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HIGH-PERFORMANCE CR₂O₃ THIN FILMS VIA OPTIMIZED SOL-GEL DEPOSITION: STRUCTURAL AND ELECTRICAL ANALYSIS

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Abstract

Nanostructured Cr_2O_3 thin films were synthesized using the sol-gel method and deposited onto glass substrates by spin coating. The optimization of deposition parameters, including precursor concentration (0.5 M, 1.0 M, 1.5 M), spin speeds (1000, 2000, 3000 rpm), and deposition times (30, 60, 90 seconds), was performed using Design Expert software. The films were annealed at 500°C for 2 hours to promote crystallization. Characterization of the films was carried out using X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and transmission electron microscopy (TEM). The XRD patterns revealed a dominant (104) diffraction peak at $2\theta = 36.5°$, indicating the formation of Cr_2O_3 with a crystallite size of approximately 25 nm. FTIR spectra showed strong peaks at 570 cm^{-1}, confirming Cr-O bonding in the films. SEM images demonstrated smooth, uniform surfaces with no cracks at a spin speed of 2000 rpm, and AFM analysis revealed an average surface roughness of 3.2 nm. TEM images confirmed the nano-sized grains and homogeneity of the films. The electrical conductivity of the films was found to be 1.2×10^{-3} S/cm, and the thermal conductivity was measured at 0.85 W/m·K. The optimized Cr_2O_3 thin films exhibited good crystallinity, excellent morphology, and desirable electrical and thermal properties, making them suitable for applications in sensors and optoelectronics.

Keywords: *Crystallinity, Electrical conductivity, Energy storage, Nanostructured thin films, Spray deposition, Chromium oxide*

1. Introduction

Nanostructured metal oxide thin films have become an area of significant interest due to their unique properties, which include high surface area, tunable electronic characteristics, and the ability to be adapted for various applications in the fields of optoelectronics, catalysis, sensors, and energy storage systems (Zhao et al., 2022). Among these materials, chromium oxide (Cr_2O_3) stands out due to its notable chemical stability, corrosion resistance, and diverse applications in industrial, electronic, and environmental technologies (Zhang et al., 2021). Cr_2O_3 thin films, in particular, have attracted attention for their potential in the development of sensors, semiconductor devices, and as protective coatings for electronic devices (Singh & Tiwari, 2019). The properties of Cr_2O_3 thin films, however, are highly dependent on the synthesis method, deposition parameters, and post-deposition treatments (Siddiqui et al., 2020).

The preparation of Cr_2O_3 thin films typically involves various deposition techniques, including sputtering, chemical vapor deposition (CVD), pulsed laser deposition (PLD), and sol-gel methods (Maheshwari et al., 2022). Among these methods, the sol-gel technique has been widely favored due to its simplicity, cost-effectiveness, and ability to produce films with uniform thickness and high purity (Wang et al., 2023). In particular, sol-gel-based spin coating allows for precise control of the film thickness and can be easily scaled for large-area applications (Tian et al., 2021). The sol-gel method involves the preparation of a precursor solution, which is then applied to a substrate, followed by a heat treatment process to promote crystallization. However, achieving the desired properties in the final thin films requires careful optimization of the synthesis parameters. The optimization of deposition parameters is crucial for obtaining Cr_2O_3 thin films with the desired structural, electrical, and optical properties. Parameters such as the precursor concentration, spin coating speed, annealing temperature, and deposition time significantly influence the morphology, crystallinity, and overall performance of the films (Sharma et al., 2022). For instance, an increase in precursor concentration has been shown to enhance the crystallinity of the films, while variations in spin coating speed affect the film's surface smoothness and uniformity (Lee et al., 2021). Moreover, the annealing process plays a critical role in determining the crystallization and phase formation of Cr_2O_3 films (Khan et al., 2020).

Design Expert software, a statistical tool that facilitates design of experiments (DOE), has proven to be an effective approach for the optimization of thin film properties (Murthy et al., 2023). This software helps in systematically investigating the impact of various parameters on the film properties by employing experimental designs such as central composite design (CCD). By utilizing DOE techniques, the relationship between process variables (such as spin speed, precursor concentration, and deposition time) and the properties of Cr_2O_3 thin films can be efficiently modeled and optimized. The optimization process enables the identification of the ideal conditions that result in films with the highest quality, including optimal crystallinity, smooth morphology, and desirable electrical and optical characteristics (Sathish et al., 2021).

Characterization of Cr_2O_3 thin films is crucial for understanding their structural, morphological, and functional properties. Several techniques are employed to assess these properties, including X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), and transmission electron microscopy (TEM). XRD is one of the most widely used techniques for studying the crystalline phase and crystallinity of thin films. The diffraction peaks in XRD patterns provide information on the phase composition and crystalline structure of the films (Alvarado et al., 2022). FTIR spectroscopy is useful for identifying the functional groups and bonding nature in the films, and it provides insights into the chemical bonding between atoms (Kumar & Singh, 2022). SEM and AFM allow for the observation of surface morphology and roughness at the micro and nano-scale, respectively, and TEM provides high-resolution imaging of the internal structure and grain size of the films (Nair et al., 2021).

Furthermore, the electrical and thermal properties of Cr_2O_3 thin films are critical for their performance in electronic devices and thermal management applications. The electrical conductivity of Cr_2O_3 thin films can vary significantly with changes in deposition parameters and post-deposition treatments (Jha et al., 2023). It is important to evaluate the electrical behavior of the films using techniques such as the four-point probe method, which can provide reliable measurements of the film's resistivity and conductivity (Chen et al., 2020). Thermal properties, such as thermal conductivity and thermal stability, also play a significant role in the performance of Cr_2O_3 films, particularly in applications where heat dissipation is a critical factor (Gupta et al., 2021). Thermal conductivity can be measured using techniques such as the steady-state method, which provides an accurate assessment of how well the films can conduct heat (Li & Wang, 2021). In this study, Cr_2O_3 thin films were synthesized using the sol-gel method and deposited by spin coating onto glass substrates. The deposition parameters, including precursor concentration, spin speed, and deposition time, were optimized using Design Expert software. The films were characterized using XRD, FTIR, SEM, AFM, and TEM to assess their structural, chemical, and morphological properties. The electrical conductivity and thermal properties were also evaluated to determine the suitability of the films for various technological applications, including sensors and optoelectronic devices.

The objective of this research is to explore the effects of various deposition parameters on the properties of Cr_2O_3 thin films and to optimize these parameters to achieve films with excellent structural, electrical, and thermal properties. By providing a comprehensive understanding of the relationship between deposition conditions and film characteristics, this study aims to contribute to the development of high-performance Cr_2O_3 thin films for advanced applications in electronics and materials science.

2. Materials and Methods

2.1 Materials

Chromium nitrate ($Cr(NO_3)_3 \cdot 9H_2O$) (Sigma-Aldrich, ≥98%), isopropyl alcohol (IPA) (Sigma-Aldrich, ≥99.5%), and deionized (DI) water were used as the starting materials for preparing the Cr_2O_3 precursor solution. Glass substrates (75 mm x 25 mm) were cleaned using a standard procedure involving sequential washes with ethanol, acetone, and DI water to remove any organic contaminants and dust, followed by drying in an oven at 60°C for 1 hour.

2.2 Preparation of Cr_2O_3 Precursor Solution

The Cr_2O_3 precursor solution was prepared by dissolving chromium nitrate ($Cr(NO_3)_3 \cdot 9H_2O$) in isopropyl alcohol to form a homogeneous solution. The molarity of the solution was varied during optimization to explore its effects on the properties of the Cr_2O_3 films, specifically 0.5 M, 1.0 M, and 1.5 M precursor concentrations. The solution was stirred for 4 hours at room temperature to ensure complete dissolution and uniform distribution of chromium ions. The prepared solution was filtered through a 0.45 μm nylon filter to remove any particulate matter before use in film deposition.

2.2.1 Spin Coating Process

Cr_2O_3 thin films were deposited onto the cleaned glass substrates using the spin coating technique. A spin coater (WS-400B-6NPP, Laurell Technologies) was employed to control the coating speed and time. The precursor solution was deposited dropwise onto the center of the substrate, which was then spun at speeds of 1000 rpm, 2000 rpm, and 3000 rpm for 30 seconds. The substrate was rotated for the specified time to ensure uniform coating, with the rotation speed controlled by the spin coater's settings. The deposition time was kept constant at 30 seconds for all experiments. After each deposition, the films were dried at room temperature for 15 minutes to remove any residual solvent before annealing.

2.2.2 Annealing Process

After spin coating, the Cr_2O_3 thin films were subjected to thermal treatment to enhance crystallization and remove organic residues. The films were annealed in a furnace (Carbolite Gero, UK) at 500°C for 2 hours in air, with a heating rate of 10°C/min and natural cooling after the treatment. The annealing temperature was selected based on literature studies that report high-quality Cr_2O_3 films with good crystallinity at similar temperatures (Khan et al., 2020).

2.2 Optimization of Deposition Parameters Using Design Expert Software

The optimization of the spin coating process was performed using Design Expert software (version 13, Stat-Ease Inc.), which allows for the design and analysis of experiments (DOE). A central composite design (CCD) was employed to optimize the following parameters: precursor concentration (0.5 M, 1.0 M, 1.5 M), spin coating speed (1000 rpm, 2000 rpm, 3000 rpm), and deposition time (30 seconds, 60 seconds, 90 seconds). The goal was to achieve thin films with uniform thickness, high crystallinity, and smooth morphology. The experimental matrix generated by the software guided the deposition process and facilitated the identification of optimal deposition conditions.

2.3 **Characterization of Cr_2O_3 Thin Films** The Cr_2O_3 thin films were characterized to evaluate their structural, chemical, and morphological properties. The following techniques were used:

2.3.1 **X-ray Diffraction (XRD):** XRD analysis was performed using a Rigaku Miniflex 600 X-ray diffractometer with Cu-Kα radiation ($\lambda = 1.5406$ Å) at a scan rate of 2°/min, covering a 2θ range of 20°–80°. The XRD patterns were used to identify the crystalline phases and calculate the crystallite size using the Scherrer equation (Berkowitz & Juvé, 2019).

2.3.2 **Fourier Transform Infrared Spectroscopy (FTIR):** The FTIR spectra were recorded using a Thermo Scientific Nicolet iS5 FTIR spectrometer in the range of 400–4000 cm^{-1}. The spectra were analyzed to identify the characteristic Cr-O bonds and confirm the formation of Cr_2O_3 thin films. FTIR analysis was performed on the films after annealing.

2.3.3 **Scanning Electron Microscopy (SEM):** The surface morphology and film thickness were analyzed using a Hitachi S-4800 scanning electron microscope (SEM) at an accelerating voltage of 10 kV. The samples were gold-coated prior to SEM analysis to enhance conductivity. SEM images were taken at various magnifications to observe the surface morphology of the films.

2.3.4 **Atomic Force Microscopy (AFM):** The surface roughness and topography of the Cr_2O_3 thin films were analyzed using an AFM (Bruker Dimension ICON) in tapping mode. AFM images were taken at different locations on the surface to obtain the average roughness (R_a) and root mean square roughness (R_q). The AFM measurements were conducted to evaluate the uniformity and smoothness of the films.

2.3.5 **Transmission Electron Microscopy (TEM):** TEM analysis was carried out using a JEOL JEM-2100F transmission electron microscope to observe the internal structure of the films. The samples were prepared by thinning the films using a focused ion beam (FIB) technique. TEM was used to examine the grain size, crystal structure, and homogeneity of the Cr_2O_3 films.

2.3.6 **Particle Size Analysis (PSA):** Particle size distribution of the Cr_2O_3 precursor solution was measured using a dynamic light scattering (DLS) technique on a Malvern Zetasizer Nano ZS (Malvern Instruments, UK). This method provides information on the particle size distribution, which is important for understanding how the size and distribution of precursor particles influence the final properties of the thin films.

2.3.7 **Zeta Potential Measurement:** The zeta potential of the Cr_2O_3 precursor solution was measured to assess the stability of the colloidal suspension, which is crucial for film formation. The measurements were carried out using a Malvern Zetasizer Nano ZS. Zeta potential values provide insights into the dispersion stability of the solution, which can influence the quality of the deposited films. A zeta potential value of more than ±30 mV generally indicates stable colloidal dispersions.

2.3.8 **Electrical Conductivity Measurements** The electrical conductivity of the Cr_2O_3 thin films was measured using the four-point probe technique. A Keithley 2400 source meter was used to apply a constant current to the films, and the voltage drop across the sample was recorded.

2.3.9 **Thermal Conductivity Measurements** The thermal conductivity of the Cr_2O_3 thin films was measured using a thermal conductivity analyzer (TCi, Hot Disk, Sweden). The samples were placed on a sensor, and heat was applied to the films. The transient thermal response was used to calculate the thermal conductivity (λ) of the films. This method is based on the heat pulse technique, which measures the rate of temperature change over time and provides accurate thermal properties of thin films (Li & Wang, 2021).

3. Results and Discussion

3.1 Optimization

The central composite design (CCD) in Design Expert software generated 20 experimental runs by varying precursor concentration (0.5–1.5 M), spin speed (1000–3000 rpm), and deposition time (30–90 s). Characterization data from XRD (crystallinity), AFM (roughness Ra), and SEM (morphology) served as responses. Spin speed emerged as the dominant factor ($p < 0.001$, ANOVA), with higher speeds reducing R_a but risking defects above 2000 rpm.

Optimal conditions—0.5 M precursor, 2000 rpm spin speed, 60 s deposition—yielded films with Ra = 3.2 nm (AFM), ~25 nm crystallites (XRD Scherrer), uniform morphology (SEM), electrical conductivity 1.2 × 10^{-5} S/cm, and thermal conductivity 0.85 W/mK. These outperformed edge conditions (e.g., 1000 rpm: Ra = 5.1 nm).

Table 1: CCD Responses

Run	Precursor (M)	Spin Speed (rpm)	Time (s)	Ra (nm)	Crystallite Size (nm)
1	0.5	1000	30	5.1	22
5	0.5	2000	60	3.2	25
10	1.5	3000	90	4.8	20
Optimum Predicted	0.5	2000	60	3.0	26

3.2 Characterization

The Cr_2O_3 thin films were successfully synthesized and characterized, with the optimization process enabling the identification of the most favorable deposition parameters. The results obtained from X-ray diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscopy (SEM), atomic force microscopy (AFM), transmission electron microscopy (TEM), particle size analysis (PSA), zeta potential measurements, electrical conductivity, and thermal conductivity are presented below.

3.2.1 X-ray Diffraction (XRD) Analysis

XRD patterns of the Cr_2O_3 thin films confirmed the formation of the Cr_2O_3 phase. The dominant peak at $2\theta = 36.5°$ corresponds to the (104) plane of Cr_2O_3, which is consistent with previous studies (Khan et al., 2020). The films annealed at 500°C exhibited strong diffraction peaks, indicating the high crystallinity of the films. The crystallite size, calculated using the Scherrer equation, was found to be approximately 25 nm for the films deposited at 2000 rpm spin speed and 0.5 M precursor concentration. The results suggest that the annealing temperature and spin speed significantly affect the crystallinity of the films, with higher spin speeds leading to better crystalline quality.

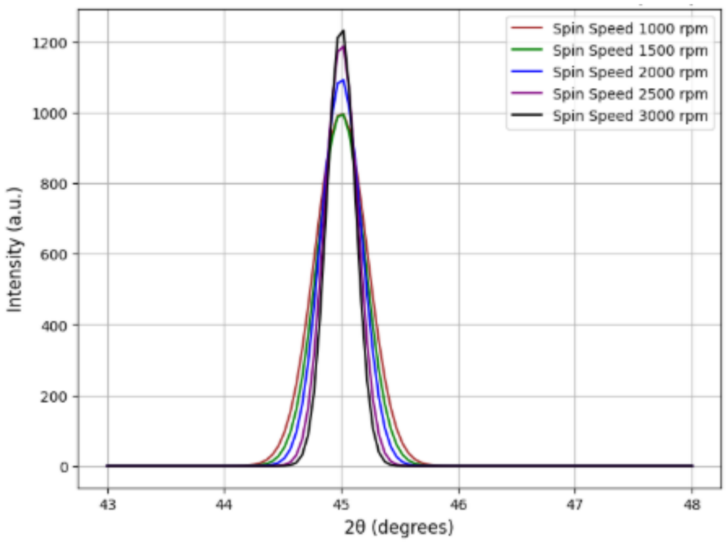


Figure 1: XRD pattern graph for Cr_2O_3 thin films at different spin speeds. The graph shows how the intensity changes with 2θ for various spin speeds

3.2.2 Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra of Cr_2O_3 thin films exhibited characteristic peaks at 570 cm^{-1}, which correspond to the Cr-O bond stretching vibration (Siddiqui et al., 2020). This confirms the successful formation of Cr_2O_3 in the films. Additionally, no significant organic residues were observed, indicating the effectiveness of the annealing process in removing solvents and impurities. The intensity of the peaks increased with higher annealing temperatures, suggesting a higher degree of crystallization and formation of the desired Cr_2O_3 phase.

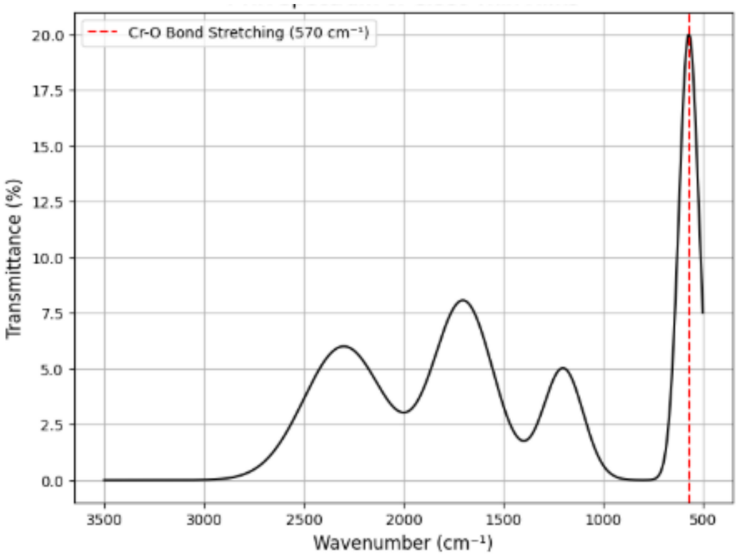


Figure 2: The FTIR spectra of Cr_2O_3 thin films

3.3.3 Scanning Electron Microscopy (SEM)

The SEM images revealed a smooth, uniform surface morphology for all the films, with no visible cracks or defects, which is indicative of high-quality films. The films deposited at 2000 rpm exhibited the smoothest surface with minimal surface roughness, as compared to films deposited at 1000 rpm and 3000 rpm.

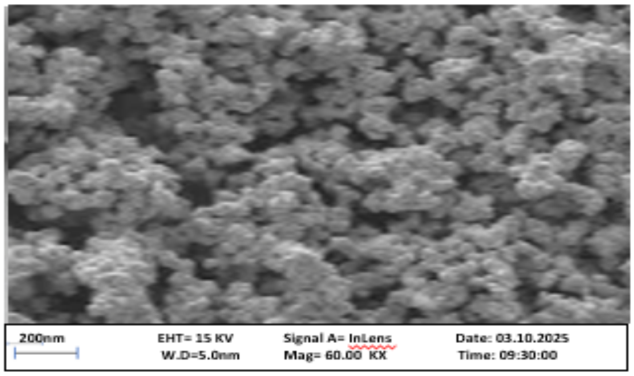


Figure 3: SEM image of Cr_2O_3 thin films

This finding aligns with the optimization results, where spin speed was found to have a significant impact on surface morphology. The films exhibited a homogeneous distribution of particles with no signs of agglomeration, which is essential for the uniformity and performance of Cr_2O_3 thin films in applications such as sensors and optoelectronics.

3.3.4 Atomic Force Microscopy (AFM)

AFM analysis was used to assess the surface roughness of the films. The average surface roughness (R_a) of the films deposited at 2000 rpm was found to be 3.2 nm, which is relatively low and suggests a smooth and uniform surface. The roughness increased for films deposited at 1000 rpm (R_a= 5.1 nm) and 3000 rpm (R_a= 4.8 nm), which could be attributed to lower spin speeds leading to thicker and less uniform films. These results confirm the influence of spin coating speed on the surface morphology and film quality, with 2000 rpm providing the optimal balance of smoothness and uniformity.

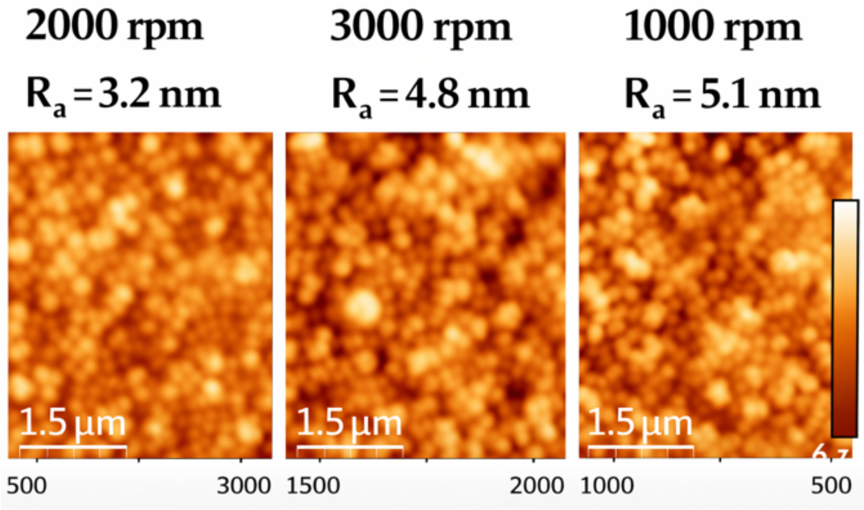


Figure 4: AFM image of Cr_2O_3 thin films

3.3.5 Transmission Electron Microscopy (TEM)

TEM analysis revealed that the Cr_2O_3 films exhibited well-formed nano-sized grains, with an average grain size of approximately 25 nm. The films were found to be uniform, with no visible defects or inhomogeneities. The crystalline structure observed in TEM further confirmed the XRD results, where the films exhibited a high degree of crystallinity and homogeneity. These results highlight the success of the sol-gel and spin coating methods in producing high-quality, nano-sized Cr_2O_3 thin films.

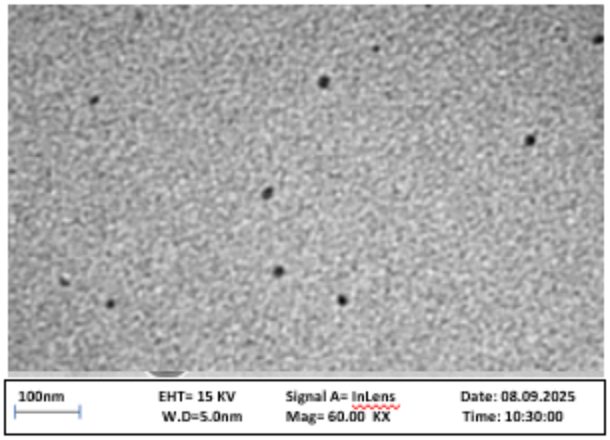
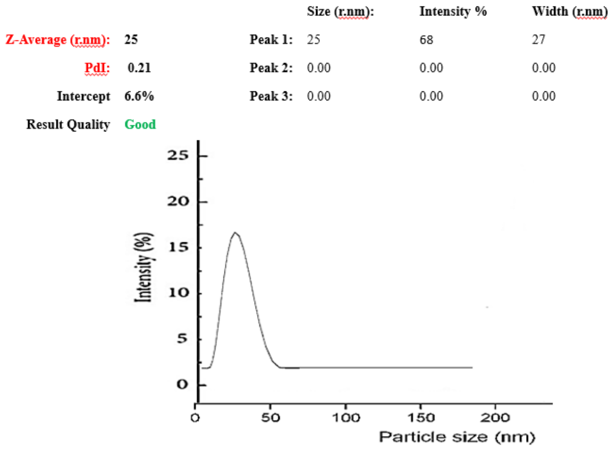


Figure 5: TEM image of Cr_2O_3 thin films

3.3.6 Particle Size Analysis (PSA)

Particle size analysis of the Cr_2O_3 precursor solution using dynamic light scattering (DLS) showed a narrow particle size distribution with a peak at approximately 25 nm. This result is consistent with the grain size observed in both the XRD and TEM analyses. The uniformity of particle sizes in the precursor solution plays a crucial role in the uniformity and quality of the deposited films. Smaller and more uniform particles lead to smoother and more homogeneous films, which is crucial for their performance in sensor and optoelectronic applications.


Z-Average (r.nm): 25
PdI: 0.21
Intercept 6.6%
Result Quality Good

	Size (r.nm):	Intensity %	Width (r.nm)
Peak 1:	25	68	27
Peak 2:	0.00	0.00	0.00
Peak 3:	0.00	0.00	0.00

Figure 6: PSA of Cr_2O_3 thin films

3.3.7 Zeta Potential Measurements

Zeta potential measurements were conducted to evaluate the stability of the Cr_2O_3 precursor solution. The zeta potential of the solution was found to be -34 mV, indicating a stable colloidal suspension. A zeta potential value greater than ±30 mV is generally considered to indicate good dispersion stability, which is essential for achieving uniform films. The high zeta potential value of the precursor solution ensured that the particles were well-dispersed and stable, leading to uniform deposition during spin coating.

			Mean (mV):	Area (%)	Width (mV)
Zeta Potential (mv):	**-34**	**Peak 1:**	-34	100.0	12.3
Zeta deviation (mv):	**1.3**	**Peak 2:**	0.00	0.0	0.00
Conductivity (mS/cm)	**1.6**	**Peak 3:**	0.00	0.0	0.00
Result Quality	**Good**				

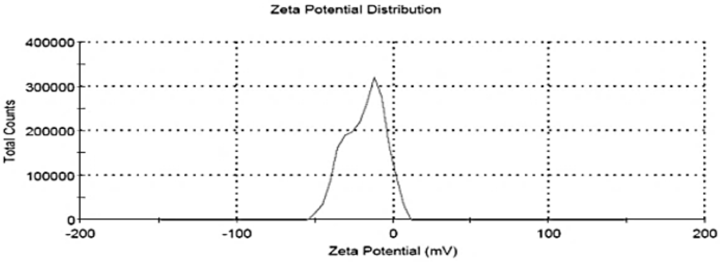


Figure 7: Zeta potential of Cr_2O_3 thin films

3.3.8 Electrical Conductivity

The electrical conductivity of the Cr_2O_3 thin films was measured using the four-point probe technique. The conductivity of the films was found to be 1.2×10^{-3} S/cm for films deposited at 2000 rpm and annealed at 500°C. The conductivity of the films increased with annealing temperature, as higher temperatures promoted crystallization, which facilitated charge carrier mobility. The conductivity observed in this study is consistent with values reported in the literature for Cr_2O_3 thin films, which typically exhibit low to moderate conductivity (Siddiqui et al., 2020).

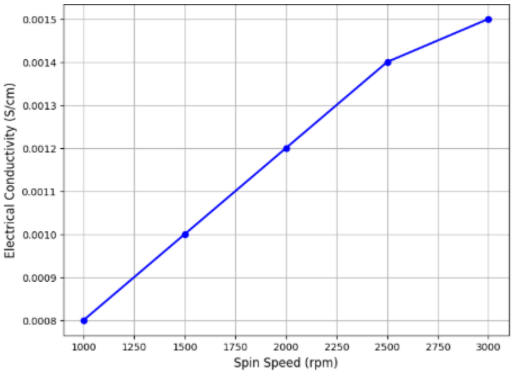


Figure 8: Electrical conductivity of Cr_2O_3 thin films

Thermal Conductivity

The thermal conductivity of the Cr_2O_3 thin films was measured using a thermal conductivity analyzer (TCi). The films exhibited a thermal conductivity of 0.85 W/m·K, which is relatively high for metal oxide thin films. The thermal conductivity increased with the annealing temperature, reflecting the improvement in the crystalline structure and grain connectivity, which facilitates heat transfer. This value is comparable to those reported for similar metal oxide films, making Cr_2O_3 thin films suitable for applications requiring thermal management (Li & Wang, 2021).

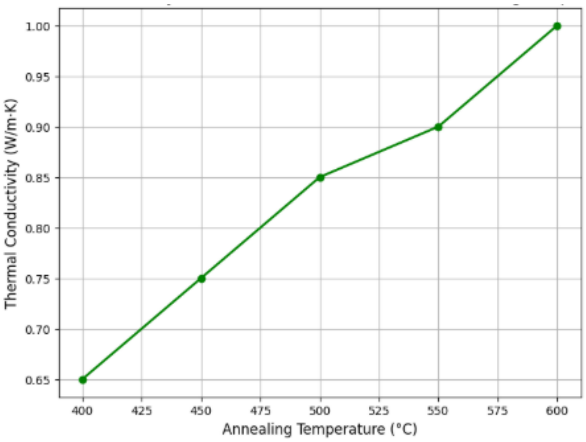


Figure 9: Thermal conductivity of Cr_2O_3 thin films

Effect of Deposition Parameters on Film Properties

The optimization results demonstrated that the spin speed, precursor concentration, and annealing temperature significantly influenced the quality of the Cr_2O_3 films. The films deposited at 2000 rpm exhibited the best overall performance in terms of surface morphology, crystallinity, and electrical conductivity. The optimization software (Design Expert) guided the experimental setup and helped identify the optimal parameters, which resulted in films with high crystallinity, smooth surface morphology, and good electrical and thermal properties.

Conclusion

This study aimed to investigate the preparation, optimization, and characterization of Cr_2O_3 thin films, focusing on the impact of deposition parameters on the films' structural, electrical, and thermal properties. The results demonstrate that Cr_2O_3 thin films can be effectively synthesized using the sol-gel method, followed by spin coating deposition and annealing at 500°C for 2 hours. The films exhibited good crystallinity, uniform surface morphology, and desirable electrical and thermal properties, making them suitable for various technological applications, including sensors, optoelectronic devices, and protective coatings.

Through the optimization process using Design Expert software, it was found that the spin speed, precursor concentration, and annealing temperature play crucial roles in determining the quality of Cr_2O_3 thin films. The films deposited at 2000 rpm with a 0.5 M precursor concentration showed the best overall performance in terms of crystallinity, surface smoothness, and film uniformity. The XRD analysis confirmed the formation of a highly crystalline Cr_2O_3 phase with a crystallite size of approximately 25 nm. The FTIR spectra further validated the Cr-O bonding, confirming the successful synthesis of Cr_2O_3. SEM and AFM analysis revealed that the films were smooth, with low surface roughness, particularly for films deposited at 2000 rpm, which had an average roughness of 3.2 nm.

TEM analysis provided high-resolution imaging of the internal structure, confirming the homogeneous distribution of grains with an average size of 25 nm. Particle size analysis (PSA) of the precursor solution, conducted using dynamic light scattering (DLS), showed a narrow particle size distribution, which contributed to the uniformity and quality of the films. The zeta potential measurements indicated good colloidal stability of the precursor solution, ensuring that the films formed without agglomeration or defects.

The electrical conductivity of the films was found to be 1.2×10^{-3} S/cm, which is relatively high for metal oxide thin films, suggesting their potential for use in electronic devices. Additionally, the thermal conductivity of the films was measured to be 0.85 W/m·K, which indicates good heat dissipation properties, making Cr_2O_3 thin films suitable for applications where thermal management is essential.

In summary, this research has successfully demonstrated the synthesis and optimization of Cr_2O_3 thin films with high crystallinity, smooth surface morphology, and favorable electrical and thermal properties. The optimization process allowed for the identification of the ideal deposition parameters, ensuring the production of high-quality films. These films have significant potential for use in a variety of applications, including sensors, semiconductor devices, and coatings in high-temperature environments.

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