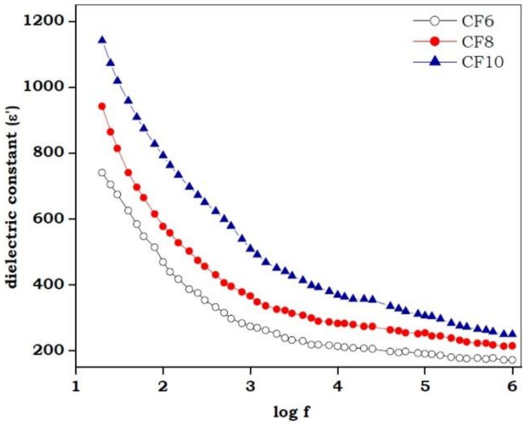

Fig. 3: Variation of dielectric constant (ε') with frequency of cobalt ferrite nanoparticles prepared by sol-gel auto-combustion technique

The values of dielectric constant measured at different frequency for varying annealing temperature are presented in Table 2. It can be observed from this table and Figure 3 that the dielectric constant increases as annealing temperature increases. The increase in dielectric constant is attributed to increase in grain boundaries due to increase in annealing temperature. The behavior of dielectric constant of the present samples is in confirmation with the reported data [18].

Table 2: Room temperature dielectric constant (ε'), dielectric loss (ε") and dielectric loss tangent (tan δ) for cobalt ferrite prepared by sol-gel auto-combustion technique as function of annealing temperature

Sample	f = 100Hz			f = 1MHz		
	(ε')	(ε'')	(tan δ)	(ε')	(ε'')	(tan δ)
CF6	704	2816	4	171	3.42	0.02
CF8	864	7162	8.29	214	128.4	0.60
CF10	934	9377	13.32	249	311.25	1.25

4. Dielectric Loss Tangent (tan δ):

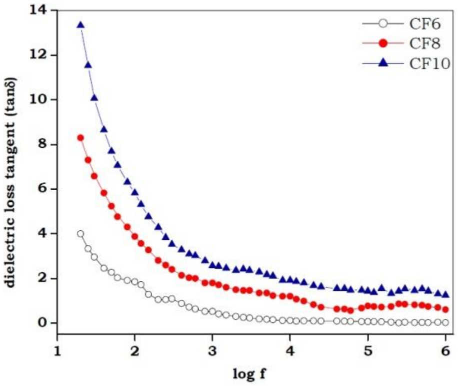

Fig. 4: Variation of dielectric loss tangent (tan δ) with frequency of cobalt ferrite nanoparticles prepared by sol-gel auto-combustion technique

Figure 4 displays the variation of dielectric loss tangent as a function of applied frequency. It can be seen from this figure that the dielectric loss tangent also decreases with increase in frequency in same fashion to that of dielectric constant. The maximum dielectric loss tangent is observed at lower frequency and minimum dielectric loss tangent is observed at higher frequency. The values of dielectric loss tangent tan δ measured at different frequency increases with increase in annealing temperature. Due to increase in annealing temperature the crystallite size of the cobalt ferrite sample decreases which results in increasing the dielectric constant of the studied samples. The behavior of the dielectric loss tangent as a function of frequency of the present sample is similar to that reported in the literature [19].

CONCLUSIONS:

The saturation magnetization increases while the coercivity decreases with increase in annealing temperature. The magneton number also increases with increasing temperature. The resistivity plot displays semiconducting nature for all the samples under investigation. The activation energy decreases with increase in annealing temperature. The dielectric constant and dielectric loss tangent both decreases exponentially with increase in applied frequency. The values of dielectric constant and dielectric loss tangent both shows increasing trend with respect to annealing temperature.

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