

Morphological and Crystallographic Analysis of Cobalt Ferrite Thin Films Fabricated Through Spray Pyrolysis Methodology

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Abstract: This study investigates the morphological and crystallographic characteristics of cobalt ferrite (CoFe_2O_4) thin films synthesized using spray pyrolysis techniques. The research examines how various processing parameters influence the structural development and resultant properties of these magnetic oxide films. Comprehensive characterization reveals that substrate temperature, precursor concentration, and post-deposition thermal treatments significantly affect crystalline quality, grain morphology, and phase composition. X-ray diffraction analysis confirms the formation of single-phase cubic spinel structures, while electron microscopy techniques provide insights into surface topography and microstructural evolution. The findings demonstrate that controlled manipulation of deposition parameters enables precise tailoring of film properties for specific technological applications. This work contributes to the understanding of structure-property relationships in ferrite thin films and establishes guidelines for optimizing synthesis conditions

Keywords: Cobalt Ferrite, Thin Films, Spray Pyrolysis, Structural Characterization

I. INTRODUCTION

Ferrite materials have emerged as essential components in modern technological applications due to their unique combination of magnetic, electrical, and chemical properties [1]. Among these materials, cobalt ferrite (CoFe_2O_4) stands out as a particularly promising candidate for various applications, including magnetic storage devices, sensors, catalysts, and microwave components [2]. The material's inverse spinel crystal structure, characterized by the formula $(\text{Co}_{1-x}\text{Fe}_x)(\text{Fe}_{2-x}\text{Co}_x)\text{O}_4$, provides exceptional thermal stability and tunable magnetic properties through controlled cation distribution [3]. The development of thin film forms of cobalt ferrite has opened new possibilities for miniaturized devices and enhanced performance characteristics. Thin films offer advantages such as reduced material consumption, improved surface-to-volume ratios, and enhanced interface effects that can significantly modify bulk properties [4]. These dimensional constraints lead to novel physical phenomena, including altered magnetic anisotropy, modified electrical conductivity, and enhanced catalytic activity [5]. Among various thin film deposition techniques, spray pyrolysis has gained considerable attention due to its cost-effectiveness, scalability, and ability to produce high-quality films without requiring vacuum conditions [6]. This chemical vapor deposition variant involves the atomization of precursor solutions onto heated substrates, where thermal decomposition and chemical reactions occur to form the desired compound. The technique offers excellent control over film composition, thickness, and microstructure through systematic variation of processing parameters [7]. The spray pyrolysis process encompasses three fundamental stages: precursor solution preparation, aerosol generation and transport, and substrate-based synthesis reactions. Each stage provides opportunities for controlling the final film properties through careful selection of chemical precursors, solvents, carrier gases, and thermal conditions [8, 9]. This investigation aims to establish comprehensive relationships between spray pyrolysis processing parameters and the resulting structural characteristics of cobalt ferrite thin films [10]. The research focuses on characterizing the crystallographic evolution as a function of synthesis conditions while analyzing morphological development and surface topography variations. Additionally, the study seeks to establish



correlations between processing parameters and film properties, ultimately providing optimization guidelines for targeted applications.

II. EXPERIMENTAL METHODOLOGY

2.1 Materials and Precursor Preparation

High-purity cobalt nitrate hexahydrate $[\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}]$ and iron nitrate nonahydrate $[\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$ were utilized as metal precursors. These salts were dissolved in distilled water to achieve the stoichiometric ratio corresponding to CoFe_2O_4 formation. Solution concentrations ranged from 0.05 M to 0.25 M to investigate concentration effects on film properties. The precursor solutions were prepared by dissolving appropriate quantities of metal salts in deionized water under continuous stirring at room temperature. The pH was adjusted to approximately 2-3 using dilute nitric acid to ensure complete dissolution and prevent precipitation. Solutions were filtered through 0.45 μm membranes to remove any undissolved particles.

2.2 Thin Film Deposition Process

A custom-built spray pyrolysis system was employed for film deposition. The apparatus consisted of an atomization unit, substrate heating assembly, and gas flow control system. Glass substrates were thoroughly cleaned using acetone, ethanol, and deionized water before deposition. The investigation encompassed several key processing parameters to establish comprehensive structure-property relationships. Substrate temperatures were varied from 300 $^{\circ}\text{C}$ to 500 $^{\circ}\text{C}$ to evaluate thermal effects on crystallization and grain growth. Precursor concentrations ranged from 0.05 M to 0.25 M to assess the influence of solution concentration on film formation kinetics and final properties. Spray rates were controlled between 2 and 8 mL/min to optimize droplet delivery and surface interaction dynamics. Deposition durations were systematically varied from 30 to 120 minutes to achieve desired film thicknesses while maintaining structural quality. Carrier gas flow rates were maintained between 5 and 15 L/min to ensure consistent aerosol transport and minimize compositional variations [11-13] as shown in **Figure 1**.

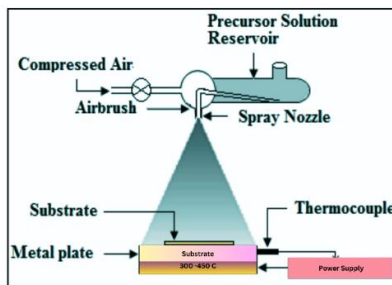


Figure 1. Spray pyrolysis deposition techniques for CoFe_2O_4 thin film

2.3 Post-Deposition Thermal Treatment

Selected samples underwent thermal annealing in air atmosphere using a programmable furnace. Annealing temperatures ranged from 400 $^{\circ}\text{C}$ to 700 $^{\circ}\text{C}$ with holding times of 2-6 hours. Heating and cooling rates were maintained at 5 $^{\circ}\text{C}/\text{min}$ to minimize thermal stress.

III. CHARACTERIZATION TECHNIQUES

Comprehensive characterization of the synthesized cobalt ferrite thin films was conducted using multiple complementary analytical techniques to provide detailed insights into structural, morphological, and optical properties. X-ray diffraction (XRD) measurements were performed using a Rigaku MiniFlex diffractometer with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$), conducting scans over 2θ ranges of 20° to 80° with step sizes of 0.02° , where crystallite sizes were calculated using the Scherrer equation and lattice parameters were determined through peak position analysis. Surface morphology was examined using field emission scanning electron microscopy (FESEM) with a JEOL JSM-7600F instrument, while energy dispersive X-ray spectroscopy (EDS) provided elemental composition analysis, and



transmission electron microscopy (TEM) observations were conducted using a JEOL JEM-2100F microscope operating at 200 kV for detailed microstructural examination. Atomic force microscopy (AFM) measurements were performed using a Park NX10 system in non-contact mode, with surface roughness parameters calculated from $5 \times 5 \mu\text{m}$ scan areas using statistical analysis software to quantify topographical features. Spectroscopic analysis was conducted through Fourier transform infrared (FTIR) spectra recording using a Shimadzu IRTracer-100 spectrometer in the $400\text{--}4000 \text{ cm}^{-1}$ range, while UV-visible absorption spectra were obtained using a Shimadzu UV-2600 spectrophotometer for comprehensive optical property evaluation [14-16].

IV. RESULTS AND DISCUSSION

4.1 Phase Formation and Crystallographic Analysis

4.1.1 X-ray Diffraction Studies

XRD analysis confirmed the successful formation of single-phase cobalt ferrite with cubic spinel structure across all synthesis conditions investigated. The diffraction patterns exhibited characteristic peaks corresponding to (220), (311), (400), (422), (511), (440), and (533) reflections, consistent with the $Fd3m$ space group. No secondary phases were detected within the instrumental detection limits, indicating high phase purity. The relative intensities of diffraction peaks varied with processing conditions, suggesting changes in preferred crystallographic orientation. Films deposited at higher substrate temperatures showed enhanced (311) peak intensity, indicating preferential growth along this direction. This texturing behavior is attributed to surface energy minimization during crystal nucleation and growth [17, 18] as shown in Figure 2.

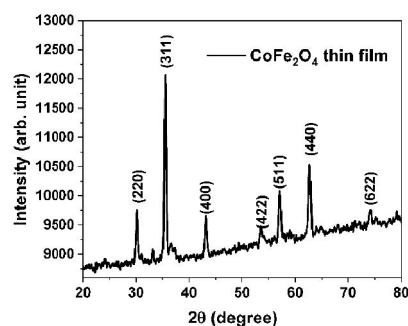


Figure 2. X-ray diffraction pattern of CoFe_2O_4 thin film

4.1.2 Crystallite Size Evolution

Crystallite sizes calculated from XRD peak broadening using the Scherrer equation ranged from 15 to 45 nm, depending on synthesis conditions. Higher substrate temperatures and longer deposition times promoted crystallite growth, while increased precursor concentrations led to finer crystallites due to enhanced nucleation density. The relationship between annealing temperature and crystallite size followed an expected growth trend after annealing at 600°C . This growth is attributed to thermally activated grain boundary migration and coalescence processes [19]. Crystallite sizes was calculated by Debye-formula. Scherrer's [20], as eq. (1)

$$D = (k\lambda) / (\beta \cos\theta) \quad (1)$$

4.1.3 Lattice Parameter Determination

Lattice parameters calculated from peak positions showed values ranging from 8.350 to 8.425 \AA , which are consistent with reported literature values for cobalt ferrite. Slight variations observed across different samples are attributed to compositional fluctuations and internal strain effects. Higher synthesis temperatures generally resulted in lattice parameter values closer to bulk cobalt ferrite, suggesting improved stoichiometry and reduced defect concentrations. Lattice parameters calculated from the equation (2)



$$a = d_{hkl} \sqrt{h^2 + k^2 + l^2} \quad (2)$$

4.2 Morphological Characteristics

4.2.1 Surface Morphology Evolution

FESEM observations revealed significant morphological variations as functions of processing parameters. Films deposited at lower substrate temperatures (300 °C) exhibited fine-grained structures with average grain sizes of 50-80 nm. Increasing the substrate temperature to 450 °C resulted in larger, well-defined grains ranging from 100-200 nm with improved crystalline faceting. Precursor concentration effects were particularly pronounced in surface morphology development. Lower concentrations (0.05 M) produced sparse, isolated island-like structures, while higher concentrations (0.20 M) yielded continuous, uniform films with complete substrate coverage. Intermediate concentrations provided optimal balance between film continuity and grain definition. **Figure 3** depicts a SEM image of CoFe₂O₄ thin film prepared via spray pyrolysis technique.

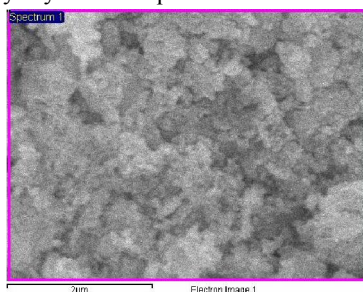


Figure 3. SEM image of CoFe₂O₄ thin film

4.2.2 Elemental Distribution Analysis

EDS mapping confirmed uniform distribution of cobalt, iron, and oxygen throughout the film thickness and lateral extent. The atomic ratios closely matched the intended stoichiometry (Co:Fe = 1:2), with slight variations attributed to preferential evaporation of volatile species during high-temperature deposition.

4.3 Surface Topography Analysis

4.3.1 AFM Surface Characterization

AFM measurements provided quantitative assessment of surface roughness parameters. Root mean square (RMS) roughness values ranged from 8 to 35 nm, showing strong correlation with deposition conditions. Films deposited at moderate temperatures (400 °C) exhibited the smoothest surfaces (RMS ~10 nm), while higher temperatures led to increased roughness due to enhanced grain growth and agglomeration. The surface topography revealed three-dimensional grain structures with well-defined boundaries. Grain sizes measured from AFM images as shown in **Figure 4**, correlated well with XRD crystallite size calculations, validating the structural analysis results.

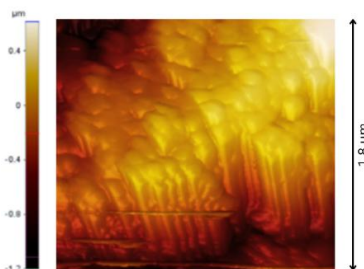


Figure 4. AFM image of CoFe₂O₄ thin film



4.3.2 Statistical Surface Analysis

Statistical analysis of AFM data revealed Gaussian height distributions for most samples, indicating random surface variations typical of polycrystalline films. Skewness and kurtosis parameters provided insights into surface asymmetry and peak sharpness, respectively. These parameters showed systematic variations with processing conditions, enabling quantitative correlation with functional properties.

4.4 Spectroscopic Characterization

4.5 FTIR Analysis

FTIR spectra exhibited characteristic absorption bands associated with metal-oxygen vibrations in the spinel structure. The primary absorption band around 580 cm^{-1} corresponds to Fe-O stretching vibrations in tetrahedral sites, while the band at 400 cm^{-1} is attributed to Co-O vibrations in octahedral sites. The presence and intensity of these bands confirmed the formation of the inverse spinel structure. Band position shifts observed in **Figure 5**, with varying synthesis conditions indicate changes in bond lengths and cation distribution, consistent with XRD lattice parameter variations. Higher synthesis temperatures resulted in sharper, more well-defined absorption bands, suggesting improved structural ordering.

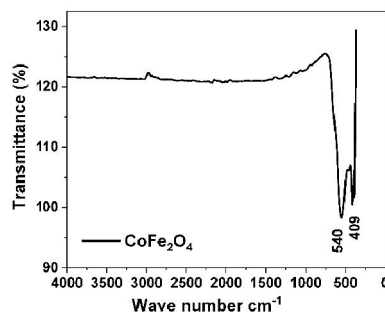


Figure 5. FTIR spectra of CoFe₂O₄ thin film

4.5.1 Optical Properties

UV-visible absorption spectra revealed optical band gap values ranging from 1.8 to 2.3 eV, depending on film microstructure. The band gap showed inverse correlation with crystallite size, consistent with quantum confinement effects in nanoscale systems. Films with larger crystallites exhibited red-shifted absorption edges due to reduced confinement and increased defect states. Absorption coefficients derived from transmission measurements indicated strong light absorption in the visible region, making these films potentially suitable for photocatalytic applications. The absorption edge sharpness improved with enhanced crystallinity, reflecting reduced disorder and defect concentrations.

4.6 Processing Parameter Correlations

4.6.1 Substrate Temperature Effects

Substrate temperature emerged as the most influential parameter affecting film properties. Temperature increases from 300 °C to 500 °C resulted in significant crystallite size increases from 15 to 40 nm, accompanied by improved crystalline ordering and phase purity. These temperature elevations also enhanced grain connectivity and film continuity while reducing surface roughness at intermediate temperatures. Furthermore, higher temperatures promoted lattice parameter convergence toward bulk values, indicating improved structural quality and reduced defect concentrations.

4.6.2 Precursor Concentration Influence

Precursor concentration variations produced systematic changes in film characteristics that directly influenced the final material properties. Higher concentrations promoted increased nucleation density and the formation of finer



microstructures, while optimal concentrations in the range of 0.15-0.20 M yielded the best combination of substrate coverage and crystallinity. However, excessive concentrations led to solution instability and non-uniform deposition patterns, whereas lower concentrations resulted in incomplete substrate coverage and discontinuous film formation.

4.6.3 Thermal Treatment Impact

Post-deposition annealing significantly enhanced structural quality through multiple simultaneous mechanisms. The thermal treatment increased crystallite sizes by 50-100% after annealing at 600 °C, while simultaneously improving phase purity through the elimination of metastable phases. The annealing process also regularized surface morphology with reduced defect density and promoted lattice strain relaxation, leading to parameter stabilization and improved overall structural integrity.

4.7 Structure-Property Relationships

4.7.1 Morphology-Crystallinity Correlations

Strong correlations were established between surface morphology and crystalline quality. Well-faceted grains observed in FESEM images corresponded to sharp, intense XRD peaks, indicating high crystalline ordering. Conversely, rounded, poorly defined grains correlated with broad diffraction peaks characteristic of structural disorder. The grain size distribution measured from microscopy showed good agreement with crystallite size calculations from XRD, validating both measurement approaches. This correlation enables prediction of microstructural characteristics from simple XRD measurements.

4.7.2 Processing-Structure Relationships

Systematic analysis revealed that optimal structural quality is achieved through carefully balanced processing conditions that address multiple competing factors simultaneously. Moderate substrate temperatures in the range of 400-450 °C provide the best combination of crystallinity and morphology by promoting adequate thermal energy for crystallization while avoiding excessive grain growth. Precursor concentrations of 0.15-0.20 M ensure uniform substrate coverage with good crystalline quality by maintaining appropriate nucleation density without causing solution instability. Post-deposition annealing at temperatures between 550-600 °C enhances material properties without inducing excessive grain growth that could compromise surface area or introduce unwanted stress. Additionally, controlled spray rates of 4-6 mL/min optimize droplet-surface interactions by ensuring adequate material delivery while preventing solution accumulation or non-uniform distribution.

4.7.3 Property Optimization

Based on the structure-property correlations established, specific guidelines have been developed for targeted applications across different technological domains. For magnetic applications, maximizing crystallite size through high-temperature processing and controlled annealing enhances magnetic ordering and reduces superparamagnetic behavior, leading to improved magnetic performance and stability. For catalytic applications, optimizing surface area through carefully controlled grain size and surface roughness provides the ideal balance between crystallinity requirements and surface accessibility for reactant molecules. For optical applications, precise control of crystallite size enables tuning of the optical band gap while maintaining sufficient structural quality for efficient charge separation and transport processes.

V. APPLICATIONS AND FUTURE PERSPECTIVES

5.1 Technological Applications

The structural control achieved through optimized spray pyrolysis enables precise tailoring of cobalt ferrite thin films for diverse technological applications across multiple domains. In magnetic storage devices, films with controlled crystallite size and preferred orientation demonstrate significant promise for high-density recording applications [21, 22] particularly where perpendicular magnetic anisotropy is crucial for device performance as shown in **Figure 6**. For chemical sensors, the unique combination of magnetic properties and chemical stability makes these films exceptionally



suitable for selective gas sensing applications, especially for detecting oxidizing gases in challenging environments [23-25]. In photocatalytic systems, the tunable optical band gap combined with high surface area enables efficient photocatalytic degradation of organic pollutants under visible light irradiation, offering environmental remediation solutions. For energy storage applications, the redox activity of iron and cobalt cations provides substantial potential for supercapacitor electrode applications with both high energy density and high power density characteristics [26-30].

VI. FUTURE RESEARCH DIRECTIONS

Several critical areas warrant further investigation to fully exploit the potential of spray pyrolysis-synthesized cobalt ferrite thin films. Advanced processing techniques represent a promising frontier, including the development of pulsed spray deposition methods for ultra-thin film fabrication, investigation of hybrid precursor systems that combine organic and inorganic compounds for enhanced control, and exploration of reactive atmosphere processing for controlled defect engineering. Multifunctional property engineering offers another avenue for advancement, focusing on simultaneous optimization of magnetic, optical, and electrical properties, development of heterostructured films with graded compositions for enhanced functionality, and integration with other functional materials for comprehensive device applications. Scale-up and industrial implementation remain crucial considerations, requiring process optimization for large-area uniform deposition, comprehensive cost analysis and economic feasibility studies, and development of continuous processing techniques suitable for industrial production environments [31, 32].

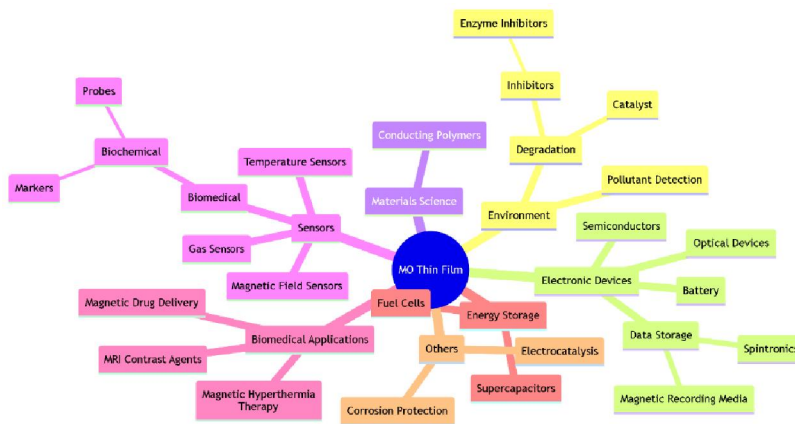


Figure 6. Various applications of CoFe_2O_4 thin film

VII. CONCLUSION

This comprehensive investigation of cobalt ferrite thin films synthesized via spray pyrolysis has established fundamental relationships between processing parameters and structural characteristics through systematic analysis. The research demonstrates that single-phase cubic spinel structures are consistently achieved across a wide range of processing conditions, with crystallite sizes controllable from 15 to 45 nm through systematic parameter variation, providing excellent flexibility for application-specific optimization. Surface morphology and grain structure can be precisely tailored through substrate temperature and precursor concentration optimization, enabling achievement of either smooth, continuous films or textured surfaces as required for specific technological applications. Optimal structural quality is achieved at moderate substrate temperatures of 400-450 °C, intermediate precursor concentrations of 0.15-0.20 M, and controlled post-deposition annealing at 550-600 °C, providing clear guidelines for future synthesis efforts. Strong correlations established between processing conditions, structural characteristics, and functional properties enable predictive design of films for targeted applications, significantly reducing development time and resources. The demonstrated structural control opens substantial possibilities for applications in magnetic storage, chemical sensing, photocatalysis, and energy storage systems, positioning these materials at the forefront of advanced technological development. The spray pyrolysis technique proves to be a versatile and cost-effective method for producing high-quality cobalt ferrite thin films with controllable properties. The fundamental understanding developed



in this work provides a foundation for further advancement in ferrite thin film technology and its implementation in next-generation devices.

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