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Research Paper

Annealing Induced Coercivity in Cobalt-ferrite Nanoparticles Prepared by Coprecipitation Method

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Abstract— Cobalt-ferrite magnetic nanoparticles of particle size/crystallite size of about 50 nm were prepared by the coprecipitation method. Structural analysis of the particles was done by XRD measurement and the magnetic hysteresis measurements were performed by a VSM. All the measurements were performed at room temperature. The crystallite size of particles was estimated by using Debye Scherrer's formula. The synthesized particles were annealed at different temperatures to study the variation of particle size with annealing temperature. The crystallinity of the particles was improved with the increase in annealing temperature. The dependence of the coercivity of the nanoparticles with particle size was also investigated in this article.

Keywords—Cobalt ferrite nanoparticle, Crystallite size, Coercivity, Magnetocrystalline anisotropy, Annealing, Coprecipitation Method.

1. Introduction

In recent years magnetic nanoparticles (MNPs) have been widely investigated for their practical application in various technological fields. The potential application of the particles can be found in biomedical science and engineering. In biomedical applications, they have been used in magnetic fluid, catalysis, magnetic resonance imaging (MRI), proton exchange membrane, actuator, tissue engineering, bioanalysis, molecular detection, hyperthermia, drug delivery etc. [1-6]. The feasible applications of the particles in the field of engineering can also be found in high-frequency systems, switching devices, gas sensing, high-density recording media etc. [7-10]. The properties of the nanoparticles are mainly governed by their composition, size and shape. The important characteristics of the MNPs can be modified by changing their composition, size, and structure which opens up the possibility of a diverse range of useful applications of the particles in different application fields. Tunable magnetic properties, chemical stability, controllable size, uniform dispersion in a liquid medium, porosity, non-toxicity and biodegradability are important and necessary criteria for the nanoparticles to be used in biomedical applications. On the other hand, magnetic properties like high coercivity (H_C), good remnant magnetization, and moderate saturation magnetization are required for engineering applications of the NPs such as in permanent magnets, magnetic data storage devices, magnetic tapes etc. The synthesis techniques of the NPs play an important role as it controls the properties of the particles. Various synthesis methods have been used to modify the physical and chemical properties of the materials to obtain desired characteristics suitable for a particular application [11]. Various kinds of wet chemistry methods to synthesize MNPs have been reported in the literature such as coprecipitation, hydrothermal coprecipitation, reversed micelles coprecipitation etc. [12-14]. Besides, microemulsions, electron beam lithography, ball-milling, flow injection synthesis, electrospray synthesis, and graft copolymer methods are used frequently to prepare ferrite MNPs [15]. Among these preparation techniques, the coprecipitation method under the hydrolytic route is one of the preferred ways to prepare ferrite NPs as it offers a low-cost reagent, mild reaction conditions, and controllable size of the particles. In this method, the diameter of the spherical particle can be varied from 10-100 nm [12]. To get improved magnetic properties in the MNPs the hydrothermal coprecipitation method is preferred. Recently, high-quality magnetic ferrites with different shapes have been prepared by non-hydrolytic routes also [16]. Nickel and cobalt-based ferrite NPs are a subject of considerable interest as they exhibit important magnetic properties like high coercivity, good remnant magnetization, magnetic anisotropy and moderate saturation magnetization. The magnetic property of the particle can be varied in a wide range from

ferromagnetism to superparamagnetism which enables them to be used in biomedical applications as well as in engineering. The coercivity (H_C) of the MNPs has a remarkable dependence on their size. With the decrease in particle size, multi-domain particles become single domains below a critical diameter (D_C) and the coercivity attains a maximum value. On the further reduction of the size of the particles, the H_C decreases gradually and becomes zero at another critical size (D_S), and particles become superparamagnetic [17]. However, the critical size of the particles (D_C and D_S) depends on the composition, shape, anisotropy constant etc. [18, 19]. When the volume (V) of the single domain uniaxial particles is so small that in the absence of an applied magnetic field the height of the potential barrier K_uV (K_u = magnetic anisotropy constant) due to magnetic anisotropy becomes comparable to the thermal fluctuation energy KT, the superparamagnetism appears. In that case, thermal activation rotates the domain magnetization about the potential barrier. The particles spontaneously reverse the magnetization from one easy direction to the other. Consequently, at H=0, the net magnetic moment of an assembly of such MNPs will be zero above a certain temperature known as blocking temperature (T_B) [20].

$CoFe_2O_4$ is especially important for its physical and mechanical properties among the spinel ferrites. It is a hard magnetic material with a high coercivity (~4.3 kOe at room temperature) [21]. The particles also possess large cubic magnetocrystalline anisotropy, high magnetostrictive, and good chemical and mechanical stability. Those features make cobalt ferrite an important material in view of technological applications in various fields. In this work, cobalt-ferrite magnetic nanoparticles of particle size/crystallite size of about 50 nm were prepared by the coprecipitation method. The synthesized particles were annealed at different temperatures to study the effect of annealing on the structural and magnetic properties of the particles.

A brief introduction to magnetic nanoparticles is given in the above section. The rest of the paper is organized as follows; Section 2 contains previous works related to Co-ferrite nanoparticles. In section 3, the preparation technique of the $CoFe_2O_4$ and experimental measurements have been discussed. Section 4 describes the results and discussion of the presented work. Finally, the conclusion and future scope of the article are discussed in Section 5.

2. Related Work

Owing to the novel magnetic, optical, and electronic properties of the spinel cobalt-ferrite nanoparticles they have gained a special interest from the application as well as fundamental research point of view. A large number of studies have been reported in the literature regarding the applicability of cobalt ferrite MNPs in biological applications as well as in applied physics and engineering [22-25]. A drug release study of folic acid functionalize $CoFe_2O_4$ was investigated by Dey et al. They found co-ferrite as a useful NPs in drug delivery for cancer treatment and in hyperthermia therapy applications [26, 27]. DNA-engineered co-ferrite NPs were prepared by Das et al. for hyperthermia application [28]. It was shown that the coercivity of the NPs can be tuned by combining them with DNA. Polymer-coated monodispersed $CoFe_2O_4$ NPs prepared by Nam et al. for hyperthermia application [29]. A maximum SAR (specific absorption rate) value of 297.4 W g^{-1} was obtained for a sample concentration of 1 mg mL^{-1} under an applied field of 300 Oe and frequency of 450 kHz. Recently, to reduce cytotoxicity, Bi-doped $CoFe_2O_4$ NPs have been prepared [30]. Alberto et al. synthesized highly crystalline, monodisperse cobalt ferrite NPs with controlled size by thermal decomposition of metal-organic precursors for the development of permanent magnets [31]. Very recently, Benali et al. prepared cobalt ferrite NPs by the sol–gel auto-combustion method suitable for permanent magnet applications as well as in breast and prostate cancer treatment [32].

3. Experimental Method

3.1 Materials and methods

The wet chemical coprecipitation method was used to prepare cobalt ferrite magnetic nanoparticles (CFMNPs). The chemicals used in this preparation method are Iron acetyl acetonate, cobalt acetate, phenyl ether, ethylene glycol, oleic acid, and urea. All the reagents were procured from Sigma-Aldrich and used without further purification.

3.2 Synthesis of cobalt ferrite nanoparticles

6 mmol of iron acetyl acetonate and 3 mmol of cobalt acetate were taken in a mixture of 80 ml phenyl ether and 20 ml of ethylene glycol. Then the mixture was stirred well to dissolve properly. Finally, 8ml oleic acid and 6 gm urea were added to it and the mixture was heated in 500 ml beaker for 1 hr at 160^{o}C. The particles formed in the solution were separated from the mixture by centrifugation method and then washed several times. Finally, the particles are dried at 300 K for 24 hours. The synthesized particles were annealed at 673 K, 873 K, 1073 K and 1273K for 2 hours and preserved separately for further measurements.

3.3 Physical properties measurements of the CFMNPs

Characterization of the phase and crystallinity of the as-synthesized and annealed cobalt ferrite magnetic nanoparticles, X-ray diffraction pattern (XRD) was carried out at room temperature using Cu K_α (λ = 0.154 nm) radiation in a Rigaku Miniflex II X-ray diffractometer within 2θ range from 20° to 80° under 1°/min scanning rate (at 40KV & 40 mA). A vibrating sample magnetometer (VSM, Lake Shore Model-7144) was employed to measure the DC magnetic properties of the CFMNPs at room temperature up to a magnetic field of 1.6 T.

4. Results and Discussion

The XRD patterns of the as-synthesized CFMNPs and the annealed powder samples are depicted in Figure 1.

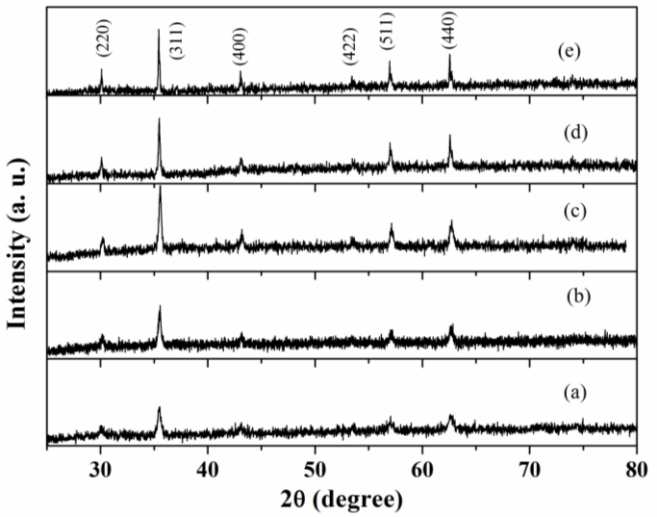

Figure 1. X-Ray diffraction pattern of (a) as-synthesized cobalt ferrite particles and samples annealed at (b) 673 K, (c) 873 K, (d) 1073 K and (e) 1273 K.

The obtained peaks of the x-ray diffraction pattern for the as-prepared particles, as well as the samples annealed at four different temperatures, are indexed and mapped with JCPDS data. A good match with the JCPDS data (card no. 22-1086) was observed. It confirms the expected spinel structure of $CoFe_2O_4$ for the prepared samples as well as the annealed samples. No undesirable impurity phase was observed from the XRD pattern. The crystallite size (d) of the samples has been estimated by using Debye Scherrer's equation ($d=0.9\lambda/(\beta\cos\theta)$). Where λ is the wavelength used in the x-ray diffraction, and β is the full width at half maximum (FWHM) of the diffraction peak. Considering the most intense peak (311) of the XRD pattern, the crystallite size (d) of all the samples (5 sets of samples) was estimated. The crystallite size of the as-prepared particles was found to be ~52 nm and that of the annealed samples was ~56 nm, ~65 nm, ~76 nm and ~84 nm for annealing temperatures 673 K, 873 K, 1073 K and 1273 K respectively. From the XRD patterns, it is obvious that the peaks are getting sharper and FWHM decreases as the annealing temperature increases. Therefore, it is evident that the crystallite size of the CFMNPs increases with the increase in annealing temperature. The crystallinity of the samples also improves with the annealing temperature.

The study of the magnetic property of the samples was performed at room temperature by using a vibrating sample magnetometer. The magnetic hysteresis curves (M vs H curve) of the as-prepared sample as well as the annealed samples were taken at room temperature and plotted in Figure 2. A non-zero coercivity is observed from the DC hysteresis loops of the CFMNPs which make it obvious that all the samples are ferromagnetic in nature. The coercivity (H_C) of the synthesized CFMNPs and the annealed samples were estimated from the corresponding hysteresis loops of the samples (Fig. 2). The coercivity of the synthesized particles was found to be 390 Oe and that for the annealed samples was 929 Oe, 580 Oe, 428 Oe and 400 Oe for annealing temperatures 673 K, 873 K, 1073 K and 1273 K respectively. The saturation magnetization (M_S) and magnetic remanence (M_R) increase with the increase in annealing temperature evident from the hysteresis loops depicted in Figure 2.

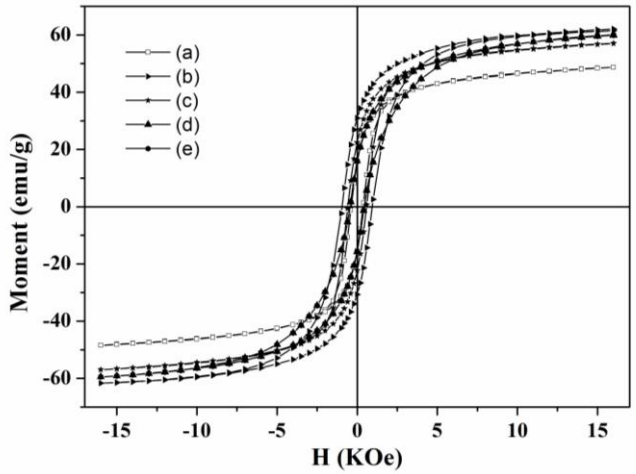

Figure 2. Magnetic hysteresis loops of (a) as-synthesized cobalt ferrite particles and samples annealed at (b) 673 K, (c) 873 K, (d) 1073 K and (e) 1273 K.

The coercivity of the as-synthesized CFMNPs increases initially when the particles were annealed at 673 K. Then H_C decreases with the increase of annealing temperature. Since there is a correlation among the annealing temperature, crystallite size and coercivity, those parameters have been plotted in the same graph. The variation of crystallite/particle size and coercivity with annealing temperatures have been plotted in Figure 3.

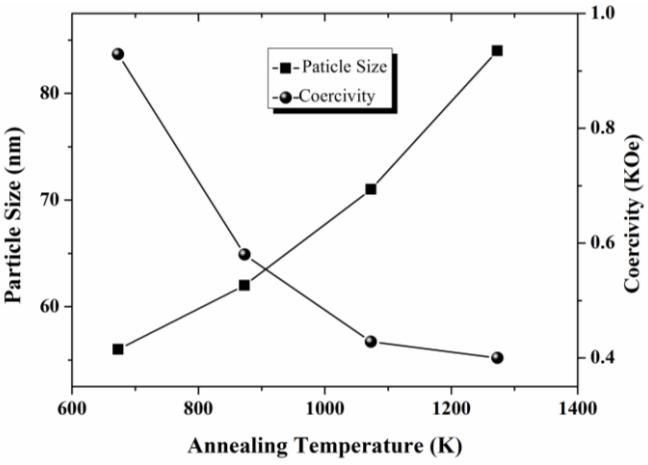

Figure 3. Variation of particle size and coercivity with annealing temperatures.

From Figure 3 it has been observed that with the increase in annealing temperature, the crystallite size of the CFMNPs increases while the coercivity decreases (The H_C of the as-synthesized CFMNPs has not been shown in this figure). The increase of crystallite size with the increase in annealing temperature is attributed to the re-crystallization process and decrease in strain broadening [33]. The small grains merge through coalescence and coarsening processes that produce larger crystallite during annealing. Therefore, the crystallite size of the as-synthesized CFMNPs can be varied by regulating the annealing temperature. Initially when the samples are annealed at 673 K, the coercivity and saturation magnetization (M_S) increases as the particles become more crystalline compared to the as-synthesized particles. As the critical diameter (D_C) of single-domain $CoFe_2O_4$ NPs for

maximum coercivity is around 40 nm [34, 35], the particle sizes of our samples are well above the single-domain range. However, the coercivity of ferrite nanoparticles depends on various factors in a complex way for single-domain and multi-domain particles but it mainly depends on the particle size. A decreasing H_C value for multi-domain CFMNPs with the increase in particle size is observed due to the crystallite size effect. Similar results were also reported in the literature for ferrite MNPs [36, 37]. The increase of saturation magnetization with the increase of particle size (or annealing temperature) is observed in Figure 3 which may be ascribed due to the increase of surface-spin canting with particle size [38].

5. Conclusion and Future Scope

Cobalt ferrite magnetic nanoparticles of crystallite size of about 50 nm were prepared by the wet chemical coprecipitation method. The particles prepared in this method show good crystallinity. The synthesized CFMNPs were annealed at different temperatures to study the effect of annealing on structural and magnetic properties. By extensive analysis of the results, it appears that the crystallite size of the as-synthesized CFMNPs can be tuned by varying the annealing temperature of the sample. The coercivity of the CFMNPs strongly depends on their particle/crystallite size which can also be induced and/or tuned by varying annealing temperatures. To enhance the magnetic properties such as coercivity, remanence magnetization etc. in CFMNPs further investigation is needed.

Data Availability

Data will be made available on request.

Conflict of Interest

The author declares no conflict of interest.

Funding Source

None

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