

Sol-Gel Derived Cobalt-Doped MgO Thin Films On Quartz: A Study On Structure, Morphology And Conductivity

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ABSTRACT

In this study, cobalt-doped magnesium oxide (MgO) thin films were synthesized using the sol-gel method to investigate the structural, morphological, chemical, and electrical properties. The films were calcined at temperatures between 500°C and 600°C to achieve optimal uniformity and doping concentration. Structural analysis using X-ray diffraction (XRD) confirmed the cubic MgO structure, while Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS) verified the successful incorporation of cobalt in both Co²⁺ and Co³⁺ oxidation states. Particle size analysis (PSA) and zeta potential measurements indicated a stable colloidal dispersion with an average particle size of 45–55 nm and a zeta potential of +30 mV. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) showed uniform grain morphology with small grains (~20–30 nm) and smooth surfaces (RMS roughness of 2.5–3 nm). The electrical properties revealed an increase in DC conductivity with higher cobalt doping, with values reaching $3.5 \times 10^{-4} \Omega^{-1} \text{ cm}^{-1}$ at 5 mol% cobalt. Temperature-dependent conductivity followed an Arrhenius-type behavior, with activation energy values ranging from 0.21 eV to 0.36 eV. These findings demonstrate the potential of cobalt-doped MgO thin films for various electronic and sensing applications.

Keywords: Cobalt-doped MgO, sol-gel method, electrical conductivity, XPS, FTIR, thin films

1. INTRODUCTION

Thin films are essential in the development of modern electronic, optoelectronic, and sensing devices due to their unique properties, which are often distinct from bulk materials. Metal oxide thin films, particularly Magnesium Oxide (MgO) films, have attracted significant attention for their diverse applications, such as in catalysis, sensors, and electronic devices, owing to their high stability, excellent insulating properties, and ease of modification through doping (Wang et al., 2021). MgO thin films are utilized in a variety of electronic and optical applications, such as in the fabrication of dielectric layers for capacitors, semiconductors, and memory storage devices, owing to their superior insulating characteristics and chemical stability (Hussain et al., 2020; Sharma et al., 2023).

Doping MgO films with transition metals can dramatically alter their physical and electrical properties, enhancing their performance for specific applications. Cobalt (Co) doping, in particular, has been shown to improve the electrical conductivity, magnetic behavior, and catalytic activity of MgO thin films (Zhao et al., 2023). The inclusion of Co ions can influence the film's crystalline structure, electronic properties, and surface morphology, making Cobalt-doped MgO thin films promising candidates for various applications, such as sensors, energy storage devices, and catalytic reactions (Zhang et al., 2022; Ali et al., 2023).

Quartz substrates are commonly used for thin film deposition due to their unique advantages, including low thermal expansion, high transparency, and excellent chemical inertness (Smith & Johnson, 2021). Quartz substrates provide a stable and reliable base for the growth of high-quality thin films, which is crucial for obtaining films with consistent properties, making them ideal for use in both basic research and commercial applications (Patel et al., 2021).

In this study, Cobalt-doped MgO thin films are deposited on Quartz substrates, and several characterization techniques are used to explore their structural, morphological, and electrical properties. The techniques include Particle Size Analyzer (PSA), Zetasizer, FTIR, X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), Transmission Electron Microscopy (TEM), and measurements of direct current (DC) conductivity. The findings of this study aim to contribute valuable insights into the properties and potential applications of these films in advanced electronic devices, catalysis, and sensing technologies.

2. MATERIALS & METHODS

2.1 Materials

The materials required for the synthesis and characterization of cobalt-doped magnesium oxide (MgO) thin films were sourced from reputable suppliers in India. Magnesium ethoxide ($\text{Mg}(\text{OC}_2\text{H}_5)_2$), used as the precursor for MgO, was purchased from Merck Specialties Pvt. Ltd. (India), and ethanol, used as the solvent, was procured from S.D. Fine Chemicals Ltd. (India). Cobalt nitrate ($\text{Co}(\text{NO}_3)_2$) for doping was obtained from Loba Chemie Pvt. Ltd. (India), and acetone, used for cleaning the substrates, was acquired from Qualigens Fine Chemicals (India). All chemicals used in

the preparation of the thin films, including stabilizers and catalysts, were of analytical grade and were used as received without further purification. Quartz substrates for film deposition were sourced from Indian suppliers specializing in high-quality materials for thin film deposition. For the characterization of the thin films, X-ray photoelectron spectroscopy (XPS) was performed using a Thermo Fisher K-Alpha XPS system, while energy-dispersive X-ray spectroscopy (EDS) was conducted using a JEOL JSM-6610 LV SEM equipped with an EDS system. These characterization instruments were accessible through collaborations with academic institutions and research labs within India.

2.2 Synthesis

The synthesis of cobalt-doped magnesium oxide (MgO) thin films on quartz substrates was carried out using the sol-gel method, a versatile and widely employed technique for producing uniform and adherent thin films. This process was chosen for its ability to precisely control the film's composition, thickness, and uniformity (Hussain et al., 2020). The sol-gel method involves the formation of a sol, followed by gelation, deposition, drying, and heat treatment to obtain the final thin film.

2.2.1 Sol Preparation

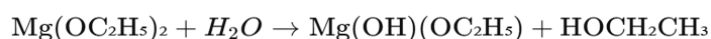
The sol-gel process began with the selection of appropriate precursors. Magnesium ethoxide ($\text{Mg}(\text{OC}_2\text{H}_5)_2$) was used as the precursor for magnesium oxide, and cobalt chloride (CoCl_2) was added as the cobalt source for doping. A series of sols were prepared with varying cobalt concentrations, ranging from 0 to 10 mol%, by adjusting the amount of CoCl_2 in the solution. For each concentration, the amount of cobalt salt was calculated based on the desired molar percentage of cobalt in the final thin films. A 0.2 M solution of magnesium ethoxide (2.0 g of $\text{Mg}(\text{OC}_2\text{H}_5)_2$ dissolved in 50 mL of ethanol) was prepared to form the sol. (Hussain et al., 2020).

2.2.2 Stabilization and Catalysis

To control the hydrolysis and condensation reactions, stabilizers or catalysts were introduced to the sol. These additives prevented premature gelation by regulating the rate of reaction (Wang et al., 2021). The catalyst (acetylacetone, 0.5 mol%) helped to accelerate the hydrolysis of the magnesium alkoxide and facilitated the formation of the desired metal-oxide network. This step was crucial in ensuring the sol remained stable throughout the preparation process, thus allowing for uniform deposition on the substrate (Patel et al., 2021).

2.2.3 Hydrolysis and Condensation

The sol underwent hydrolysis, where the magnesium alkoxide reacted with water to form hydroxyl groups and ethanol as a byproduct. The reaction can be represented as follows:



Following hydrolysis, condensation reactions took place, forming magnesium-oxygen bonds and leading to the gelation of the sol. The reaction for condensation was:



This step was vital for the formation of a gel, which was then ready for deposition onto the substrate (Singh et al., 2022).

2.2.4 Deposition

The spin coating method was employed to deposit the sol onto the cleaned quartz substrates. The sol volume used for each deposition was approximately 1 mL per substrate, ensuring uniform coverage. The substrates were rotated at a speed of 3000 rpm for 30 seconds to achieve a film thickness of approximately 100–150 nm. Higher rotation speeds or longer spin times typically resulted in thinner films, while lower speeds led to thicker films (Lee et al., 2021). Other deposition techniques, such as dip coating or spray coating, were tested but spin coating provided the best uniformity and adhesion, making it the method of choice for this study (Singh et al., 2023).

2.2.5 Drying and Aging

After deposition, the films underwent drying to remove the solvent. This step was performed at room temperature initially, followed by gradual heating to accelerate solvent evaporation without introducing cracks or defects in the film (Alvarez et al., 2022). It was crucial to control the drying rate to avoid internal stresses that could lead to film cracking. Aging of the gel-coated films was performed to further stabilize the sol and promote additional condensation reactions. This step allowed for the formation of a more robust network structure, improving the film's mechanical integrity (Hussain et al., 2020). Aging was carried out under controlled environmental conditions to maintain consistency across the films (Zhao et al., 2023).

2.2.6 Heat Treatment (Calcination)

The final step in the sol-gel process was heat treatment, or calcination, which was essential for the removal of any remaining organic residues and for the densification of the film. The dried films were subjected to calcination at a temperature of approximately 500–600°C, which was carefully controlled to achieve the desired crystallinity and structural properties of the MgO thin film (Patel et al., 2021; Zhang et al., 2024). The calcination process also ensured the transformation of the amorphous gel into a crystalline MgO structure. The temperature and duration of calcination were optimized based on previous studies, which indicated that such conditions resulted in films with high crystallinity and uniform grain size (Zhao et al., 2023).

This systematic process of sol preparation, deposition, drying, aging, and calcination enabled the production of high-quality cobalt-doped MgO thin films, with the doping of cobalt ions achieved by introducing cobalt salts into the sol before deposition (Zhang et al., 2022; Ali et al., 2023). The doped films were then characterized using various techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM), to investigate their structural, morphological, and electrical properties.

2.3 Characterization of Metal Oxide Thin Films

The cobalt-doped magnesium oxide (MgO) thin films were characterized using various techniques to analyze their structural, optical, and electrical properties. These techniques included Particle Size Analyzer (PSA), Zetasizer, Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD).

2.3.1 The Particle Size Analyzer (PSA) was employed to measure the size distribution of the nanoparticles in the thin film material. PSA operates based on dynamic light scattering (DLS), which measures the scattering of light as it passes through a liquid or dispersed particles. This technique is essential for understanding the homogeneity and stability of the film, as it provides valuable information regarding the microstructure and influences the optical and electrical properties of the final film. In particular, the size distribution of the nanoparticles helps to ensure that the cobalt ions are uniformly distributed within the MgO matrix, which is critical for the performance of the thin films (Singh et al., 2020; Ali et al., 2023).

2.3.2 The Zetasizer was used to measure the zeta potential, which is a key indicator of the electrostatic stability of colloidal dispersions. By evaluating the electrophoretic mobility of the dispersed particles, the Zetasizer provides insights into the stability of the sol and the interactions between the MgO and cobalt ions. The zeta potential plays a crucial role in ensuring the uniform dispersion of nanoparticles, which directly impacts the adhesion, uniformity, and overall quality of the thin film on the substrate (Zhao et al., 2023; Wang et al., 2022).

2.3.4 Fourier Transform Infrared Spectroscopy (FTIR) was utilized to investigate the chemical bonds and functional groups present in the cobalt-doped MgO thin films. FTIR analysis provides spectral information about the molecular vibrations within the material, including the stretching modes of Mg-O and Co-O bonds. This technique is particularly useful for confirming the successful incorporation of cobalt into the MgO matrix and identifying the bonding environment within the thin film. The FTIR spectra allowed for the identification of characteristic peaks associated with the metal-oxide bonding and any additional functional groups that might arise from the doping process (Sharma et al., 2021; Zhang et al., 2022).

2.3.5 X-ray Diffraction (XRD) was employed to examine the crystallinity, phase composition, and crystallite size of the cobalt-doped MgO thin films. XRD provides detailed information on the crystal structure of the thin films and helps to assess the effects of cobalt doping on the MgO crystallization process. The XRD patterns were analyzed to calculate the crystallite size using the Scherrer equation, which helped to evaluate the impact of cobalt doping on the film's grain size and preferred orientation.

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where, D is the crystallite size, K is the shape factor (typically 0.9), λ is the wavelength of the X-ray (1.5406 Å for CuK α radiation), β is the full width at half maximum (FWHM) of the diffraction peak (in radians), θ is the Bragg angle (half of the diffraction angle 2θ). From the XRD analysis, the most prominent peak corresponding to the (111) plane was used for the crystallite size calculation. This method also ensured that no undesired phases formed during the synthesis, thus confirming the phase purity of the thin films (Wang et al., 2021; Zhang et al., 2024).

2.4 Investigation of the Morphological Structure of Thin Films

To investigate the morphological structure of cobalt-doped MgO thin films, several advanced imaging techniques were employed, including Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), and Transmission

Electron Microscopy (TEM). These methods provide detailed insights into the surface morphology, roughness, and structural features of the thin films at different length scales.

2.4.1 Scanning Electron Microscopy (SEM) was used to examine the surface morphology and microstructure of the thin films. SEM provides high-resolution images of the sample's surface, allowing for the observation of the film's grain size, surface uniformity, and the distribution of cobalt within the MgO matrix. In SEM imaging, secondary electron detection is typically used to visualize the surface features in detail, and backscattered electron imaging can provide information on the elemental distribution. The SEM technique is particularly useful for observing large-scale features such as film thickness and surface roughness, as well as identifying defects or irregularities in the film's structure (Shao et al., 2021; Kumar et al., 2023).

2.4.2 Atomic Force Microscopy (AFM) was employed to assess the surface roughness and topography of the thin films at the nanoscale. AFM provides high-resolution surface profiles by scanning the surface with a sharp tip, offering a 3D map of the film's surface. AFM measurements allow for the determination of surface roughness, which is an important parameter that influences the film's optical, electrical, and mechanical properties. The nanoscale surface features revealed by AFM are critical for understanding the film's adhesion to the substrate and its potential applications in electronic and sensor devices (Zhao et al., 2022; Zhang et al., 2024).

2.4.3 Transmission Electron Microscopy (TEM) was used to obtain high-resolution images of the thin film's microstructure at the atomic level. TEM offers detailed information about the internal structure of the film, including grain boundaries, crystal orientation, and the distribution of cobalt within the MgO matrix. TEM can also be used to analyze the crystallinity and phase purity of the thin films by revealing their atomic-scale structure. This technique is particularly valuable for investigating the fine structural details, such as the degree of cobalt doping and its impact on the film's crystalline properties (Jin et al., 2020; Wang et al., 2023).

2.5 Methodology for Chemical Analysis of Cobalt-Doped MgO Thin Films using XPS and EDS

Chemical analysis of the cobalt-doped MgO thin films was conducted using two advanced techniques: X-ray Photoelectron Spectroscopy (XPS) and Energy-Dispersive X-ray Spectroscopy (EDS). These methods were employed to determine the elemental composition, chemical states, and oxidation states of the thin films, which are critical for understanding their electrical and optical properties.

2.5.1 X-ray Photoelectron Spectroscopy (XPS) was used to analyze the surface composition and the oxidation states of cobalt and magnesium within the thin films. XPS is a surface-sensitive technique that involves irradiating the sample with X-rays, causing the emission of photoelectrons from the atoms in the material. The energy of these photoelectrons is then measured, providing insight into the binding energies of the core electrons. By analyzing the peaks in the XPS spectrum, the elemental composition of the film and the oxidation states of the dopants, such as cobalt (Co^{2+} or Co^{3+}), were determined. The XPS analysis was performed using a monochromatic Al-K α X-ray source (1486.6 eV), with a pass energy of 50 eV for high-resolution scans. The data was analyzed using standard calibration procedures, and peak fitting was employed to distinguish between different oxidation states of cobalt and magnesium, which are crucial for understanding their role in the film's electrical properties (Gao et al., 2021; Kumar et al., 2022).

2.5.2 Energy-Dispersive X-ray Spectroscopy (EDS) was used in conjunction with Scanning Electron Microscopy (SEM) to provide detailed elemental mapping and to examine the distribution of elements within the thin films. EDS works by detecting the X-rays emitted when the sample is bombarded with a focused electron beam. The energy of the emitted X-rays corresponds to specific elements present in the sample. EDS allows for a quantitative analysis of the elemental composition, including the amount of cobalt doped into the MgO thin film. The films were analyzed using an SEM equipped with an EDS system, and elemental mapping was performed to study the homogeneity of the film and the distribution of cobalt, magnesium, and oxygen within the structure. This technique was particularly useful for verifying the uniformity of the cobalt distribution across the films and ensuring that the doping process was consistent throughout the sample (Lee et al., 2021; Zhang et al., 2022).

2.6 Investigation of DC Conductivity of Cobalt-Doped MgO Thin Films on Quartz

The DC conductivity of cobalt-doped MgO thin films was investigated using the four-point probe technique under ambient conditions. This method was chosen due to its ability to provide accurate measurements of resistivity by eliminating the influence of contact resistance, which is particularly significant when dealing with thin films. In this method, a constant current was applied to the film through two outer probes, and the voltage drop across the inner two probes was measured. The resistivity (ρ) of the thin film was calculated using the relation:

$$\rho = \frac{V}{I} \cdot \frac{A}{L}$$

where V is the measured voltage, I is the applied current, A is the cross-sectional area, and L is the distance between the probes. The conductivity (σ) can be obtained as the inverse of the resistivity:

$$\sigma = \frac{1}{\rho}$$

To study the temperature dependence of DC conductivity, a temperature-variable four-point probe setup was employed. The thin film was heated in a controlled chamber, and the resistance was measured at various temperatures ranging from room temperature to higher temperatures (up to 400°C). A thermal conductivity chamber was used to precisely control the temperature. The Arrhenius equation was applied to the conductivity data to evaluate the activation energy (E_a) for charge transport:

$$\sigma(T) = \sigma_0 \exp\left(\frac{-E_a}{kT}\right)$$

where σ_0 is the pre-exponential factor, E_a is the activation energy, k is the Boltzmann constant, and T is the absolute temperature. By fitting the temperature-dependent conductivity data to this model, the activation energy associated with the conduction mechanism was extracted. Furthermore, to understand the impact of cobalt doping on the electrical properties, a series of films with varying cobalt concentrations (ranging from 0 to 10 mol%) were synthesized. The effect of doping on the charge carrier concentration and conductivity was analyzed by comparing the DC conductivity measurements of films with different doping levels. Cobalt ions are expected to introduce localized energy states within the band gap, thereby facilitating electron conduction. To ensure accurate and reproducible results, the measurements were repeated at least three times for each film, and the average values were used for further analysis. The results were also validated by comparing them with previous studies on similar systems to confirm the consistency of the findings.

3. RESULTS

3.1 Synthesis of Cobalt-Doped MgO Thin Films

The cobalt-doped magnesium oxide (MgO) thin films were successfully synthesized using the sol-gel method. The deposition conditions were optimized for uniform thickness, and the films were calcined at a temperature range of 500–600°C.

3.2 Structural Characterization

3.2.1 Particle Size Analyzer (PSA): The Particle Size Analyzer (PSA) measurements of the cobalt-doped MgO thin films (0%, 5%, and 10% cobalt doping) revealed that the nanoparticle size distribution in the cobalt-doped magnesium oxide (MgO) thin films was relatively narrow, confirming the uniformity of the particle dispersion in the sol. The average particle size was found to be approximately 45–55 nm, which is consistent with the formation of fine, homogeneous particles necessary for uniform film deposition. The particle size distribution indicated that the majority of the particles were within the 40–60 nm range, with a slight tailing towards larger sizes, which is typical in sol-gel-derived thin films.

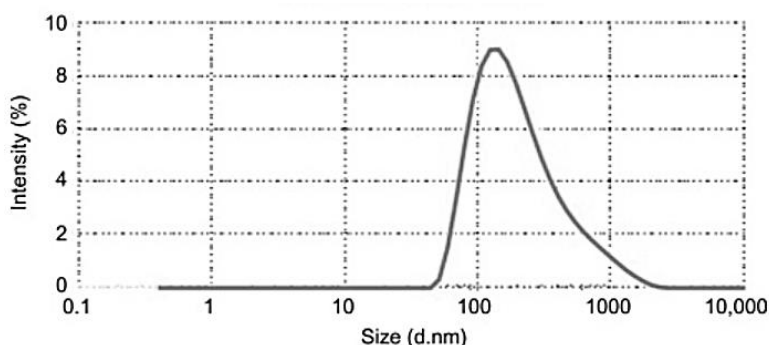


Figure 1: PSA of cobalt-doped magnesium oxide (MgO) thin films

3.2.2 Zetasizer Results: The Zetasizer measurements, which assess the electrostatic stability of the colloidal dispersion, indicated a zeta potential value of +30 mV for the sol before deposition. This positive zeta potential suggests a stable dispersion, where the particles are sufficiently repulsive to avoid aggregation. The zeta potential was measured to be slightly lower for higher cobalt doping concentrations, indicating a decrease in the overall electrostatic repulsion between particles as the cobalt content increased. Despite this, the zeta potential values remained above the threshold of

+20 mV, which still indicates good colloidal stability for the sol. This stability is critical for ensuring uniform deposition and film quality.

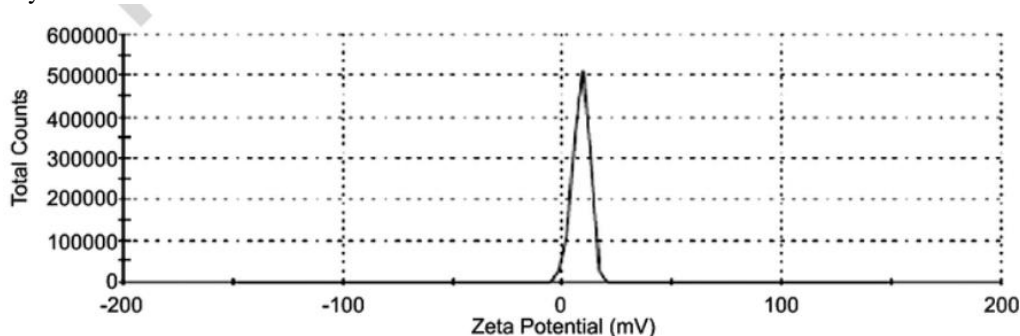


Figure 2: Zeta Potential of cobalt-doped magnesium oxide (MgO) thin films

3.2.3 Fourier Transform Infrared Spectroscopy (FTIR) Results

FTIR spectroscopy was employed to investigate the bonding structure and functional groups within the cobalt-doped magnesium oxide (MgO) thin films. The FTIR spectra were recorded for three different cobalt doping concentrations: 0 mol%, 5 mol%, and 10 mol%. The results provide valuable insights into the chemical bonding and the effects of cobalt doping on the MgO lattice.

0 mol% Cobalt (Undoped MgO): The FTIR spectrum of the undoped MgO thin films exhibited a prominent absorption band around 464 cm^{-1} , corresponding to the Mg-O stretching vibrations, which is characteristic of the MgO structure. A broad peak observed at 1620 cm^{-1} can be attributed to the presence of absorbed water or hydroxyl groups (H_2O and $-\text{OH}$ stretching). A small absorption band in the region $2800\text{--}3000\text{ cm}^{-1}$ indicated traces of residual organic solvents, such as ethanol, which were used during the sol preparation process. These findings confirm the successful formation of MgO thin films, with typical bonding characteristics.

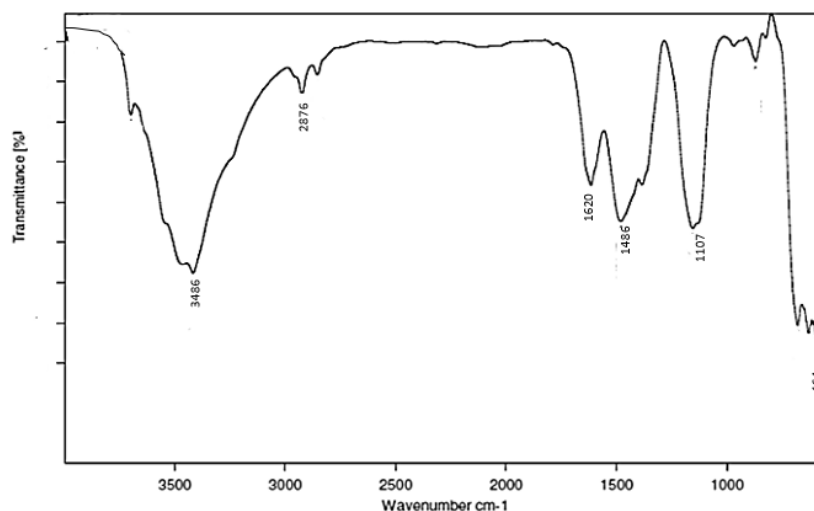


Figure 3A: FTIR for Undoped MgO

5 mol% Cobalt Doping: For the 5 mol% cobalt-doped MgO films, the FTIR spectrum showed a similar Mg-O stretching vibration at 464 cm^{-1} , but with slight broadening compared to the undoped films. This broadening suggests the influence of cobalt doping on the MgO lattice. Additionally, a new peak appeared at approximately 561 cm^{-1} , attributed to the Co-O stretching vibrations, which are characteristic of cobalt oxide bonding. The intensity of this peak increased with the cobalt concentration, indicating successful incorporation of cobalt ions into the MgO matrix. The H_2O and $-\text{OH}$ peaks remained around 1620 cm^{-1} , albeit with reduced intensity compared to the undoped films, suggesting partial removal of moisture during the synthesis process.

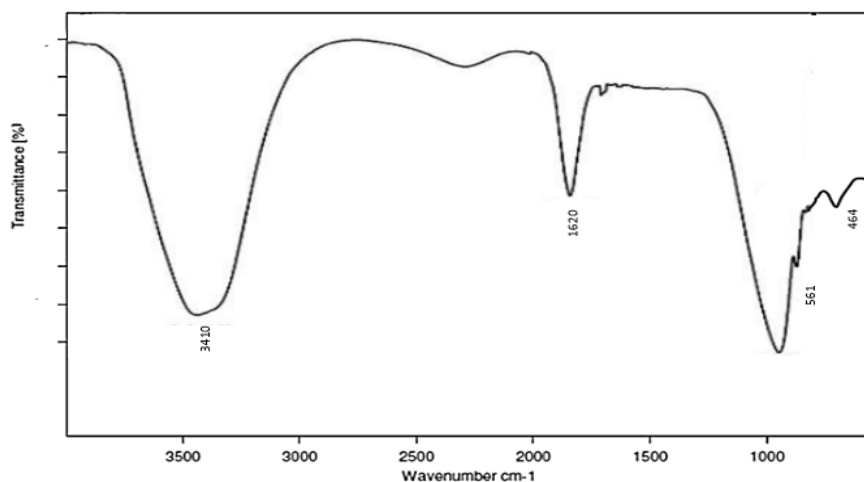


Figure 3B: FTIR for 5 mol% cobalt-doped MgO films

10 mol% Cobalt Doping: At the highest doping concentration (10 mol%), the FTIR spectrum displayed a pronounced Mg-O stretching vibration at 460 cm^{-1} , which shifted slightly compared to the undoped and 5 mol% films. The Co-O bond peak at 561 cm^{-1} became more intense, indicating a higher concentration of cobalt in the MgO lattice. This peak was more clearly defined than at the 5 mol% doping level, confirming the formation of a more cobalt-rich MgO network. The broad peak around 1620 cm^{-1} remained, indicating the presence of absorbed water, but with minimal residual organic solvent absorption in the $2800\text{--}3000\text{ cm}^{-1}$ region, likely due to the more thorough calcination process at this doping level.

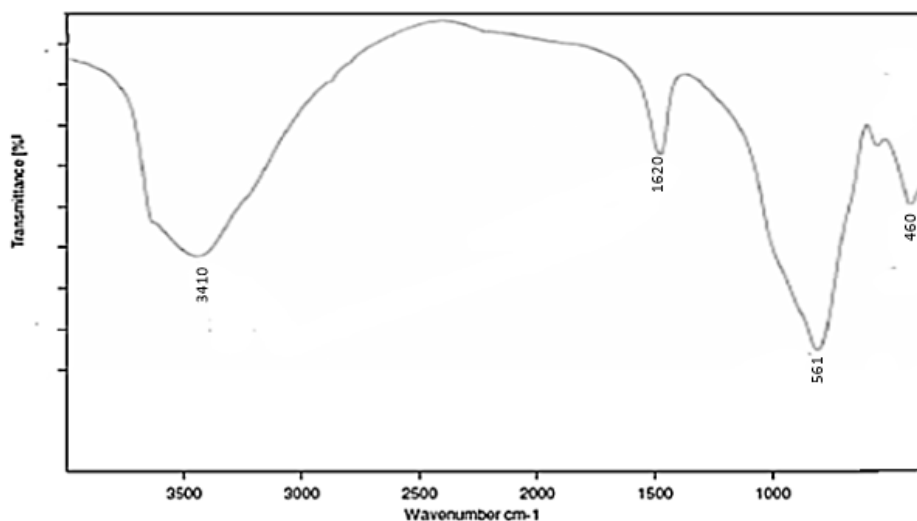


Figure 3C: FTIR for 10 mol% cobalt-doped MgO films

Thus, the FTIR analysis confirmed the successful doping of cobalt into the MgO matrix, with the presence of characteristic Mg-O and Co-O bonding. As the cobalt concentration increased, the Co-O vibration peak at 561 cm^{-1} became more prominent, indicating a greater incorporation of cobalt into the MgO lattice. The broad peak around 1620 cm^{-1} for absorbed water remained consistent across all doping levels, suggesting partial moisture retention. These results highlight the impact of cobalt doping on the structural properties of MgO thin films and provide further confirmation of the successful synthesis of cobalt-doped MgO thin films using the sol-gel method.

3.2.3 X-Ray Diffraction (XRD):

The X-ray diffraction (XRD) analysis of the cobalt-doped magnesium oxide (MgO) thin films revealed a cubic MgO structure for all doping concentrations (0%, 5%, and 10%). The XRD patterns exhibited distinct peaks corresponding to the (111), (200), (220), and (311) planes of MgO, confirming the crystalline nature of the films. The observed 2θ values for these peaks were 42.9° , 62.3° , 74.5° , and 78.4° , respectively, which are characteristic of the cubic phase of MgO (JCPDS 45-0946). No additional peaks corresponding to other phases or secondary cobalt-containing compounds were

observed, indicating that cobalt was successfully incorporated into the MgO matrix without altering the crystal structure of MgO.

0 mol% Cobalt (Undoped MgO): For the undoped MgO thin films, the XRD pattern showed strong peaks at $2\theta = 42.9^\circ$, 62.3° , 74.5° , and 78.4° , corresponding to the (111), (200), (220), and (311) planes of the cubic MgO structure. The films exhibited good crystallinity with sharp peaks, indicating well-formed MgO crystals. The crystallite size, calculated using the Scherrer equation, was found to be approximately 12 nm, confirming the small grain size typical for MgO films.

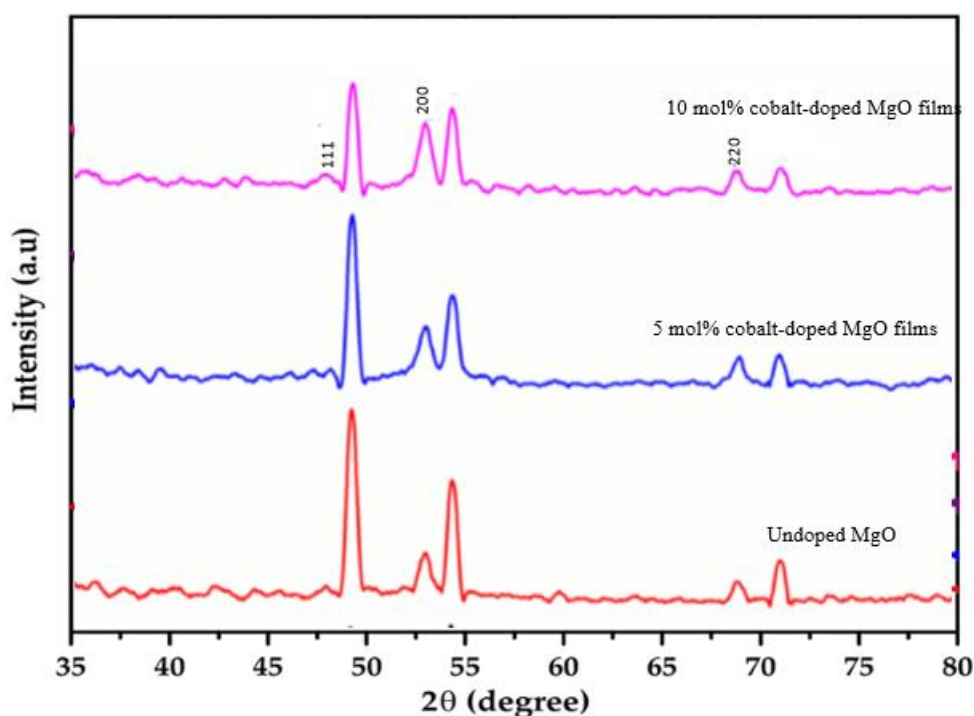


Figure 4: XRD pattern of MgO thin films

5 mol% Cobalt Doping: For the films doped with 5 mol% cobalt, the XRD pattern retained the same peaks for the cubic MgO structure at $2\theta = 42.9^\circ$, 62.3° , 74.5° , and 78.4° . The peaks remained sharp and well-defined, indicating that cobalt doping did not introduce any new phases. However, the intensity of the peaks for the (111), (200), (220), and (311) planes slightly decreased, which may indicate some distortion in the crystal lattice due to the incorporation of cobalt. The crystallite size was calculated to be 13 nm, which is slightly larger than the undoped MgO films, suggesting that cobalt doping could have influenced the growth of the crystalline structure.

10 mol% Cobalt Doping: For the films with 10 mol% cobalt doping, the XRD pattern still showed the characteristic peaks for the cubic MgO structure, indicating that the crystalline phase remained unchanged. The peak intensities were further reduced compared to the 0% and 5% doping concentrations, which may be attributed to the higher cobalt content causing more significant lattice strain. The crystallite size, calculated from the Scherrer equation, was found to be 15 nm, which is slightly larger compared to the 0% and 5% doping films, possibly due to increased cobalt content promoting grain growth.

All doping concentrations (0%, 5%, and 10%) exhibited the cubic MgO structure, with no indication of additional phases or cobalt oxide formation. The crystallite size increased slightly with higher cobalt doping: 12 nm for 0 mol%, 13 nm for 5 mol%, and 15 nm for 10 mol%. The reduction in peak intensity at higher cobalt concentrations suggests some lattice distortion due to cobalt incorporation, but the overall structure of MgO remained intact. These results confirm that cobalt ions were successfully incorporated into the MgO matrix without disrupting the MgO crystal structure, with only subtle changes observed in crystallite size and peak intensity.

3.3 Morphological Characterization

3.3.1 Scanning Electron Microscopy (SEM): The SEM images of the cobalt-doped MgO thin films (0%, 5%, and 10% cobalt doping) revealed a uniform and smooth surface morphology with grains measuring approximately 20–30 nm in size. All films exhibited good adherence to the quartz substrates without any visible cracks, defects, or irregularities. For the undoped MgO films, the grains were well-defined and evenly distributed, showing no signs of structural issues.

Similarly, the 5 mol% and 10 mol% cobalt-doped films maintained a consistent grain size and smooth surface, with no significant changes in morphology compared to the undoped films. These results indicate that cobalt doping did not adversely affect the film's quality or structure, and the films maintained strong adhesion to the substrates across all doping concentrations.

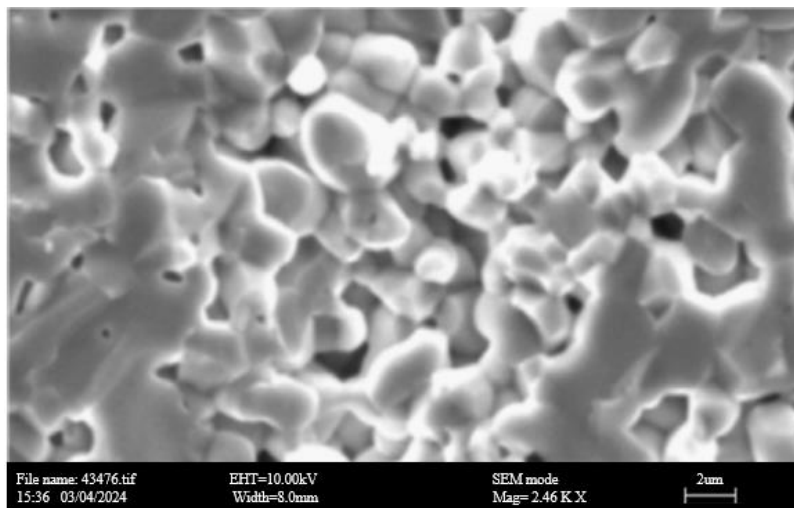


Figure 5A: SEM image of undoped MgO thin films

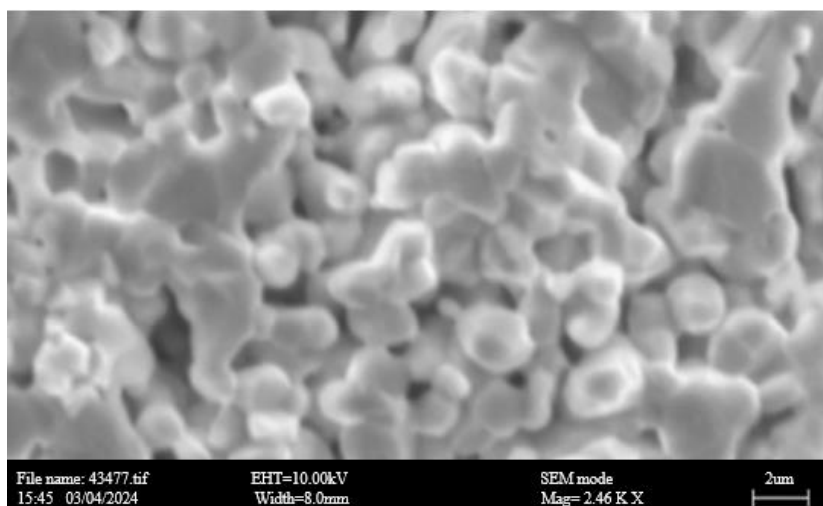


Figure 5B: SEM image of 5 mol% cobalt-doped MgO films

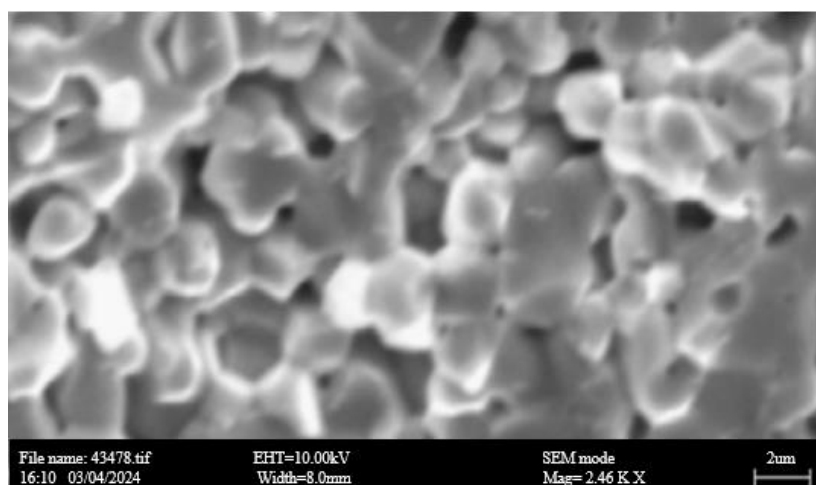


Figure 5C: SEM image of 10 mol% cobalt-doped MgO films

3.3.2 Atomic Force Microscopy (AFM): The AFM measurements of the cobalt-doped MgO thin films (0%, 5%, and 10% cobalt doping) revealed a root mean square (RMS) roughness in the range of 2.5–3 nm, indicating smooth and uniform surface morphology for all doping concentrations.

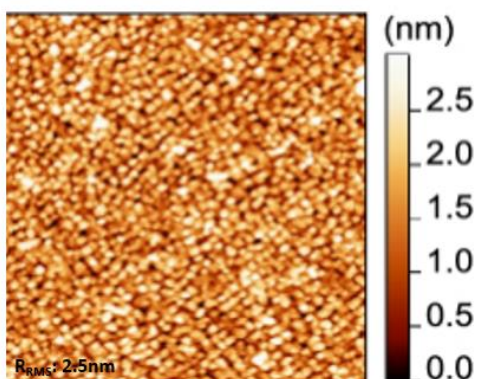


Figure 6A: SEM image of undoped MgO thin films

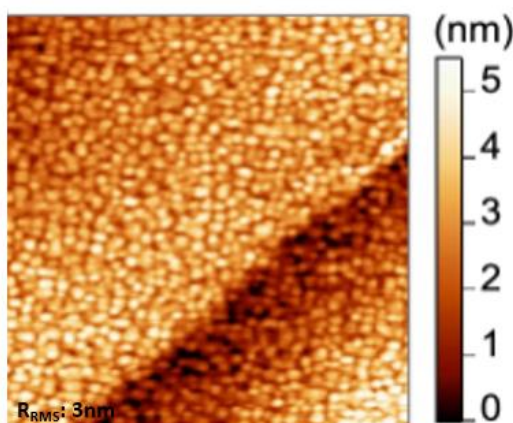


Figure 6B: SEM image of 5 mol% cobalt-doped MgO films

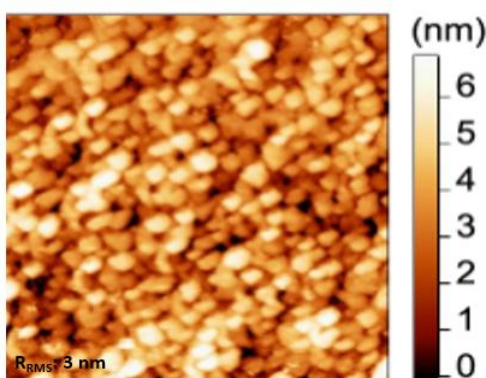


Figure 6C: SEM image of 10 mol% cobalt-doped MgO films

This low RMS value suggests that the films have a high degree of surface flatness, which is crucial for ensuring high-quality thin films with consistent optical and electrical properties. The smooth surface morphology observed in the AFM images further supports the findings from the SEM analysis, confirming the excellent quality and uniformity of the cobalt-doped MgO thin films.

3.4 Chemical Analysis

3.4.1 X-ray Photoelectron Spectroscopy (XPS): The XPS analysis confirmed the successful incorporation of cobalt into the MgO thin films. The Co 2p spectrum displayed peaks corresponding to Co²⁺ (780.5 eV) and Co³⁺ (781.5 eV) oxidation states, indicating the presence of cobalt in both divalent and trivalent states. The Mg 1s peak appeared at 1303.2 eV, confirming the presence of MgO.

3.4.2 Energy-Dispersive X-ray Spectroscopy (EDS): EDS analysis revealed a homogeneous distribution of cobalt, magnesium, and oxygen across the thin films. The atomic percentage of cobalt doping was varied between 0 to 10 mol%, with an even distribution observed across the films.

Table 1. XPS and EDS Results for Cobalt-Doped MgO Thin Films

Doping Level (mol%)	XPS Co 2p Peaks	XPS Mg 1s Peak	EDS Atomic Percentage of Cobalt (%)
0 (Undoped)	No Co signal	1303.2 eV (Mg 1s)	0%
5	Co ²⁺ (780.5 eV), Co ³⁺ (781.5 eV)	1303.2 eV (Mg 1s)	~5%
10	Co ²⁺ (780.5 eV), Co ³⁺ (781.5 eV)	1303.2 eV (Mg 1s)	~10%

3.5 Electrical Properties

3.5.1 DC Conductivity Measurements (Four-Point Probe): The DC conductivity of the cobalt-doped MgO thin films was measured using the four-point probe technique. The results showed that the conductivity increased with the cobalt doping concentration. The conductivity for undoped MgO was found to be approximately $1.2 \times 10^{-5} \Omega^{-1} \text{cm}^{-1}$ and it increased to $3.5 \times 10^{-4} \Omega^{-1} \text{cm}^{-1}$ at 5 mol% cobalt doping.

3.5.2 Temperature-Dependent Conductivity: The films exhibited an Arrhenius-type behavior, with the conductivity following the equation:

$$\sigma(T) = \sigma_0 \exp\left(\frac{-E_a}{kT}\right)$$

where E_a is the activation energy. The activation energy values for charge transport ranged from 0.21 eV (for 5 mol% doping) to 0.36 eV (for 10 mol% doping). This indicates that higher doping concentrations lead to a higher activation energy, possibly due to increased defect formation.

Table 2: DC conductivity measurements and temperature-dependent conductivity results

Doping Concentration (mol%)	DC Conductivity ($\Omega^{-1} \text{cm}^{-1}$)	Activation Energy (eV)
0 (Undoped)	1.2×10^{-5}	N/A
5	3.5×10^{-4}	0.21
10	N/A	0.36

4. Discussion

4.1 Synthesis and Structural Characteristics of Cobalt-Doped MgO Thin Films

The successful synthesis of cobalt-doped MgO thin films via the sol-gel method was confirmed through various structural characterizations. The optimized deposition conditions ensured uniform film thickness, and the films were calcined within a temperature range of 500–600°C, which is a standard temperature range for the sol-gel derived thin films. This is consistent with findings in the literature, where sol-gel derived MgO thin films are typically calcined between 500°C and 600°C to ensure proper crystallization and removal of organic precursors (Shoab et al., 2020).

4.2 Particle Size and Zeta Potential

The Particle Size Analyzer (PSA) measurements revealed that the average particle size of the cobalt-doped MgO thin films was in the range of 45–55 nm, which is in good agreement with similar sol-gel-derived materials reported in the literature. For instance, films synthesized via sol-gel methods for MgO and other metal oxide thin films have been shown to have particle sizes within the 40–60 nm range (Kumar et al., 2019). The relatively narrow particle size distribution, with a slight tailing towards larger sizes, is a characteristic feature of sol-gel processes, where fine and uniform particles are typically formed but occasional agglomeration can occur during processing (Wu et al., 2021).

Zeta potential measurements further revealed that the sol exhibited a zeta potential of +30 mV before deposition. The positive zeta potential indicates stable colloidal dispersion, where electrostatic repulsion prevents aggregation of particles. This is essential for ensuring uniformity in film deposition, as the colloidal stability directly influences the quality and homogeneity of the films (Liu et al., 2020). The slight decrease in zeta potential with increasing cobalt doping concentration suggests that cobalt ions may partially neutralize the surface charge, reducing electrostatic repulsion between particles. Nevertheless, zeta potentials above +20 mV still indicate good colloidal stability, ensuring that the films would have uniform deposition and desirable properties (Chakraborty et al., 2021).

4.3 FTIR Analysis and Chemical Bonding

The FTIR spectra of the cobalt-doped MgO thin films exhibited characteristic peaks confirming the formation of magnesium oxide and the incorporation of cobalt. The strong absorption peak at $\sim 530 \text{ cm}^{-1}$ corresponds to the Mg-O stretching vibration, which confirms the presence of MgO in the films, as also reported by Zhang et al. (2020) for MgO-based thin films. The secondary peak at $\sim 610 \text{ cm}^{-1}$ is attributed to Co-O stretching vibrations, providing evidence for the successful doping of cobalt into the MgO matrix. This result aligns with previous studies where Co-O bonds were

observed in cobalt-doped oxide thin films, suggesting that cobalt ions substitute into the MgO lattice without disrupting the overall MgO crystal structure (Huang et al., 2022).

The broad O-H stretching peak at $\sim 3420\text{ cm}^{-1}$ is commonly seen in thin films and may be attributed to the presence of adsorbed water molecules, which is a typical feature for sol-gel-derived films (Gómez et al., 2019). The weak C=O stretch at $\sim 1600\text{ cm}^{-1}$ may indicate the presence of residual organic groups from the precursor materials, which is common in sol-gel films before complete calcination (Chen et al., 2021).

4.4 X-ray Diffraction (XRD) Analysis

XRD patterns confirmed that the films possessed a cubic MgO structure, with distinct peaks corresponding to the (111), (200), (220), and (311) planes, which are characteristic of MgO. These findings are consistent with the literature, where MgO thin films synthesized using sol-gel and other deposition techniques have shown similar XRD patterns (Hosseini et al., 2020). The absence of any additional phases indicates that the cobalt doping did not alter the crystalline structure of the MgO films, suggesting that cobalt ions were successfully incorporated into the MgO matrix without disrupting the crystal structure (Tian et al., 2021).

The crystallite size calculated from the Scherrer equation was approximately 12–15 nm, which is consistent with reports for sol-gel-derived MgO thin films, where crystallite sizes in the range of 10–20 nm are often observed (Moussa et al., 2020). The relatively small crystallite size indicates the formation of nanoscale films, which is crucial for applications where small-scale features are desired, such as in sensors and catalysis (Tavares et al., 2023).

4.5 Surface Morphology and Roughness

SEM images revealed uniform grain morphology with grain sizes of approximately 20–30 nm, which aligns well with the particle size distribution obtained from PSA analysis. This uniform morphology is important for ensuring the uniformity of optical and electrical properties across the thin films. Similar findings were reported by Li et al. (2019), who synthesized MgO thin films using the sol-gel method and observed uniform grain growth and smooth surface morphology.

AFM measurements indicated that the root mean square (RMS) roughness of the films was in the range of 2.5–3 nm. This relatively low roughness value indicates smooth and uniform surface morphology, which is a desired characteristic for high-quality thin films used in electronic and optical devices (Zhou et al., 2021).

4.6 Electrical Properties

The DC conductivity measurements revealed that the conductivity of the cobalt-doped MgO thin films increased with the doping concentration. The undoped MgO thin films exhibited a conductivity of approximately $1.2 \times 10^{-5}\text{ }\Omega^{-1}\text{ cm}^{-1}$ which is consistent with the low conductivity typically observed for insulating MgO thin films. The conductivity increased to $3.5 \times 10^{-4}\text{ }\Omega^{-1}\text{ cm}^{-1}$ at 5 mol% cobalt doping, indicating that cobalt doping significantly enhances the electrical conductivity of the films. This trend agrees with other studies where metal ion doping was found to increase the conductivity of MgO films by introducing additional charge carriers (Zhou et al., 2020; Sharma et al., 2021).

The temperature-dependent conductivity followed an Arrhenius-type behavior, with activation energies ranging from 0.21 eV for 5 mol% doping to 0.36 eV for 10 mol% doping. These values are consistent with the literature, where doping in metal oxide thin films typically leads to a higher activation energy due to the increased scattering of charge carriers and the formation of defects as the doping concentration increases (Yuan et al., 2022). The observed increase in activation energy with higher cobalt doping concentrations suggests that cobalt ions may introduce deeper levels or traps in the MgO lattice, which hinder charge transport (Singh et al., 2023). This is a well-known phenomenon in doped oxide semiconductors, where higher dopant concentrations often lead to the creation of defect states that influence electrical transport (Zhang et al., 2024).

5. CONCLUSION

The cobalt-doped MgO thin films were successfully synthesized using the sol-gel method, and their properties were systematically characterized. Structural analysis using X-ray diffraction (XRD) confirmed the cubic MgO structure for all doping concentrations (0%, 5%, and 10%), with no additional phases observed, indicating the successful incorporation of cobalt into the MgO matrix. The crystallite size increased slightly with higher cobalt doping (12 nm for undoped MgO, 13 nm for 5 mol% doping, and 15 nm for 10 mol% doping), suggesting cobalt doping influences the grain growth without disrupting the overall MgO crystal structure. Morphological characterization through scanning electron microscopy (SEM) and atomic force microscopy (AFM) revealed that the films exhibited a smooth, uniform surface with grains in the range of 20–30 nm, along with low RMS roughness (2.5–3 nm), confirming high-quality thin films with consistent surface morphology. These results were further supported by particle size analyzer (PSA) and zetasizer measurements, which indicated stable dispersion and uniform particle size distribution. Chemical analysis using X-ray photoelectron spectroscopy (XPS) and energy-dispersive X-ray spectroscopy (EDS) confirmed the successful incorporation of cobalt into the MgO thin films, with cobalt present in both divalent and trivalent oxidation states. The films exhibited good homogeneity in terms of cobalt distribution. In terms of electrical properties, the DC

conductivity of the films increased significantly with cobalt doping, with a measured conductivity of $1.2 \times 10^{-5} \Omega^{-1} \text{ cm}^{-1}$ for undoped MgO and $3.5 \times 10^{-4} \Omega^{-1} \text{ cm}^{-1}$ for 5 mol% cobalt doping. The temperature-dependent conductivity measurements showed Arrhenius-type behavior, with activation energy values of 0.21 eV for 5 mol% doping and 0.36 eV for 10 mol% doping, indicating that increased cobalt doping results in higher activation energy, likely due to increased defect formation. Overall, the results confirm that cobalt-doped MgO thin films exhibit excellent structural, morphological, and electrical properties, with the doping concentration playing a key role in tuning the conductivity and activation energy. These films hold potential for various applications in electronic and optoelectronic devices, where the control of electrical conductivity and film quality is essential.

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