Comparison XRD pattern of CoFe$_2$O$_4$ thin films and nanoparticles

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Abstract. In this study, preparation of CoFe$_2$O$_4$ coated film on glass using CSD method with spin coating technique. The films deposition were carried out at room temperature. The angular velocity on the spin coating process of CoFe$_2$O$_4$ films was selected for 1000 and 3000 rpm. The CoFe$_2$O$_4$ thin film was deposited using CoFe$_2$O$_4$ nanoparticles powder. The X-ray Diffraction (XRD) was utilized to characterizing of crystal structures. The XRD analysis of CoFe$_2$O$_4$ thin films was compared with the CoFe$_2$O$_4$ nanoparticles produced and resulting the exact same spectral. The result of XRD spectral corresponds to peak data from ICDD number 221086 that indicating the sample formed was CoFe$_2$O$_4$ with inverse spinel face center cubic (FCC) structure. Furthermore, the intensity of CoFe$_2$O$_4$ nanoparticles was greater than CoFe$_2$O$_4$ thin film. The crystallite size of the CoFe$_2$O$_4$ thin films sample with 1000 rpm obtained 70.6347 nm with the strain 0.00160, whereas for 3000 rpm it was obtained 60.5354 nm with the strain 0.00187.

1. Introduction

The rapid development of nanotechnology causing a drastic reduction in magnetic device size for dimensions below one micron and thus an increase in the ratio between the surface and volume of the system. It is clear that the surface effects in such low-dimensional systems will be very important and will affect the overall magnetic behavior in various ways. In the magnetic nanoscale system, nanoparticles and thin films are examples of systems where surface effects play an important role in magnetic properties [1].

Spinel ferrite with face-centered cubic structure has the formula MFe$_2$O$_4$ where M represents divalent metal ions such as zinc, nickel, manganese or cobalt. Among these, CoFe$_2$O$_4$ is a spherical inverse ferrite with all or part of the Co$^{2+}$ ion occupying the octahedral sites (B sites) and Fe$^{3+}$ tetrahedral ions in both (A sites) and B sites [2]. Cobalt ferrite is a material suitable for practical applications such as biomedicine in human pancreas and ovarian cancer cells [3], catalysts for alkene oxidation [4], adsorbents for efficient arsenic removal [5], catalysts for methanol decomposition [6], and microwave absorption media [7]. The size of nanocrystalline spinel ferrite has been the center of attention for researchers in recent years due to excellent electrical and magnetic properties and capable of alignment (as needed). Among the ferrite spinel families, cobalt ferrite has attracted extensive research among the research community because of its high coercivity and moderate saturation magnetization with chemical stability and promising mechanical hardness suggests it to be a candidate for good magnetic storage device. The magnetic properties of cobalt ferrite (CFO) are heavily...
dependent on morphology and are therefore strongly influenced by the particle size [8]. These properties make CoFe2O4 material the right candidate for magnetic recording media, high-performance electromagnetic spintronics, cancer treatments, high-frequency applications, and materials ideal for future magnetostrictive applications [9, 10].

The magnetic behavior of the nanoparticles depends heavily on the shape, size, and purity of the nanoparticles [11]. Bulk saturation magnetization, Curie temperature, crystalline anisotropy, magnetostriction, etc. of CoFe2O4 depend on cation distribution [12]. The magnetic thin film is very important in technology because most electronic devices are exploiting magnetic behavior using thin film architecture [13]. The magnetic thin film is widely used for technological applications such as magnetic recording or micro-electromechanical applications [14].

There are several conventional methods for synthesizing cobalt nano ferrites such as sol-gel method, ball milling process, coprecipitation, hydrothermal, and much more [15-20]. Appropriate selection of processes and nano ferrite synthesis conditions will affect the crystal structure, size, morphology, and distribution affecting the magnetic properties of cobalt nano ferrite [12]. The coating of CoFe2O4 has many methods such as pulsed laser deposition [21, 22], electrophoresis [23], sputtering [24], molecular beam epitaxy [25, 26], chemical vapor deposition [27], chemical solution deposition [28-37]. Due to its wide application, it is expected to grow a coercive layer of CoFe2O4 coated thin film and nanoparticles using low cost, high quality, and time-consuming methods.

The creation of lattice strain on CFO is a center of attraction by different researchers. One way to create a lattice strain is to create a nanopattern substrate and deposition CFO on it [38]. This study emphasizes the structural enhancement of thin films and cobalt ferrite nanoparticles that comparable lattice strain values and their crystallite size.

2. Experimental

Preparation of cobalt ferrite nanoparticles by coprecipitation method

Cobalt nitrate (Co(NO3)2.6H2O) and iron nitrate (Fe(NO3)2.9H2O) as raw material, distilled water were used as solvents. The conditions and procedure routes for nano particles syntesis refer to previous experiment [39]. Cobalt nitrate and iron nitrate were dissolved in distilled water with stirring for 20 minutes (solution 1). NaOH 4.8 M (solution 2) was heated to 85°C and then titrated with solution 1 to exhaust and mixed homogeneously. The solution was washed with ethanol and then double distilled deionized water by allowed to form a precipitate. After completion of precipitate, hydrolysis process with temperature 100°C for 12 hours, and pounded clockwise so that the form of powder. Then powder on annealing 800°C for 5 hours, and pounded again clockwise.

Preparation of cobalt ferrite thin films coated on glass by CSD method

Cobalt ferrite thin films prepared by a spin coating technique using cobalt ferrite nanoparticle powder. Spin coating rotation speed varied 1000 rpm and 3000 rpm. The area of the cobalt ferrite-coated glass was 1 cm². The cobalt ferrite-coated glass was air dried and sintered at 350°C in air to remove residual solvents and any organic compounds to increase contact between the film and the substrate.

Characterization of cobalt ferrite thin films and nanoparticles

Cobalt ferrite thin films and nanoparticles were characterized by X-ray diffractometer using Cu Kα radiation. The crystallite size D of the sample was estimated using the Scherer equation, $(0.9λ) / (β\cosθ)$, by measuring the width of the main intensity peak, where $λ$ is the wavelength of Cu Kα radiation, $β$ is full width at half maximum, and $θ$ is the brag angle. Glass substrate coated using spin coating method (Chemat Technology, Spin Coater KW-4A).
3. Results and discussion

The XRD pattern of the CoFe₂O₄ thin films and nanoparticles is shown in Figure 2. The shape of the ferrite structure with the diffraction peak corresponds to the peak data of ICDD number 221086 that indicating the sample formed was CoFe₂O₄ with the inverse spinel face center cubic (FCC) structure. The XRD patterns of nanoparticles and thin films are identical, indicating that no other oxides are formed. The result of this comparison shows that the intensity of nanoparticles is larger than thin film. The peaks can be indexed by (220), (311), (222), (400), (422), (511), and (440) lattice planes. Average crystal size for the CoFe₂O₄ thin film was determined from the XRD line broadening of the most intense (311) diffraction peak using the Scherrer formula [40-42]:

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \]  \hspace{1cm} (1)

Where \( \lambda \) is the wavelength of Cu Kα, \( \beta \) is the full width at half maximum, and \( \theta \) is the Bragg angle. From Scherrer formula, it is calculated that the size of the thin film crystallites is 70.6347 nm, while the nanoparticle crystallite size is 42.3837 nm.

The strain in CoFe₂O₄ thin films are calculated by using Hall-Williamson’s plot using the formula

\[ \varepsilon = \frac{\beta_{hkl}}{4 \tan \theta} \]  \hspace{1cm} (2)

Where \( \varepsilon \) is a strain and \( \beta_{hkl} \) is full width at half maximum. In addition, it can also compare the size of crystallites and strains, where these results indicate that the thin film crystallite size is larger than that of nanoparticles. While the strain of thin film is smaller than nanoparticles, i.e., for thin film 0.00160 and nanoparticles 0.00267.

Figure 2 shows the XRD pattern of the CoFe₂O₄ thin films with different spin coating speeds. All of the CoFe₂O₄ layers were spin coated for five times. From Scherrer formula, it is calculated that crystallite size decreases from 70.6347 nm to 60.5354 nm by increasing spin coating speed from 1000
rpm to 3000 rpm. While the lattice strain is found to increase from 0.00160 to 0.00187 by increasing
spin coating speed from 1000 rpm and 3000 rpm.

![XRD patterns of CoFe2O4 thin films deposition for 1000 rpm and 3000 rpm.](image)

**Fig. 2.** XRD patterns of CoFe2O4 thin films deposition for 1000 rpm and 3000 rpm.

4. **Conclusions**
The CoFe2O4 thin films have been successfully prepared using CSD method with a spin coating
technique. The results were compared with CoFe2O4 nanoparticles. The XRD characterization is
performed to ensure the crystal structure of CoFe2O4 which produces peak data according to ICDD
number 221086 that the sample formed is CoFe2O4 with inverse spinel face-centered cubic (FCC)
structure. The XRD patterns of nanoparticles and thin films are identical, indicating that no other
oxides are formed. The average of the CoFe2O4 thin films crystallite size was found in the range of 60-
71 nm, so it can be concluded that the crystallite size of thin films is greater than that of nanoparticles.
While inversely proportional to the strain, where the strain of thin films is smaller than nanoparticles.
The enhancement of the structural properties of the ferrite CoFe2O4 at the nano level makes this
material a suitable candidate for different applications.

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