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EFFECT OF SINTERING TEMPERATURE ON THE STRUCTURAL PROPERTIES OF NANOSIZE COBALT FERRITE

Dr. G. H. Kale[a] and Dr. P. K. Gaikwad[b]

[a] Dept. of Electronics, Yeshawantrao Chavan Mahavidhyalaya, Tuljapur. (M.S.) India - 413601

[b]Dept. of Physics, Shri Chhatrapati Shivaji College, Omerga. (M.S.) India - 413606

Abstract: $CoFe_2O_4$ nano practical's synthesized by sol-gel technique at low temperature using L-ascorbic acid a fuel. The metal fuel ratio was taken as 1:3, where as pH of the sample was maintained at 7. The obtained powder was annealed at 600^0C and 800^0C temperature. X-ray diffraction technique was employed to confirm the phase purity and nano crystalline nature and lattice constant, practical size, grain size increases where as porosity decreases with increases in sintering temperature.

Keywords : Cobalt ferrites; Sintering temperature; Structural Properties.

1. Introduction:

Nanomaterials have extremely small size which having at least one-dimension 100nm or less. Nanomaterials with different size having the same composition can exhibit different properties. The size of the particles when reduced from micrometer level to nano-meter level, the properties of the material suddenly change and may be useful for certain applications [1]. In spinel-type Cobalt ferrite is well known to have a large magneto crystalline anisotropy, high coercivity, moderate saturation magnetization, high chemical stability and high mechanical hardness [2, 3]. So far, the fabrication of nanoscale cobalt ferrite powders has attracted much attention due to their exceptional physical properties. 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 and 800°C to understand the effect of varying annealing temperature on the structural properties was examined in this work [4, 5].

2. Experimental details:

Sol-gel technique was used for preparation of cobalt ferrite nanoparticles used high purity AR grade cobalt nitrate, ferric nitrate, L-ascorbic acid and ammonia. The appropriate amounts of metal nitrate to L-ascorbic acid were first dissolved in deionized water to get the clear solution. The molar ratio of metal nitrate to L-ascorbic acid was taken as 1:3 and 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 80^0c to form a gel. This gel was dried at 110^0c, which self ignites to form a fluffy product. This is then ground to form fine powder of cobalt ferrite. The obtained powder was annealed at 600^0c (CF6) and 800^0c (CF8).

3. Result and discussion:

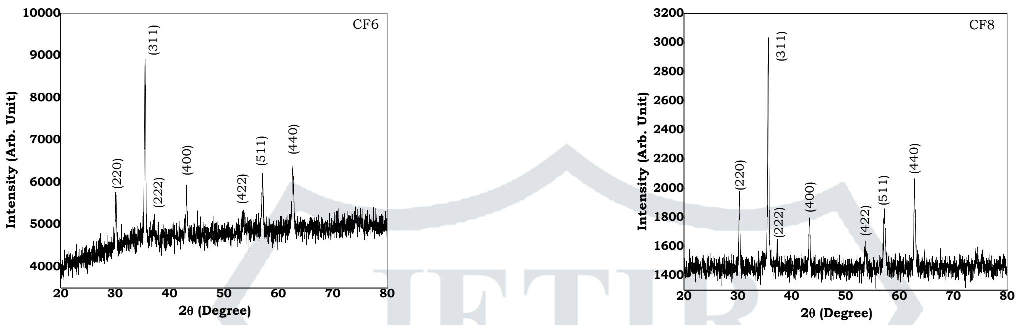

Fig 1: XRD Patterns of cobalt ferrite nano-particles prepared by sol-gel autocombustion technique using L-Ascorbic Acid as a fuel sintered at 600^0C (CF6) and 800^0C (CF8).

Fig 1 shows the XRD patterns of cobalt ferrite sintered at various temperatures viz 600^0c (CF6) and 800^0c (CF8). The X-ray diffraction pattern shows the presence of desired phase of cubic spinel structure; no extra peak of impurity phase was observed in the XRD pattern, confirming the purity of the samples. The reflections (220), (311), (400), (511), (440) belonging to single phase cubic spinel structure are present in the XRD pattern. Using the XRD data the structural properties of the samples was studied. The lattice constant 'a' is calculated using this equation ($a = \frac{\lambda[h^2 + k^2 + l^2]^{1/2}}{2\sin\theta}$). The value of lattice parameter for cobalt ferrite samples was computed using 'd' spacing values and the respective hkl parameters as shown in Fig 2. The lattice parameter 'a' increases with increase in sintering temperature [6] and 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 as shown and the X-ray density d_x is shown in Fig 2.

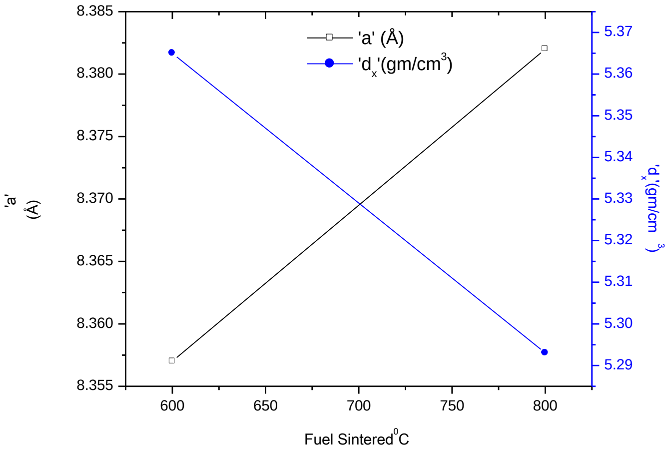

Fig 2: Lattice constant (a) and X-ray density (d_x) of cobalt ferrite nano-particles prepared by sol-gel auto-combustion technique using L-Ascorbic Acid as a fuel sintered at 600^{0}C (CF6) and 800^{0}C (CF8).

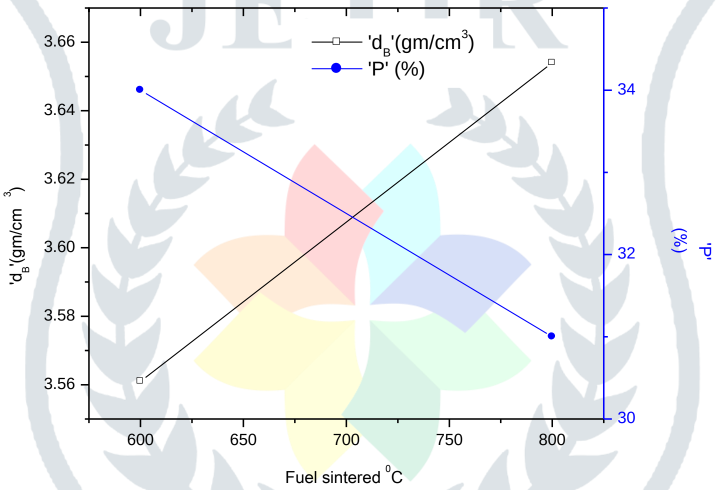

Fig 3: Bulk density (d_B), Porosity (P) of cobalt ferrite nano-particles prepared by sol-gel auto-combustion technique using L-Ascorbic Acid as a fuel sintered at 600^{0}C (CF6) and 800^{0}C (CF8).

This is due to increase in particle size. The porosity of the samples was calculated by using the relation $\left(\%P = (1-\frac{d_b}{d_x})\times 100\right)$ where the bulk density d_b found to increases with increasing sintering temperature is bulk density. As shown in Fig 3. The porosity of the samples decreases with increase in sintering temperature. This is due to increase in bulk density and decrease in x-ray density. Fig 3 shows that the bulk density of the samples was measured by using Archimedes principle [7] and the Bulk density decreases (d_B) of the sample 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 decreases with increase in annealing temperature.

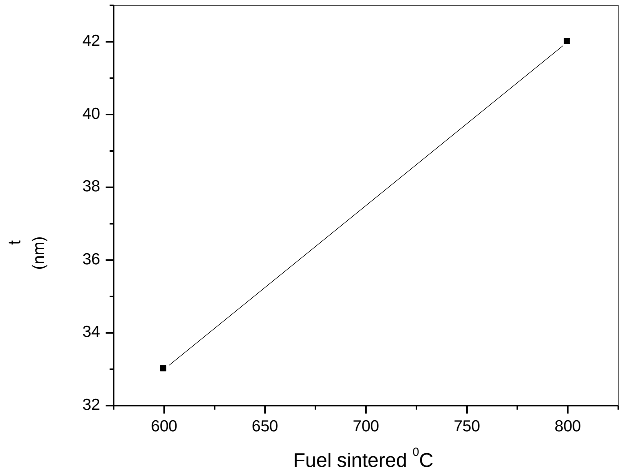

Fig 4: Crystallite size (t) of cobalt ferrite nano-particles prepared by sol-gel auto-combustion technique using L-Ascorbic Acid as a fuel sintered at 600^{0}C (CF6) and 800^{0}C (CF8).

The crystallite size (t) of all the samples was calculated using Scherer's formula $\left(t=\frac{0.9\lambda}{\beta\cos\theta}\right)$ [8]. Where 't' is the particle size, 'λ' is the wavelength of Cukα, 'β' is full width of half maxima (FWHM) and 'θ' is the Bragg's angle for (311) peaks. The reflection (311) is found to be more intense and is used to determine the crystallite size and it is observed form Fig 4 that the sintering temperature increases the particle size is also increases [9] as shown in Fig 4. Thus, the increase in annealing temperature results in increase in crystallite size their by affecting the structural properties of the cobalt ferrite.

Conclusion:

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 analysis revealed the prepared samples are single phase cubic spinel structure. Cobalt ferrite samples at two different sintering temperatures were successfully prepared by sol-gel technique and the samples show strong sintering temperature dependency. It is found that the lattice constant, bulk density and particle size increases where as x-ray density and porosity decrease with increases in sintering temperature.

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