

RESEARCH ARTICLE

Structural, Optical, and Mechanical Properties of Steam–Oxidized MgO–Al₂O₃ Co–Deposited Mixed Thin Films: Influence of Oxidation Temperature

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ABSTRACT: Thin film coatings play a crucial role in various technological applications, including optoelectronics, protective layers, and display technologies. This study investigates the structural, optical, and mechanical properties of MgO–Al₂O₃ co-deposited mixed thin films prepared via steam oxidation of vacuum-evaporated Mg–Al mixed metal thin films. The films, with a controlled thickness of 300 nm, were oxidized at varying temperatures (573 K, 623 K, and 673 K) to analyze their phase composition, surface morphology, optical transmittance, band gap, refractive index, adhesion, and intrinsic stress. X-ray diffraction (XRD) confirmed the formation of a cubic MgO-dominated structure, while Al₂O₃ remained amorphous, indicating a stable mixed oxide formation. Scanning electron microscopy (SEM) revealed a transition from granular and cauliflower-like morphologies in pure Mg and Al films to a well-decorated granular structure in co-deposited films, further evolving into agglomerated grains upon oxidation. Higher oxidation temperatures enhanced optical transmittance (above 450 nm), band gap (2.9–3.4 eV), and refractive index (1.69–1.72), attributed to improved stoichiometry and reduced grain boundary scattering. Mechanical properties, including adhesion (33–39.9 × 10⁴ N/m²) and intrinsic tensile stress (55–80 × 10⁷ N/m²), increased with oxidation temperature due to enhanced crystallinity and interfacial bonding. However, higher temperatures also induced void formation, contributing to intrinsic stress. These findings highlight the tunability of MgO–Al₂O₃ thin films for advanced applications, particularly in plasma display panels (PDPs), where improved durability and optical performance are critical. The study demonstrates that oxidation temperature is a key parameter in optimizing the functional properties of co-deposited oxide thin films.

Keywords: MgO–Al₂O₃ thin films, Steam oxidation, Optical properties, Mechanical properties, Co-deposition, Oxidation temperature effect

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1. INTRODUCTION

Thin film coating technologies have become ubiquitous in modern society, finding applications across diverse sectors

that impact our daily lives. These advanced coatings serve critical functions in industries ranging from food packaging and pharmaceutical manufacturing to electronics production, consumer goods, automotive components, and architectural elements [1]. The versatility and economic significance of thin films have driven substantial global research efforts aimed at developing novel coating materials and deposition techniques [2]. However, as technological demands continue to evolve, conventional single-material thin films increasingly face performance limitations, necessitating innovative approaches to overcome these material constraints [3]. One promising solution lies in the co-deposition of complementary materials, which enables the creation of composite thin films with tailored properties that address specific application requirements [4].

Among the various materials employed in thin film technologies, magnesium oxide (MgO) has emerged as particularly valuable for plasma display panel (PDP) applications. This material offers an exceptional combination of properties including high optical transparency, excellent dielectric characteristics, strong secondary electron emission coefficient, and refractive index closely matched to glass substrates, and high density [5-10]. In alternating current PDPs (ac-PDPs), MgO serves as a crucial protective layer that maintains plasma discharge while shielding underlying components from ion bombardment. However, a significant limitation of MgO thin films is their susceptibility to moisture absorption when exposed to ambient air, leading to corrosion that degrades protective layer performance and ultimately reduces the operational lifespan of PDP devices [11]. Research efforts to address this challenge have explored various mitigation strategies, including alternative protective materials and advanced sealing techniques to minimize environmental exposure. These ongoing investigations aim to improve the durability and reliability of MgO-based coatings under diverse operating conditions [11].

To combat the environmental stability issues associated with pure MgO thin films, researchers have turned to aluminum oxide (Al₂O₃) as a co-deposition candidate. Al₂O₃ possesses several advantageous properties including high optical transmittance, comparable secondary electron emission characteristics, and significantly improved resistance to atmospheric exposure. The combination of these two oxides presents an opportunity to create composite thin films that maintain the beneficial optical and electronic properties of MgO while incorporating the environmental stability of Al₂O₃.

The present study focuses on the development of MgO-Al₂O₃ composite thin films through a vacuum evaporation and steam oxidation process. This investigation specifically examines the effects of oxidation temperature variations (573 K, 623 K, and 673 K) on the resulting films' structural, optical, and mechanical properties. The research methodology involves co-depositing magnesium and aluminum metal films followed by controlled steam oxidation to produce the desired oxide composites. This approach allows for systematic investigation of how

processing conditions influence final film characteristics.

The importance of oxidation temperature in determining thin film properties cannot be overstated. Higher processing temperatures generally promote improved crystallinity through enhanced atomic mobility and defect annihilation. This crystalline improvement typically leads to better-defined optical characteristics, including increased transmittance and modified band structure. Simultaneously, the mechanical properties of the films, particularly adhesion strength, often benefit from the improved interfacial bonding that occurs at elevated temperatures. However, these benefits must be balanced against potential increases in intrinsic stress that may develop due to thermal expansion mismatches between film and substrate materials.

Structural characterization of the resulting films reveals critical information about phase composition and crystallinity. X-ray diffraction analysis provides insights into the crystalline structure development, while scanning electron microscopy offers detailed examination of surface morphology evolution. These structural investigations are complemented by comprehensive optical measurements, including transmittance spectroscopy and band gap determination, which collectively provide a complete picture of how oxidation temperature influences light-matter interactions in the composite films. The mechanical properties of the thin films, particularly adhesion strength and intrinsic stress, represent equally important considerations for practical applications. Strong film-substrate adhesion is essential for long-term durability, while controlled stress levels help prevent delamination or cracking during device operation. The relationship between processing temperature and these mechanical characteristics forms a key aspect of this investigation.

This research contributes to the broader field of thin film technology by providing systematic data on the optimization of MgO-Al₂O₃ composite films through controlled oxidation processing. The findings offer practical guidance for tailoring thin film properties to meet specific application requirements, particularly in the context of PDP technology where both optical performance and environmental stability are paramount. By establishing clear relationships between processing conditions and final film characteristics, this work advances the understanding of composite oxide thin films and their potential for next-generation display technologies. The results of this study demonstrate that careful control of oxidation temperature enables precise tuning of thin film properties, allowing for optimization of both optical performance and mechanical reliability. Higher oxidation temperatures generally produce films with enhanced crystallinity, improved optical transmittance, and stronger adhesion characteristics. These findings suggest that through appropriate process parameter selection, it is possible to engineer MgO-Al₂O₃ composite thin films with extended operational lifespans for PDP applications while maintaining excellent optical performance. This research provides valuable insights for materials scientists and engineers working to develop advanced thin film coatings for optoelectronic devices and other demanding applications.

2. EXPERIMENTAL DETAILS

2.1. Growth of MgO-Al₂O₃ co-deposited mixed thin films

The MgO-Al₂O₃ co-deposited mixed thin films were fabricated through a two-step process involving vacuum evaporation followed by steam oxidation. High-purity (99.98%) magnesium granules (Alfa Aesar) and (99.99%) aluminum wire (Balzers) were used as source materials for thermal evaporation in a high-vacuum chamber maintained at 10^{-5} mbar. The deposition system was equipped with two independently controlled molybdenum boats, allowing simultaneous evaporation of both metals. To prevent cross-contamination between sources, physical partitions were installed between the evaporation boats. Figure 1 demonstrates the schematic illustration of the dual-source co-deposition system configuration used for Mg-Al thin film fabrication, showing the relative positions of evaporation sources, substrate holder, and isolation partitions.

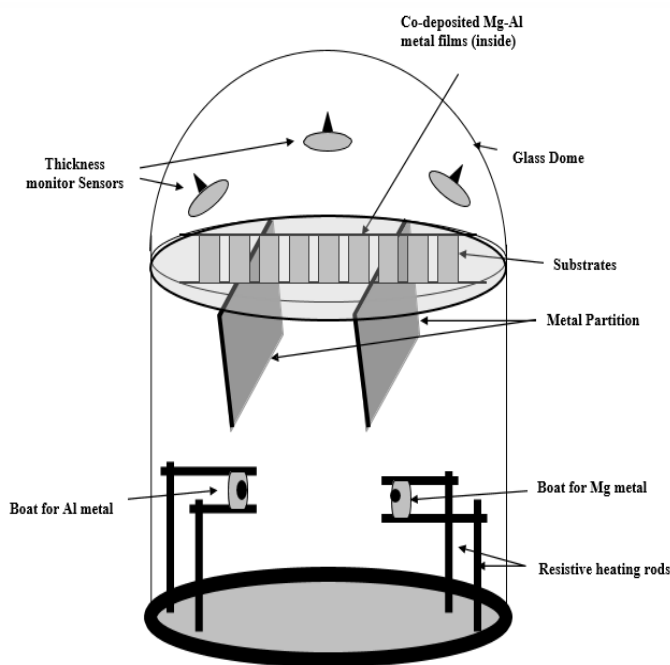


Fig. 1. Schematic illustration of the dual-source co-deposition system configuration used for Mg-Al thin film fabrication, showing the relative positions of evaporation sources, substrate holder, and isolation partitions.

The deposition parameters were carefully optimized to account for the different melting points of Mg (650°C) and Al (660°C). The evaporation rates were maintained at 7.5 Å/s for Mg and 8.5 Å/s for Al, achieved through precise control of the boat temperatures and power supplies. The substrate holder position was adjusted to ensure uniform thickness distribution across all deposited films, with the substrate maintained at room temperature during deposition. Film thickness was monitored in real-time using three independent quartz crystal microbalances, with final thickness controlled

through regulation of both evaporation rate and deposition time.

Following deposition, the Mg-Al co-deposited metal films were subjected to steam oxidation at three different temperatures (573 K, 623 K, and 673 K) to form the desired oxide composites. The oxidation process was carried out in a controlled steam environment, resulting in complete conversion to MgO-Al₂O₃ mixed oxide films with a final thickness of approximately 300 nm.

2.2. Characterization Techniques

Structural characterization was performed using a Philips PW 3710 X-ray diffractometer with Cu K α radiation ($\lambda = 1.5406$ Å) operating at 40 kV and 30 mA. Surface morphology and microstructure were examined using a JEOL JSM-6360 scanning electron microscope operated at 15 kV accelerating voltage. Elemental composition analysis was conducted via energy-dispersive X-ray spectroscopy (EDS) coupled with the SEM system. Optical properties were measured using a Hitachi U-2800 double-beam spectrophotometer over the wavelength range of 300-800 nm. The refractive index was calculated from transmittance and absorbance data at 621 nm using established analytical methods [12]. Mechanical properties including adhesion strength and intrinsic stress were evaluated using direct pull-off (DPO) testing and interferometric techniques respectively [13], with measurements performed at multiple locations on each sample to ensure statistical significance.

3. RESULTS AND DISCUSSION

3.1. Structural Properties

MgO-Al₂O₃ co-deposited mixed thin films oxidised at 673-573 K exhibited (200) and (220) diffraction peaks indicating the dominating cubic MgO structure as shown in Figure 2. We observed no characteristic peaks of any other phases. This evidenced that Al₂O₃ persists in its amorphous nature. This observation suggests that the incorporation of Al₂O₃ into the MgO does not significantly alter the crystalline phase of the material. Furthermore, the amorphous nature of Al₂O₃ contribute to the overall stability and other properties of MgO-Al₂O₃ mixed thin films. H. Yuan et al. achieved similar results using Al₂O₃/MgO thin films deposited at low temperatures utilizing filtered cathode vacuum arc (FCVA) technology, reported that no Al₂O₃ diffraction peak found in XRD even after 5:4 composition Al₂O₃/MgO ratio used in the film [14]. L. Zang et al. found that Al₂O₃ thin films showed a diffraction peak only at more than 1050°C annealing temperature; before that, Al₂O₃ thin films retained their amorphous nature [15]. P. Maiti et al. reported that the incorporation of Al³⁺ into MgO thin films improved transmittance as well as crystallinity while having no Al or Al₂O₃ diffraction peaks [16].

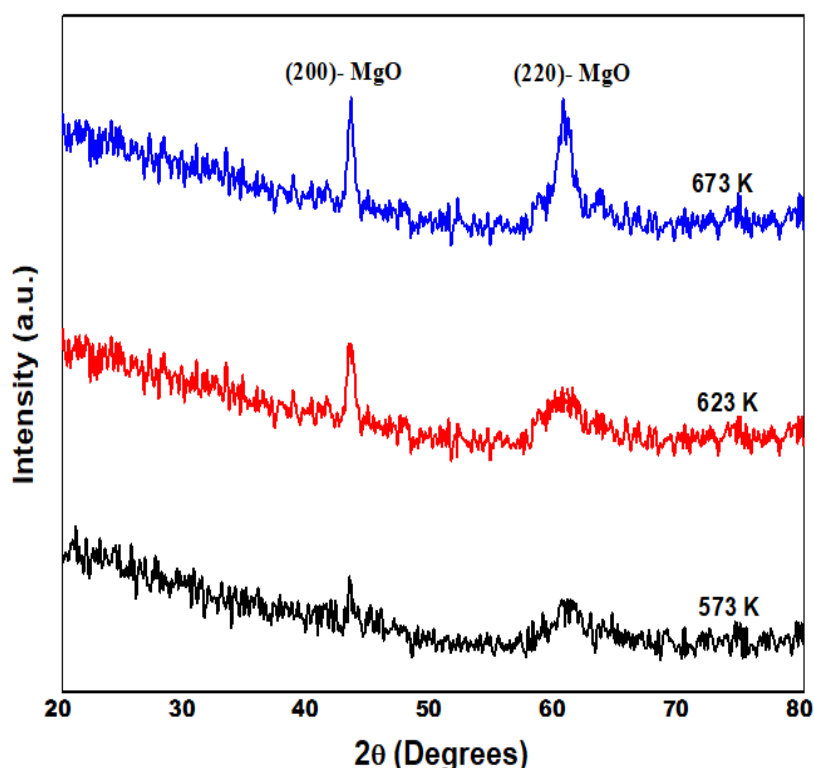


Fig. 2. X-ray diffraction pattern of co-deposited MgO-Al₂O₃ mixed thin films showing characteristic peaks of cubic MgO phase (JCPDS 45-0946). The absence of Al₂O₃ peaks indicates its amorphous nature in the composite structure.

XRD patterns didn't show any magnesium or aluminium metal diffraction peaks, indicating that the Mg-Al co-deposited mixed metal thin films had completely oxidised, resulting in MgO-Al₂O₃ thin films. These results suggest that the co-deposition process effectively promotes the formation of a stable mixed oxide structure via oxidation.

Figure 3 shows that, the E-DAX data of co-deposited mixed MgO-Al₂O₃ thin film annealed at 673K. It shows the presence of magnesium (30%), aluminium (24%), and oxygen (27%) contents. Some other elements i. e. Ca, Si etc. are the glass substrate contents. It shows the Mg, Al and O elements are in greater in percentage in co-deposition of mixed MgO-Al₂O₃ thin film.

3.2. Surface morphology

Figure 4 shows the surface morphology of Mg, Al, and their co-deposited Mg-Al mixed metal thin film as well as the MgO-Al₂O₃ thin film. The surface morphological variations in single-single metals and after co-deposition as well as oxidation are distinctly observed. Figure 4(a) shows the platelet flower-like morphology of magnesium metal thin films, whereas Figure 4(b) shows the fine granular structure of aluminium metal thin films. In the mixed Mg-Al (Figure 4(c)) metal thin film, the platelet and granular structures are combined. Fig. 4d) shows the surface morphology of MgO-Al₂O₃ co-deposited mixed thin film oxidised at 673K, revealing the agglomerated grain formation. The inset figure

shows the smaller grain formation at lower oxidation temperature (573 K), demonstrating the effect of oxidation temperature in the form of increase in grain size. This illustrates how variations in oxidation conditions can significantly influence the surface morphology of the films.

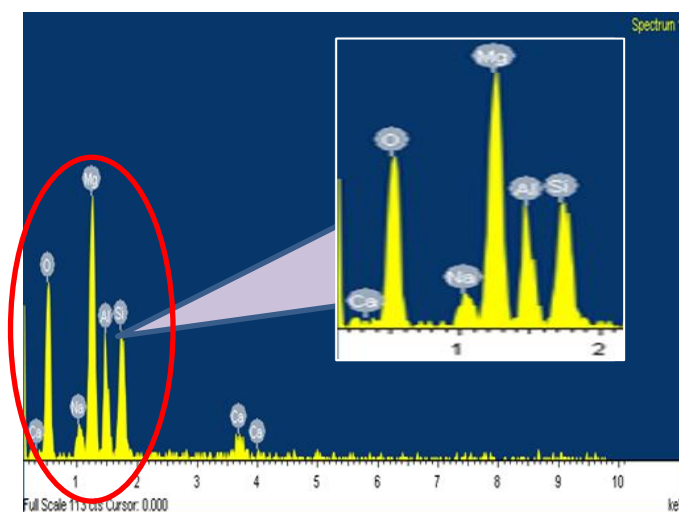


Fig. 3. Energy-dispersive X-ray spectroscopy (EDS) spectrum of MgO-Al₂O₃ co-deposited thin film oxidized at 673 K, showing characteristic peaks of Mg (30 at%), Al (24 at%), and O (27 at%). Inset displays the magnified view of the primary elemental peaks with substrate contributions (Si, Ca) indicated.

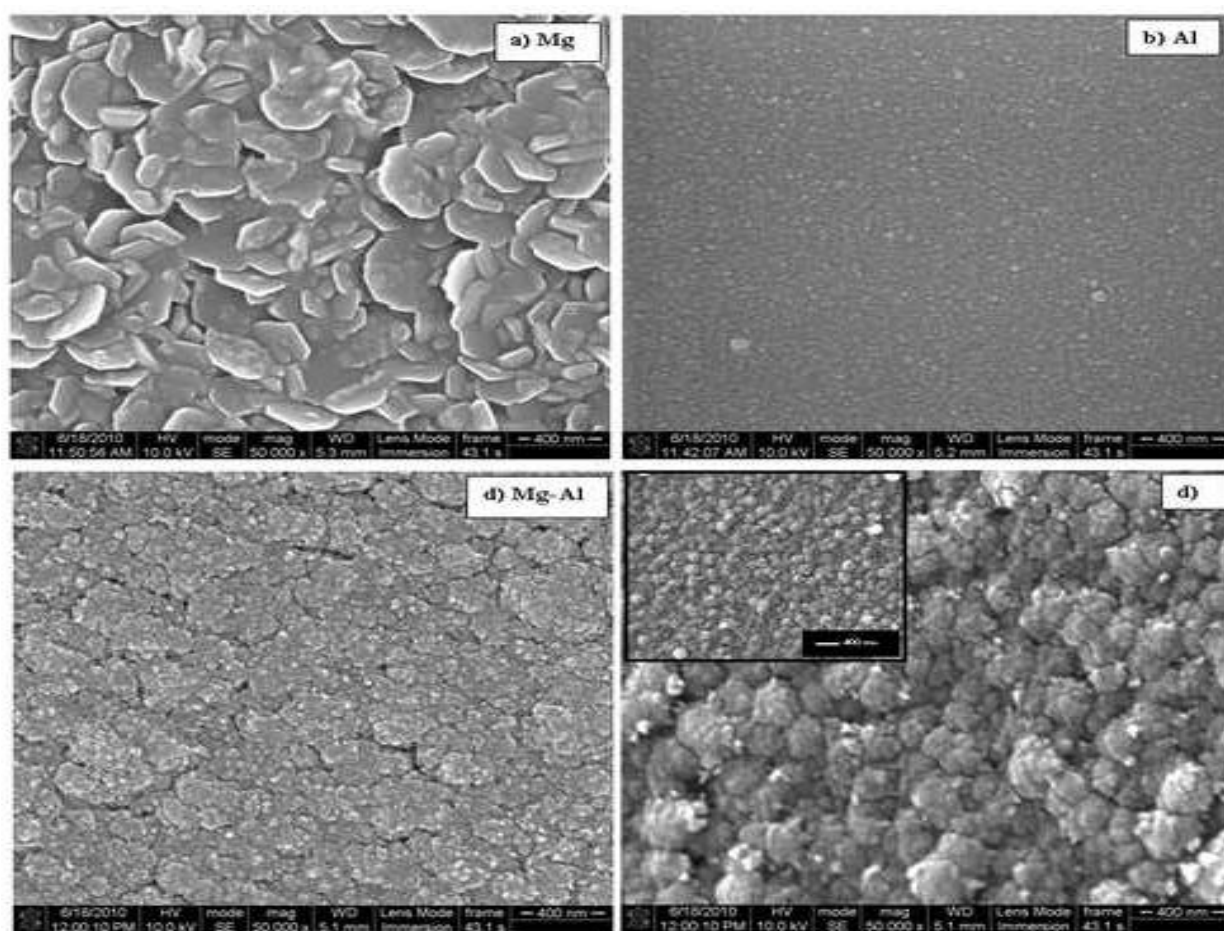


Fig. 4. Scanning electron micrographs showing morphological evolution: (a) Pure Mg thin film exhibiting platelet flower-like structure, (b) Pure Al thin film with granular morphology, (c) Co-deposited Mg-Al alloy film showing hybrid granular-cauliflower structure, and (d) Oxidized MgO-Al₂O₃ composite film at 673 K with agglomerated grains (inset: finer grain structure at 573 K oxidation).

3.3. Optical properties

Figure 5 shows the optical transmittance of a co-deposited mixed MgO-Al₂O₃ thin film. The effect of varying oxidation temperatures was investigated. It was found that the optical transmittance of the co-deposited mixed MgO-Al₂O₃ thin films increases significantly above 450 nm wavelength. The mixed thin films oxidised at a higher annealing temperature exhibited better transmittance. High annealing temperature causes drastic improvement in the thin film's stoichiometry, resulting the reduction in transmittance loss. Mostly, these losses are obtained due to the photon's scattering at grain boundaries. Higher oxidation temperature causes the grain growth, resulting in a reduction in the density of grain boundaries, which plays an important role in the photon's scattering [17].

These results are in good agreement with XRD outcomes. Moreover, the enhanced crystallinity observed in the XRD analysis further corroborates the beneficial effects on the thin film's optical properties. The direct energy band gap was calculated using standard techniques from the optical absorption data.

The value of α , the optical absorption coefficient, is determined by:

$$\alpha = [\alpha_0 (h\nu - E_g)^n] / (h\nu) \quad (1)$$

Where, α is the absorption coefficient of deposited thin film, α_0 = Absorption coefficient of bare substrate, $h\nu$ is the photon energy and E_g is the band gap energy. The determination of the optical gap of the films is deduced from the slope of the curve $\alpha h\nu$ against $h\nu$ for as-deposited films the band gap varied from 2.9 to 3.4 eV. Table 1 shown that the band gap of mixed MgO-Al₂O₃ thin films increases with an increase in annealing temperature. It obtains might be due to the reduction in oxygen vacancies at higher oxidation temperatures or might be due to the reduction in defect-related absorption or mobility gap of the predominantly amorphous films. This causes a change in the electronic structure of surfaces, resulting in the ionic gap and consequently the total band gap [6]. The effects of annealing temperature on the surface morphology and optical characteristics of various oxides have been reported in our previous reports [18, 19].

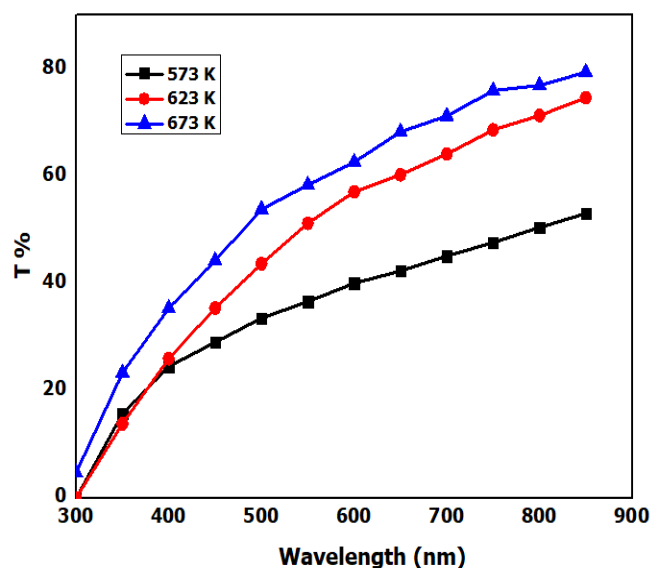


Fig. 5. Optical transmittance spectra of co-deposited MgO-Al₂O₃ thin films as a function of oxidation temperature (573–673 K), demonstrating increased transparency with higher processing temperatures in the visible spectrum (400–800 nm).

3.4. Refractive index

The analytical method has been used for the calculation of refractive index n using the following formula [12]:

$$n = \left[\frac{n_s^2 T_f + n_s (1 + \sqrt{R_f})^2}{T_f + n_s (1 - \sqrt{R_f})^2} \right]^{1/2} \quad (2)$$

Where, n = refractive index of the film, n_s = refractive index of the substrate, T_f = transmittance of the film, R_f = reflectance of the film.

The refractive index values of MgO-Al₂O₃ co-deposited mixed thin film for different oxidation temperatures are tabulated in Table 1. An increase in thin film refractive index with an increase in oxidation temperature was observed. The obtained refractive indices of co-deposited mixed thin films are in between the reported refractive index of MgO as well as Al₂O₃ thin films [6, 12, 20, 21]. J. Hu et al. investigated the influence of film growth thickness on the refractive index and crystallization of oxide thin film. According to the reports, increased annealing temperatures in oxide materials lead to an increase in refractive index due to densification, better crystallinity, and microstructural changes of the film [22].

3.5. Adhesion

Adhesion is related to the strength of the binding forces and contact point concentration at interface between the two materials, so durability of film acquires prime importance in

its application, hence researchers' shows keen interest in it. Table 1 shows the values of adhesion of MgO-Al₂O₃ co-deposited mixed thin films oxidised at different temperatures. It is seen that adhesion increases with an increase in oxidation temperature ($33 - 39.9 \times 10^4$ N/m²). Due to heating, the mobility of adatoms as well as nucleation sites increases, and the elimination of trapped excess vacancies results in increased adhesion. This adhesion enhancement is attributed to the improved interfacial bonding and structural integrity of the films [12, 13, 23]. At low oxidation temperatures, atoms have poor mobility; at this temperature, atoms may not have enough energy to rearrange themselves into optimal orientations with minimal defects along with maximum packing density. The increase in oxidation temperature causes the mobility enhancement, due to which atoms can easily move at equilibrium atomic sites, resulting in increased crystallinity [6, 24–26]. Simultaneously, the grain growth creates more contact points at the film-substrate interface; ultimately, adhesion is directly related to the interface contact point concentration [27].

3.6. Intrinsic stress

The intrinsic stress characteristics of MgO-Al₂O₃ co-deposited thin films exhibit a strong dependence on oxidation temperature, as quantified in Table 1. A systematic increase in tensile stress is observed with rising oxidation temperatures (573 K to 673 K), attributed to multiple interrelated mechanisms. During thermal oxidation, enhanced atomic mobility facilitates grain coalescence and surface diffusion, leading to microstructural densification and grain growth. This restructuring process alters the film's density while simultaneously inducing thermal expansion mismatches between the oxide layer and glass substrate. The resulting lattice strain generates tensile stress, which becomes more pronounced at higher temperatures due to accelerated kinetic processes.

The tensile nature of the stress is further exacerbated by two key phenomena: first, the differential thermal expansion coefficients between the growing oxide film (MgO: $\alpha \approx 13.5 \times 10^{-6}$ K⁻¹; Al₂O₃: $\alpha \approx 8.1 \times 10^{-6}$ K⁻¹) and the soda-lime glass substrate ($\alpha \approx 9 \times 10^{-6}$ K⁻¹) create interfacial strain during cooling. Second, elevated temperatures promote partial re-evaporation of metallic components, introducing nanoscale voids that amplify stress concentration, as confirmed by SEM analysis of surface morphology [28, 29]. These observations align with established models of thermal oxidation stress generation, where interfacial deformation and stoichiometric changes collectively contribute to stress enhancement [30, 31].

The measured stress values ($55\text{--}80 \times 10^7$ N/m²) demonstrate the critical balance between improved crystallinity at higher temperatures and the consequent mechanical instability. This trade-off underscores the importance of optimizing oxidation parameters to achieve desired functional properties while maintaining structural integrity in device applications.

Table 1. Optical band gap and refractive index of co-deposited mixed MgO-Al₂O₃ thin films for different oxidation temperature.

Oxidation Temp. (K)↓	Band gap (eV)	Refractive Index	Adhesion ($\times 10^3$ N/m ²)	Intrinsic stress ($\times 10^7$ N/m ²)
673	3.4	1.72	399 $\pm$ 6	80 $\pm$ 8
623	3.2	1.71	352 $\pm$ 2	66 $\pm$ 3
573	2.9	1.69	330 $\pm$ 5	55 $\pm$ 9

The findings correlate well with previous studies on oxide film stress evolution, providing insights for stress management in mixed-oxide systems.

4. CONCLUSION

This study successfully demonstrated the fabrication of MgO-Al₂O₃ co-deposited mixed thin films via steam oxidation of vacuum-evaporated Mg-Al metal films, emphasizing the critical role of oxidation temperature (573–673 K) in tailoring their structural, optical, and mechanical properties. XRD analysis confirmed the formation of a cubic MgO phase, while Al₂O₃ remained amorphous, suggesting a stable composite structure without phase segregation. SEM imaging revealed a progressive evolution in surface morphology—from granular and cauliflower-like structures in pure metal films to agglomerated grains in oxidized films, with higher temperatures promoting larger grain growth and densification. Optical characterization showed that increasing oxidation temperature significantly improved transmittance (beyond 450 nm), band gap (2.9–3.4 eV), and refractive index (1.69–1.72), attributed to enhanced crystallinity and reduced defect density. These improvements are crucial for applications requiring high optical clarity, such as protective coatings in plasma display panels (PDPs). Mechanically, the films exhibited enhanced adhesion ($33\text{--}39.9 \times 10^4$ N/m²) and increased intrinsic tensile stress ($55\text{--}80 \times 10^7$ N/m²) at higher oxidation temperatures. The improved adhesion stems from better interfacial bonding and increased grain contact points, while the rise in intrinsic stress is linked to thermal expansion mismatch and void formation at elevated temperatures. Despite the stress increase, the films maintained structural integrity, making them viable for durable coatings. The co-deposition approach proved effective in combining the beneficial properties of MgO (high transparency, dielectric strength) and Al₂O₃ (environmental stability), offering a synergistic solution for corrosion-resistant PDP protective layers. Future work could explore intermediate oxidation temperatures or post-deposition annealing to further optimize stress-adhesion trade-offs. Overall, this study provides valuable insights into tuning thin film properties via oxidation temperature, paving the way for advanced functional coatings in optoelectronic and protective applications.

DECLARATIONS

Ethical Approval

We affirm that this manuscript is an original work, has not been previously published, and is not currently under consideration for publication in any other journal or conference proceedings. All authors have reviewed and approved the manuscript, and the order of authorship has been mutually agreed upon.

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Availability of data and material

All of the data obtained or analyzed during this study is included in the report that was submitted.

Conflicts of Interest

The authors declare that they have no financial or personal interests that could have influenced the research and findings presented in this paper. The authors alone are responsible for the content and writing of this article.

Authors' contributions

All authors contributed equally in the preparation of this manuscript.

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