

THE EFFECT OF ANNEALING TEMPERATURE UNDER OXYGEN GAS ON ZnO THIN FILM MICROSTRUCTURE

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Abstract

Zinc oxide (ZnO) thin films were deposited using the radio-frequency (RF) magnetron sputtering technique. The influence of post-annealing temperature under an oxygen atmosphere on the surface morphology, crystalline quality, and molecular vibrational properties of the films was systematically investigated. XRD analysis revealed that all ZnO films crystallized in the hexagonal wurtzite structure with a preferred (002) orientation, and that the crystalline quality improved with increasing annealing temperature. A significant reduction in the FWHM of the diffraction peaks was observed, accompanied by an increase in the effective crystallite size from 21.53 to 39.58 nm. AFM and SEM analyses showed that, upon reaching an optimal annealing temperature, the films became denser with increased surface roughness, indicating enhanced grain growth and surface diffusion. These results demonstrate that post-annealing treatment plays a crucial role in tailoring the microstructural and crystalline properties of ZnO thin films.

Keywords: Oxygen Annealing; RF Magnetron Sputtering; Thin Film; ZnO

Introduction

Zinc oxide (ZnO) exhibits encouraging piezoelectric properties and has emerged as one of the most promising functional materials for a wide range of applications, including gas sensors (Bhati *et al.*, 2020; Guo *et al.*, 2021; Kang *et al.*, 2021), solar energy conversions (Zhang, 2015; Shiyani *et al.*, 2020), ultraviolet- lasers (Dong *et al.*, 2017), MEMS actuators (Yuan *et al.*, 2013), thin film bulk acoustic resonators (TFBARs) (Sun *et al.*, 2020), and surface acoustic wave (SAW) devices (Jin *et al.*,

2020; Liu *et al.*, 2021; Magnusson *et al.*, 2015). ZnO thin films are commonly deposited on silicon substrates due to their large-area scalability, cost-effectiveness, compatibility with microfabrication processes, high crystalline quality, and favourable electrical and thermal conductivities. Among various deposition techniques, Radio-Frequency (RF) magnetron sputtering is one of the most widely employed methods for both thin and thick film deposition owing to its simplicity, precise process

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control, excellent film uniformity and orientation, high repeatability, high film density and purity, high deposition rate, and suitability for large-area substrates. The control of surface morphology, crystalline quality, and film texture is critical for optimizing the performance of ZnO-based devices. Achieving the desired physical properties requires careful control of several deposition and post-deposition parameters, including the deposition technique, power source, sputtering power, substrate temperature, substrate orientation, annealing temperature, and growth parameters, such as working gas pressure, deposition time, and target-to-substrate distance (Kong *et al.*, 2013; De Mesa, 2016; Kamble *et al.*, 2018; Liu & Liu, 2019; Kek *et al.*, 2020; Majeed *et al.*, 2022; Sonklin *et al.*, 2022). One of the key challenges in ZnO thin-film fabrication is optimizing the post-deposition annealing temperature to achieve high crystallinity while minimizing structural defects. Post-annealing treatment has been widely reported as an effective approach to modify the crystalline quality, lattice orientation, residual strain and stress, surface morphology, grain and crystallite size, and defect concentration of ZnO films. In this study, we systematically investigated the effect of annealing temperature under an oxygen atmosphere on the microstructural properties of ZnO thin films. The results provide insight into the optimal annealing conditions required to enhance film quality for future piezoelectric and optoelectronic device applications.

Materials and Methods

ZnO thin films were deposited on commercially purchased boron-doped p-type silicon (Si) substrates using the RF magnetron sputtering technique. Prior to deposition, the substrates were subjected to a rigorous cleaning procedure to minimize surface contamination. A 3-inch-diameter, 2 mm thick, and 99.99% pure ZnO target was used as a sputtering target. Initially, the Si substrates were cleaned in a piranha solution at 120 °C for 10 min to remove organic and metallic residues. Subsequently, the substrates were immersed in buffered oxide etch (BOE) for 1 min to remove the native oxide layer, followed by soaking in 65% nitric acid (HNO₃) at 115 °C for 10 min to form a uniform and homogeneous native oxide layer. The substrates were then thoroughly rinsed with deionized water (DIW) and dried using nitrogen (N₂) gas. Finally, the cleaned Si substrates were transferred to an oven and dried at 200 °C for 2 h to relieve residual strain induced during the cleaning process. After the cleaning procedure, the

substrates were mounted onto the substrate holder and loaded into the sputtering chamber. The chamber was evacuated to a base pressure of 8.6×10^{-7} mTorr and reduced to 1.54×10^{-2} mTorr by interdating the Argon (Ar) gas to deposit the films. Pre-sputtering was carried out for 5 min to remove surface contamination from the target. The ZnO films were deposited at 200 W RF power for 3 hours to achieve the desired film thickness. Subsequently, the ZnO films were post-annealed in an oxygen atmosphere (O₂, 99.99% purity) at a pressure of 0.5 mbar for 1 hour. The annealing temperature was systematically varied between 150 and 900 °C (150, 300, 450, 600, 750, and 900 °C) to examine its effect on the microstructural properties of the films. The detailed deposition and annealing parameters are summarized in Table 1.

Table 1. Deposition parameters of ZnO thin film

Sputtering parameter	Conditions
Target material	ZnO (99.99%)
Target diameter	3 inch
Sputtering gas	Ar
Base pressure	8.67×10^{-7} mTorr
Working pressure	1.54×10^{-2} mTorr
RF Sputtering power	200 W
Substrate-to-target distance	6 cm
Substrate material	100-Silicon
Deposition time	3 hours

The deposited and post-annealed ZnO films were characterized using scanning electron microscopy (SEM, JEOL JSM-6010LV), contact-mode atomic force microscopy (AFM, Parker, USA), X-ray diffraction (XRD, Bruker D8 Advance), and Fourier transform infrared (FTIR, Bruker, Tensor 27) spectroscopy. Surface morphology and microstructural features were examined using SEM. Surface topography and roughness were analysed by contact-mode AFM over a scan area of $5 \times 5 \mu\text{m}^2$. The crystalline structure and phase orientation of the films were investigated using XRD with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). FTIR spectra were recorded using an FTIR spectrometer to identify vibrational modes and bonding characteristics. These complementary techniques were employed to evaluate the structural properties, surface morphology, surface roughness, and crystalline quality of the ZnO films.

Results and Discussion

Figure 1(a) presents the XRD patterns of the As-deposited (ASD) and post-annealed ZnO thin films. All diffraction peaks corresponded well to the hexagonal wurtzite structure of ZnO (JCPDS-PDF

No. 03-0888), with no detectable secondary phases, confirming the phase purity of the deposited films. A dominant diffraction peak located at $2\theta \approx 34.4^\circ$, indexed to the (002) plane, was observed for all samples, indicating a strong preferential c-axis orientation perpendicular to the substrate. Weak diffraction peaks corresponding to the (101) and (102) planes were also detected, confirming the polycrystalline nature of the films with a pronounced texture along the c-axis. Figure 1b displays a magnified view of the (002) XRD diffraction peak of the ZnO thin films. A systematic shift of the (002) diffraction peak toward higher 2θ values was observed as the annealing temperature increased up to 600 °C. This shift indicated a reduction in the interplanar spacing (d_{002}), which can be associated with lattice contraction due to defect annihilation and oxygen vacancy compensation during annealing under an oxygen atmosphere. The maximum peak shift occurring at 600 °C reflects the highest degree of lattice contraction and strain evolution. At higher annealing temperatures (750 and 900 °C), the peak shift decreased and tended to stabilize, indicating stress relaxation and strain saturation. At these temperatures, grain coalescence and microstructural reorganization dominate, allowing the lattice parameter to approach an equilibrium, bulk-like value.

The degree of preferred orientation was quantified using the relative peak intensity, P_{hkl} , defined as (Malek *et al.*, 2014):

$$P_{hkl} = \frac{I_{(hkl)}}{\sum I_{(hkl)}} \quad (1)$$

Where $I_{(hkl)}$ is the relative peak intensity from (hkl), and $\sum I_{(hkl)}$ is the sum of intensities of all observed diffraction peaks.

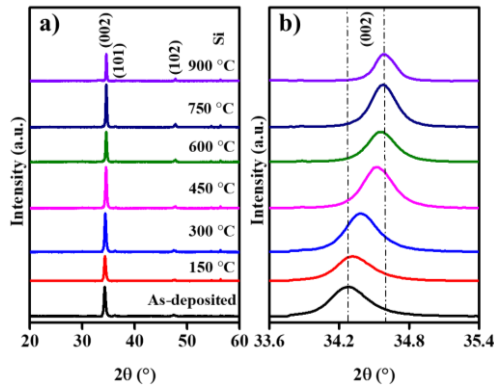


Figure 1. X-ray diffraction (XRD) patterns and (b) a magnified view of (002) the diffraction peaks of ZnO thin films before and after annealing in oxygen

The calculated P_{002} values (Table 2) increased with annealing temperature and reached a maximum at 750 °C, indicating enhanced c-axis texturing and improved crystalline quality. Beyond this temperature, no significant improvement was observed, suggesting the existence of an optimal annealing condition.

The effective crystallite size (D) was estimated from the (002) peak using the Scherrer equation (Scherrer, 1918):

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (2)$$

where K is the Scherrer constant (0.94), λ is the X-ray wavelength (1.5406 Å), β is the full width at half maximum (FWHM) of the (002) peak (in radians), and θ is the Bragg angle.

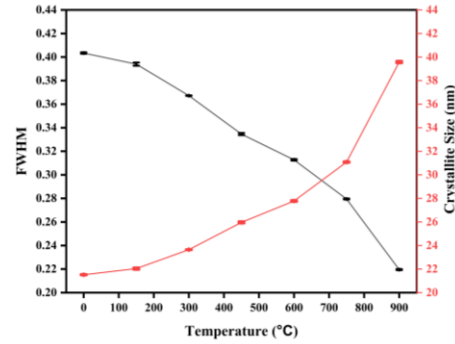


Figure 2. Variation of FWHM and crystallite size of ZnO thin film as a function of annealing temperature

As shown in Figure 2, the FWHM decreases continuously with increasing annealing temperature, indicating reduced lattice disorder and improved crystallinity. Correspondingly, the calculated effective crystallite size increased from approximately 21.53 ± 0.33 nm for the As-deposited film to about 39.58 ± 0.76 nm for the annealed films. It should be noted that the referred crystallite size indicates an estimate of coherent diffraction domain size (~grain size) rather than true crystal size. Nevertheless, the observed trend clearly reflected enhanced crystalline ordering with annealing.

The lattice parameter c of the hexagonal ZnO structure was calculated using the interplanar spacing relation for hexagonal crystals :

$$\frac{1}{d_{hkl}^2} = \frac{3}{4} \left(\frac{h^2 + hk + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (3)$$

where hkl is the Miller index, and a , c are the lattice parameters. The interplanar spacing d_{hkl} was related to the diffraction angle through Bragg's law:

$$\lambda = 2d_{hkl}\sin\theta \quad (4)$$

For the dominant (002) reflection, the lattice parameter c can be directly obtained as:

$$c = \frac{\lambda}{\sin\theta} \quad (5)$$

The calculated lattice parameter c decreased with annealing temperatures up to 450 °C and remained nearly constant at higher temperatures. This behavior is attributed to the relaxation of residual stresses generated during film growth. High-temperature annealing enhances atomic diffusion, enabling the removal of point defects, such as oxygen vacancies and zinc interstitials, which result in lattice contraction and stabilization. The strain along the c -axis perpendicular to the substrate was evaluated using the following relation (Williamson & Hall, 1953):

$$\varepsilon(\%) = \frac{c-c_0}{c_0} \times 100 \quad (6)$$

where c is the lattice parameter of the ZnO film and c_0 is the standard lattice parameter of bulk ZnO (0.5206 nm). The calculated strain values are shown in Table 2. At lower annealing temperatures (ASD-300 °C), the ZnO films exhibited tensile strain, as indicated by positive strain values decreasing from 0.443% to 0.096%. This tensile strain was accompanied by a progressive reduction in the c -axis lattice parameter from 5.229 Å to 5.211 Å, suggesting gradual relaxation of growth-induced defects such as oxygen vacancies and zinc interstitials. During this temperature range, the preferred (002) orientation improved moderately, as reflected by an increase in $P(002)$ and the $P(002)/ASD$ ratio, reaching 1.311 at 300 °C. Upon further annealing at 450 °C, a transition from tensile to compressive strain was observed, with the strain changing from positive to -0.306%. This transition corresponded to a pronounced contraction of the c -axis lattice parameter to 5.190 Å, indicating significant defect annihilation and enhanced oxygen incorporation into the ZnO lattice. Simultaneously, $P(002)$ increased to 0.177 (1.401 times that of the ASD film), demonstrating improved c -axis texturing and crystallinity. At higher annealing temperatures (600-900 °C), the films exhibited stable compressive strain, with the strain saturating at -0.421% and the c -axis lattice parameter remaining constant at approximately 5.184 Å. This saturation suggests that the lattice had reached a near-equilibrium configuration, where further thermal treatment did not induce additional lattice contraction. In this regime, the preferred orientation behavior becomes non-monotonic, while the highest

$P(002)$ value (0.183, ratio 1.454) achieved at 750 °C, indicating optimal crystalline ordering, further annealing to 900 °C results in a reduction of $P(002)$. This decrease was attributed to excessive grain growth, partial texture relaxation, or stress redistribution at very high temperatures. Similar tensile-to-compressive strain transitions have been reported in ZnO thin films annealed at elevated temperatures (Malek *et al.*, 2014). The combined analysis of peak intensity, peak position, FWHM, crystallite size, lattice parameter, and strain clearly demonstrated that post-annealing under an oxygen atmosphere significantly enhanced the structural quality of ZnO thin films. An annealing temperature of approximately 750 °C provided an optimal balance between crystallinity enhancement, defect reduction, and strain relaxation.

Table 2. The intensities of (002) plane, lattice constant (c -axis), and lattice strain of ZnO thin film at different annealing temperatures under oxygen gas.

Temp (°C)	$P(002)$	$P(002)$ to ASD film ratio	C -axis length (Å)	Strain (%)
ASD	0.126	1.000	5.229	0.443
150	0.105	0.833	5.223	0.327
300	0.165	1.311	5.211	0.096
450	0.177	1.401	5.190	-0.306
600	0.129	1.020	5.184	-0.421
750	0.183	1.454	5.184	-0.421
900	0.115	0.913	5.184	-0.421

Figure 3 (a-g) illustrates the surface morphology of ZnO thin films before and after annealing at different temperatures, and Figure 3 (h-n) shows the corresponding cross-sectional morphologies and film thicknesses. The deposited ZnO film (Figure 3(a, h)) exhibits a flake-like surface morphology and a coarse columnar microstructure with a thickness of approximately 814.9 ± 6.2 nm. This morphology is characteristic of RF-sputtered ZnO films grown under high nucleation rates and limited surface diffusion of adatoms, resulting from a relatively high deposition rate. The formation of a flake-like structure within the film at high deposition powers and under specific deposition conditions has been explained elsewhere (Dhanunjaya *et al.*, 2018; Munthala *et al.*, 2022).

With annealing at moderate temperatures (150-450 °C), the columnar microstructure is largely preserved; however, a systematic reduction in film thickness is observed, decreasing from 814.9 ± 6.2 nm to approximately 650.7 ± 7.0 nm at 450 °C (Figure 3i-k). This reduction is attributed to thermally induced densification, driven by grain

boundary rearrangement, pore elimination, and enhanced atomic diffusion, resulting in a more compact microstructure. This densification behavior is consistent with the contraction of the c-axis lattice parameter and the reduction of tensile strain observed in the XRD analysis.

At higher annealing temperatures (≥ 600 °C), the film thickness exhibits a slight increase or tends to stabilize (Figure 3(l-m)), with values ranging from 760.7 ± 17.2 to 777.8 ± 5.2 nm.

At the highest annealing temperature of 900 °C (Figure 3(g, n)), the ZnO film exhibits a dense and smooth surface morphology with larger, well-aggregated grains and a reduced thickness of approximately 651.8 ± 26.9 nm. This behavior suggests extensive grain coalescence and structural stabilization, associated with strain saturation and stress relaxation, as supported by the constant c-axis lattice parameter and compressive strain derived from the XRD analysis. The disappearance of surface pinholes further confirms the formation of a compact and dense film structure at this temperature. The combined surface and cross-sectional SEM analyses demonstrate that annealing temperature plays a critical role in governing densification, grain growth, and thickness evolution in ZnO thin films. These morphological trends are in good agreement with the structural evolution inferred from XRD measurements.

Figure 4 (a-g) shows the three-dimensional AFM surface topography images of ZnO thin films subjected to different annealing temperatures. The deposited film (Figure 4(a)) exhibits a relatively uniform surface with densely packed nanostructured features and an RMS roughness of 6.3 nm. Upon annealing at 150 °C (Figure 4(b)), the surface morphology remains largely unchanged, with a comparable RMS value of 6.4 nm, indicating that low-temperature annealing does not significantly affect surface roughness. Annealing at 300 °C (Figure 4(c)) results in a moderate increase in surface roughness to 7.8 nm, which can be attributed to enhanced surface diffusion and partial grain coalescence.

With further annealing at 450, 600, and 750 °C (Figure 4(d-f)), the RMS roughness stabilizes at approximately 6.7 nm, suggesting that grain growth and densification occur without introducing significant surface undulations. In this temperature range, the AFM images display elongated surface features consistent with a columnar growth morphology and a preferred c-axis orientation, in agreement with the strong (002) texture observed in XRD and the columnar microstructure revealed by cross-sectional FESEM. In contrast, the film annealed at 900 °C (Figure 4(g)) exhibits a pronounced increase in RMS roughness to 11.2 nm,

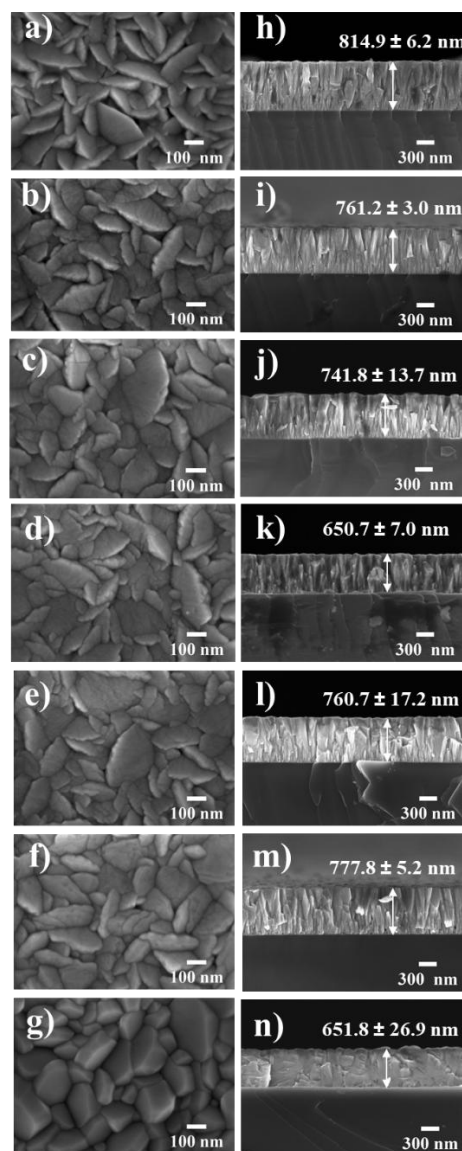


Figure 3. FESEM images of ZnO thin films deposited on Si substrates: (a–g) surface morphologies and (h–n) corresponding cross-sectional morphologies of (a,h) the deposited film, and films annealed at (b,i) 150 °C, (c,j) 300 °C, (d,k) 450 °C, (e,l) 600 °C, (f,m) 750 °C, and (g,n) 900 °C for 1 h under an oxygen atmosphere. The film thickness values are indicated in the cross-sectional images

accompanied by the emergence of larger surface features and increased height variations. This behavior is attributed to extensive grain coalescence and microstructural reorganization at high annealing temperatures, leading to the disappearance of the columnar structure, as confirmed by FESEM analysis. The increased roughness at 900 °C reflects

a transition toward a dense, but morphologically coarser surface, consistent with strain saturation and stress relaxation as inferred from the XRD results. The AFM analysis demonstrates that annealing up to 750 °C maintains relatively smooth surfaces with controlled roughness, while higher annealing temperatures induce significant morphological changes, which results in increased surface roughness.

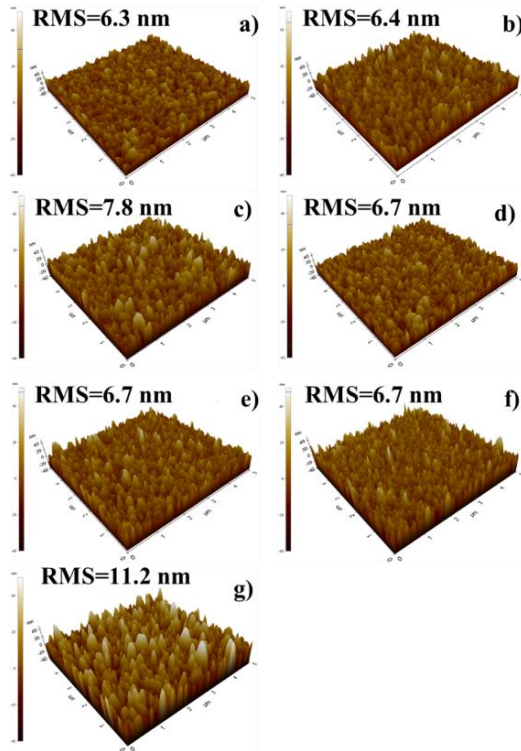


Figure 4. Three-dimensional AFM surface topography images of ZnO thin films: (a) Deposited and annealed films at (b) 150 °C, (c) 300 °C, (d) 450 °C, (e) 600 °C, (f) 750 °C, and (g) 900 °C for 1 h under an oxygen atmosphere. The corresponding RMS surface roughness values are indicated in each image

Figure 5 shows the FT-IR absorption spectra of ZnO films at various annealing temperatures, which were measured between 500–1800 cm^{-1} wavenumber region. The fundamental Zn–O stretching vibration mode corresponding somewhere between 450–600 cm^{-1} (Poddar & Mukherjee, 2019; Ahmad Yusof *et al.*, 2019). The measured spectra show that the FT-IR absorption peak varied between 476 to 514 cm^{-1} depending on the annealing temperature, and it was found that the peak position was red-shifted with an increase in annealing temperature. The observation peak positions at 476, 486, 484, 487, 476, and 514 cm^{-1}

correspond to the pristine film and films annealed at 150, 300, 450, 600, and 750 °C, respectively. An additional peak is seen at 1139 cm^{-1} in the 900 °C annealed film, which is attributed to the functional C–O group (Etcheverry *et al.*, 2018). The shift in the peak position may be due to the stress in the ZnO film induced by the annealing process. These results are consistent with the XRD results.

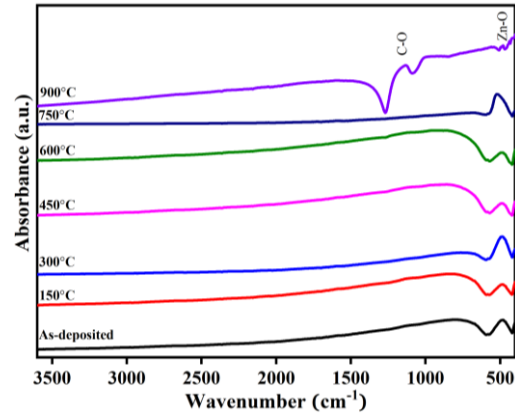


Figure 5. FT-IR spectra analysis of As-deposited film and ZnO thin films annealed at different temperatures, measured in the range 400–1800 cm^{-1}

Conclusion

ZnO thin films were deposited on Si substrates by RF magnetron sputtering and post-annealed under an oxygen atmosphere at temperatures between 150 and 900 °C. XRD analysis confirmed that all films crystallized in the hexagonal wurtzite phase with a preferred (002) orientation. The crystalline quality improved with increasing annealing temperature, reaching an optimum at 750 °C, as indicated by enhanced (002) peak intensity, reduced FWHM, and increased effective crystallite size. The evolution of the (002) peak position revealed a transition from tensile to compressive strain, attributed to defect reduction, oxygen vacancy compensation, and stress relaxation during annealing. SEM observations showed a nano-flake-like surface morphology and a columnar microstructure in the As-deposited films, which remained stable up to 750 °C. At higher temperatures, grain coalescence and densification led to the disappearance of columnar features. AFM results supported these findings, showing nearly constant surface roughness up to 750 °C and a significant increase at 900 °C due to microstructural reorganization. FTIR spectroscopy confirmed Zn–O bond formation in all films, with vibrational peak shifts consistent with strain evolution.

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Author Contributions

Thita Sonklin: Conceptualization, Methodology, Investigation, Data Collection, Formal Analysis, Visualization, Writing – Original Draft, Writing – Review & Editing.

Dhanunjaya Munthala: Methodology, Investigation, Writing – Review & Editing.

Pimchanok Leuasongnoen: Investigation, Data Collection.

Pattanaphong Janphuang: Validation

Soodkhet Pojprapai: Conceptualization, Methodology, Supervision, Project Administration, Funding Acquisition, Validation, Writing – Review & Editing.

All authors reviewed and approved the final version of the manuscript.

Ethics Approval and Consent to Participate

Not applicable. Ethical approval and informed consent were not required for this study because the research did not involve human participants, animals, or human biological samples.

Declaration of Generative AI

Generative AI was used solely for language editing and grammar improvement. The authors reviewed and edited all AI-generated content and take full responsibility for the final manuscript.

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