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Correlation Between the Structural, Morphological and Optical Characteristics of ZnO Thin Films Prepared by Thermal Evaporation

W. Darenfad[1,*], N. Guermat[2], K. Mirouh[1]

[1] *Thin Films and Interfaces Laboratory (LCMI), University of Constantine 1, 25000 Constantine, Algeria*
[2] *Department of Electronics, Faculty of Technology, University of M'sila, PO Box 166 Ichebilia, 28000 M'sila, Algeria*

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In this study, we used thermal evaporation to deposit thin films of zinc oxide (ZnO). The films were then subjected to annealing at various temperatures ranging from 350 °C to 500 °C, with a fixed annealing time of 2.5 hours. The film thickness was kept constant at 300 nm. The morphological, optical properties and structural changes of the ZnO films were investigated using scanning electron microscopy (SEM), X-ray diffraction (XRD) and visible-ultraviolet spectroscopy (VIS-UV) techniques. The XRD pattern also confirmed that the ZnO films exhibited a hexagonal wurtzite crystal structure. The full width at half maximum (FWHM) values of the diffraction peaks decreased as the annealing temperature increased, indicating better crystallinity of the thin films at higher temperatures. SEM images show that the grain size of thin films tends to increase as the annealing temperature increases. The contact angles of the samples were significantly increased and the surface wettability of the layers changed from hydrophilic to hydrophobic after annealing temperature. The VIS-UV data showed that the ZnO films were transparent in the visible region. The optical transmittance slightly increased with increasing annealing temperature. The optical gap (E_g) of the films decreased as the annealing temperature increased. The calculated Urbach energy values indicated that the defects in the ZnO films decreased with annealing temperatures. Finally, the correlation between the structural, morphological, wettability and optical features of the samples was determined. The optical band gap was observed to correlate proportionally with crystallite size and inversely with Urbach energy as a function of annealing temperature.

Keywords: Thin films, ZnO, Thermal evaporation, XRD, SEM, Contact angle.

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1. INTRODUCTION

Zinc oxide (ZnO) thin film is one of the most important n-type semiconductors with hexagonal wurtzite structure [1-3]. It is an important optoelectronic device material among II-IV semiconductors. Several properties including wide band gap and huge exciton binding energy make ZnO useful for photovoltaic cell preparation [4-5]. During the past few years, ZnO has attracted the interest of many researchers due to its high chemical stability, high electron mobility, good transparency and high luminous transmittance [6]. ZnO has some advantages over other oxide materials (such as In_2O_3, TiO_2 and SnO_2) such as low cost, non-toxicity and better electrical as well as optical performance. These advantages make it a promising material for applications in gas sensing, anti reflecting coating and solar cells [7]. Particularly, research on enhanced efficiency solar cells is in great demand recently. Properties of ZnO can be altered favorably by doping with foreign materials as well as by changing annealing condition. Various methods are available for depositing ZnO thin films, including spray pyrolysis, sol-gel, sputtering, chemical vapor deposition, pulsed laser deposition, and thermal evaporation. Among these methods, thermal evaporation is preferred due to its ability to deposit multiple films, environmental friendliness, non-pollutant nature, precise thermal temperature control, and good deposition rate and ease of use [8]. Zinc oxide thin films are grown on variety of substrates like glass [1-2], aluminum [9], silicon and quartz. A preferred option is fabricating ZnO thin films on glass due to their low cost and easy availability. In addition, the preparation of ZnO on glass substrates makes it possible to combine excellent optical properties, electrical and morphologic [1], making these devices more compatible with photovoltaic systems. The performance of a photovoltaic device, such as a thin film solar cell, can be significantly influenced by the defects present in the substrate and the *n*- or *p*-type semiconductor films. The surface condition and adhesion of the layers play crucial roles in improving the efficiency of thin film solar cells. Guermat et al. [2] deposited Ni and/or Co doped ZnO thin films on ordinary glass substrates by spray pyrolysis. The morphological characterization shows good homogeneity, a smooth surface with good adhesion. The same observation was obtained by the work of Darenfad et al. [1] for films of pure ZnO, Mg doped, co-doped Mg/Mn and Mg/Mn/F de-posited on ordinary glass substrates prepared by spray pyrolysis. Guermat et al. [10] used ordinary glass substrates to fabricate homogeneous un-doped and Zn-doped SnO_2 thin films. The work of Daranfed et al. [11], who investigated the effect of deposition time on Co_3O_4 films pepared on ordinary glass substrates, also observed the same behavior. Heat treatment is an important technique used to improve the surface condition of thin films in photovoltaic devices. It achieves a more homogeneous and smoother surface, reduces surface defects, improves adhesion between layers and promotes the removal of impurities, thus helping to improve the efficiency and overall performance of the photovoltaic device.

In this paper, the thermal evaporation deposition

* daranfed.warda@umc.edu.dz

technique was used to fabricate thin layers of ZnO on a glass substrate. The effect of annealing temperature has been studied to understand how it influences the structural, morphological and optical properties of ZnO films. This provides information on the relationship between heat treatment and the properties of ZnO thin films.

2. EXPERIMENTAL DETAILS

Thin films of zinc oxide (ZnO) were grown on glass substrates under specific conditions. The substrates were first cleaned ultrasonically using acetone and distilled water to remove any impurities. The cleaned substrates were then placed in a vacuum chamber. To prepare the ZnO thin films, thermal evaporation was employed. High-purity ZnO powder (99.995 %) was placed on a tungsten crucible, which was then heated by Joule effect. The vacuum chamber was initially pumped to achieve a base vapor pressure of approximately 10 $^{-4}$ mbar. The deposition process resulted in ZnO thin films on the glass substrates. These films were subsequently annealed for duration of 2.5 hours. The annealing process was performed at different temperatures within the range of 350 – 500 °C. The film thickness for all samples was maintained at a constant value of 300 nm.

To determine the structural properties of the ZnO thin films, X-ray diffraction (XRD) analysis was conducted using a Philips X' Pert system. Cu Kα radiation with a wavelength of λCuKα = 1.5418 Å was employed for the XRD measurements. The morphology of the films was analyzed using scanning electron microscopy (SEM). This technique allows for visual examination of the film's surface. The contact angle (CA) measurements were conducted at ambient temperature using an optical system consisting of a lamp that provided white light for illumination. The light source used in this system was a LEYBOLD type light source, operating at 6 *V* and 30 *W*. The optical system also included a projection lens, which allowed for the enlargement of the image of the drop deposited on the sample. The enlarged image of the drop was projected onto a translucent screen with dimensions of 30 cm by 30 cm. To ensure consistent measurements and avoid the effects of water evaporation, the contact angle measurements were taken 5 seconds after depositing a 5 μl drop of water onto the prepared films. This delay allowed sufficient time for the drop to stabilize before capturing the image and measuring the contact angle. The optical transmission properties of the ZnO thin films in the UV-visible range (300 – 800 nm) were measured using a Shimadzu UV-3101 PC spectrophotometer. This analysis provides information about the film's ability to transmit light across different wavelengths.

3. RESULTS AND DISCUSSION

3.1 X-Ray Diffraction Study

In the XRD analysis of the ZnO thin films deposited by thermal evaporation and annealed at different temperatures, shown in Fig. 1, the diffraction peaks corresponding to various crystallographic planes were observed. The peaks at 31.7°, 34.6°, 36.2°, 47.5°, 56.7°, 63.2°, 66.4°, 68.0°, 69.1°, 72.3°, and 76.9° were indexed to the (100), (002), (101), (102), (110), (103), (200), (112), (201), (004), and (202) planes of ZnO, respectively. All these diffraction peaks can be attributed to the wurtzite structure, which is the hexagonal phase of ZnO [1-2]. The intensity of the peaks corresponding to (100), (002), and (101) gradually increases with increasing annealing temperature, indicating a more ordered and crystalline structure. This phenomenon is commonly observed in films prepared by spray [1] or sputtering methods [12]. The growth mechanisms involved in these methods result in the condensation of zinc and oxygen species on the substrate. As the annealing temperature increases, the surface reactions and species mobility are enhanced, allowing the incoming species to occupy more favorable sites on the substrate.

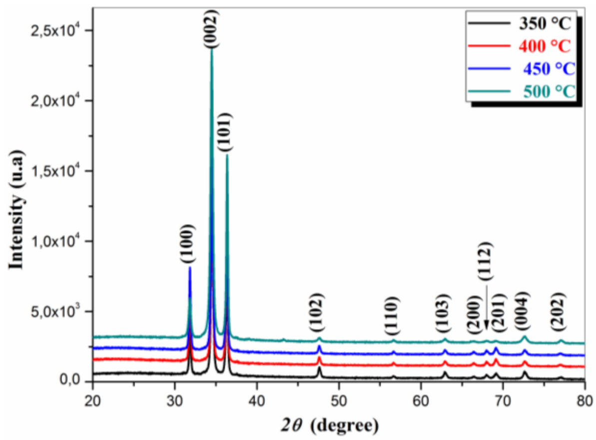


Fig. 1 – XRD pattern of ZnO thin film annealed at different temperature.

The crystallite size (*D*) of zinc oxide nanocrystals for the (002) orientation can be calculated using the Scherrer formula. The Scherrer formula is expressed as [13-14]:

$$D = \frac{0.9\lambda}{\beta\cos\theta} \quad (1)$$

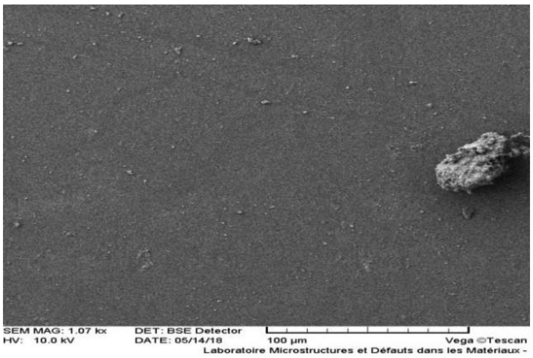
Where, λ is the X-ray wavelength (0.15406 nm for Cu Kα radiation), β is the full width at half maximum (FWHM) of the peak corresponding to the (002) orientation and θ is the Bragg angle corresponding to the (002).

Table 1 – XRD parameters of annealed ZnO thin films for (002) plane

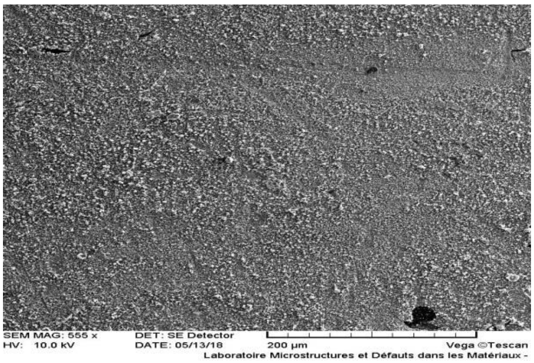
Sample	*D*, (nm)	FWHM
Annealed at 400 °C	32	0.2652
Annealed at 450 °C	35	0.1404
Annealed at 500 °C	37	0.1023
Annealed at 550 °C	39	0.0468

In Table 1, the variation of particle crystallite size and Full Width at Half Maximum (FWHM) with an-nealing temperature is presented. The results demon-strate that as the annealing temperature increases, the FWHM of all peaks narrows slightly, indicating an in-crease in the crystallite size of ZnO films from 32 nm to 39 nm. This enlargement in crystallite size indicates that higher annealing temperatures facilitate the growth of larger crystal grains and enhance the overall crystallinity of the films [15]. Additionally, the significant de-crease in FWHM with increasing annealing temperature signifies an improvement in the crystallinity of the films. This implies that the films become more structurally perfect, with a reduced number of defects, which has a positive impact on their optical properties [15].

3.2 Morphological Study



(a)



(b)

Fig. 2 – Surface morphology of ZnO thin films at (a) 400 °C and (b) 500 °C

Fig. 2 illustrates scanning electron microscopy (SEM) images of ZnO thin films that were deposited on a glass substrate at various annealing temperatures. The SEM images provide visual evidence that the grain size of the thin films tends to increase as the annealing temperature rises. This observation aligns with the findings from X-ray diffraction (XRD) analysis, where an increase in annealing temperature resulted in larger crystallite sizes. The SEM studies confirm the XRD results and support the notion that the crystallite size of the ZnO thin films increases with higher annealing temperatures.

3.3 Wettability Study

Fig. 3 shows the variations in contact angles of water droplets as a function of the annealing temperature of the films. The results indicate that the contact angle gradually increases with annealing temperatures. At 350 °C, the contact angle is 65°, at 400 °C it is 73°, at 450 °C it is 103°, and at 500 °C it is 98°. Based on the experimentally measured contact angles, the films annealed at 350 °C and 400 °C exhibit a hydrophilic nature [13], as their contact angles are less than 90°. In contrast, the films treated at 450 °C and 500 °C demonstrate a hydrophobic behavior [9], with contact angles greater than 90°.

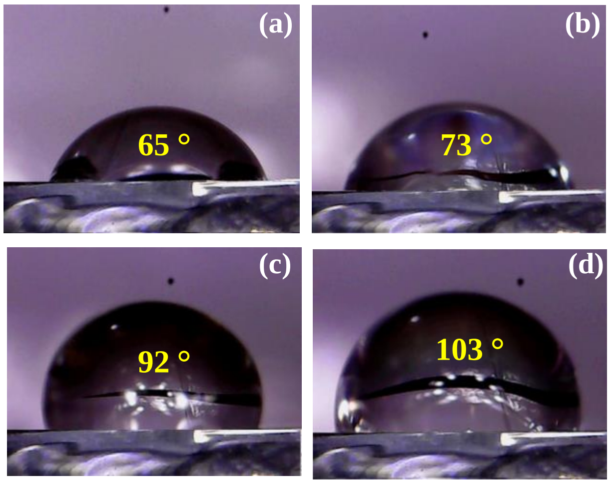


Fig. 3 – Water contact angles of our samples at: (a) 350 °C, (b) 400 °C, (c) 450 °C and (d) 500 °C

This hydrophobic characteris-tic is significant in the context of solar cells. The varia-tion in contact angles with annealing temperature is likely influenced by several factors, including the size and radius of available pores, their distribution [16-17], the density of the layers [14], the deposition technique, the roughness [9] and the size of the crystallites [14]. The findings suggest that the annealing temperature of 400 °C represents a critical point in the transition from a hydrophilic to a hydrophobic surface morphology for the films. These conclusions align with the measured crystallite sizes and the images obtained through scanning electron microscopy (SEM).

3.4 Optical Study

Knowledge of the optical properties of materials is indeed crucial for the design and analysis of optoelec-tronic devices. The information provided in Fig. 4 indi-cates that the optical transmission of ZnO thin films is influenced by the annealing temperature. The trans-mission spectra in the visible wavelength range (300 – 800 nm) demonstrate a clear trend: as the annealing temperature increases at 500 °C, the transmission value also increase. For instance, the transmission values in the visible region are reported as 76.29 %, 75.76 %, 75 %, and 79.42 % for annealing temperatures of 350 °C, 400 °C, 450 °C, and 500 °C, respectively. This suggests that up to 500 °C, higher annealing temperatures enhance the transmission of the thin films. Several researchers have previously reported that the increase in transmittance after annealing is closely associated with an improvement in crystallinity [18, 19]. Enhanced crystallinity leads to a reduction in optical scattering and defects within the films, consequently resulting in increased transmittance [19]. The same behavior was observed by Humayan Kabir et al. [18] and Chaitra et al. [20]. In addition, many researchers have reported that the films exhibited an average transparency of over 78% in the visible region, which indicates good structural homogeneity and crystallinity. This level of transparency is advantageous for optical device applications [18, 21].

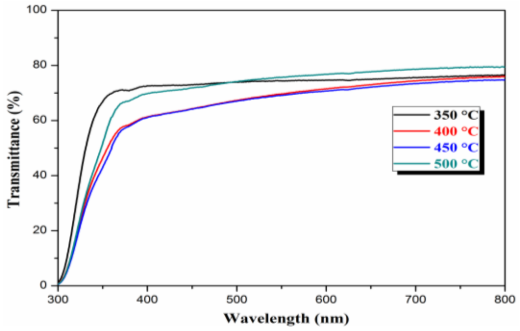


Fig. 4 – Optical transmission T (%) of ZnO thin films as a function of photon wavelength at different annealing temperature

We determined the optical gap (E_g) of the ZnO thin films by analyzing the dependence of the absorption coefficient values (α) on the photon energy. We employed Tauc's relation [22] to calculate the optical gap. Tauc's relation is a commonly used empirical formula that relates the absorption coefficient (α) to the photon energy ($h\nu$) for direct bandgap materials. It can be expressed as:

$$(\alpha h\nu)^n = B(h\nu - E_g) \qquad (2)$$

In this equation, B is a parameter related to the transition probability, E_g represents the optical band gap energy of the material, $h\nu$ denotes the photon energy, and n is an index that characterizes the optical absorption process. The value of n is theoretically equal to 2 and 1/2 for direct allowed and indirect allowed transitions, respectively.

To determine the optical gap, the authors plotted $(\alpha h\nu)^2$ as a function of the photon energy ($h\nu$). By examining this plot, they identified the linear portion of the curve that intercepts the x-axis (Fig. 5). The energy value at this intercept corresponds to the optical gap of the ZnO thin films. The optical gap of the films was evaluated using the Tauc's plot of $(\alpha h\nu)^2$ versus $h\nu$, as shown in Fig. 6 of the study. By extrapolating the linear portion of the plot to the x-axis, the authors obtained the optical gap value of the ZnO thin films.

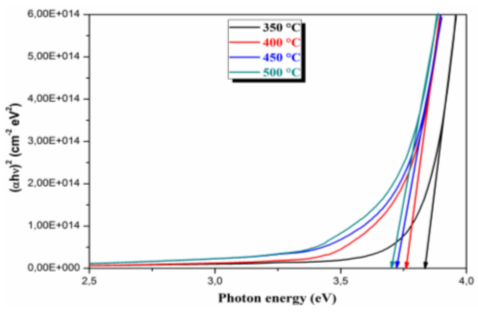


Fig. 5 – Optical band gap of annealed ZnO thin films with photon energy

We notice that the optical gap of the ZnO thin films decreases as the annealing temperature increases. This decrease in the optical gap can be attributed to several factors. Firstly, the annealing process can lead to the removal of defect levels within the films. Defects in the material can introduce energy levels within the band structure, affecting its optical properties. Additionally, the dominant behavior of tensile strain during annealing can contribute to the decrease in the optical gap. Liu et al. [23] reported that the direct optical band gap shifted to the lower energy as a consequence of the annealing temperature increasing in air.

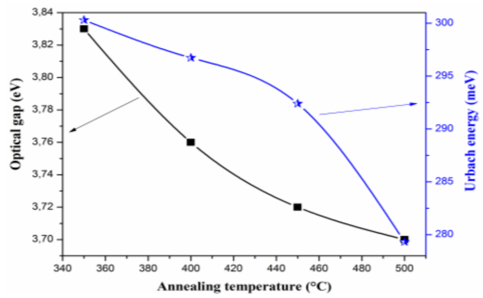


Fig. 6 – Variation of the optical gap & Urbach energy in ZnO thin films vs. the annealing temperature

The heat treatment process during annealing can help eliminate stacking faults and promote the alignment of individual crystallites, resulting in defect-free grain boundaries. Furthermore, the increase in crystalline size of the ZnO thin films after annealing, as indicated by the X-ray diffraction (XRD) data in Table 1, can also be associated with the decrease in the optical gap. As the crystalline size increases, the confinement effects on the electronic states diminish, leading to a decrease in the energy gap. It is worth noting that other authors have also reported similar findings, where the annealing process improves the crystallinity, increases the average grain size, reduces defect concentration, and consequently decreases the strain in the films, leading to a decrease in the optical band gap energy [18, 24].

The Urbach tail is an important parameter used to assess the level of crystallinity, structural defects, or the degree of disorder present in film materials [1]. It is commonly employed to characterize localized states within the optical bandgap of materials. The width of the Urbach tail, also known as the Urbach energy, influences optical transmission and the structure of the optical bandgap. It is related to the distribution of localized states within the bandgap. This Urbach tail manifests as an exponential decay of transmission in poorly crystalline, poorly crystalline, disordered, or amorphous materials, because these materials exhibit localized states that extend into the bandgap [25]. The Urbach energy (E_0) of ZnO thin films can be determined using the following relation, as stated in references:

$$\alpha = \alpha_0 \exp\frac{h\nu}{E_0} \qquad (3)$$

In this equation, α represents the absorption coefficient, $h\nu$ denotes the photon energy ($h\nu (eV) = 12400/\lambda(nm)$), α_0 is a constant, and E_0 represents the Urbach energy.

To calculate the Urbach energy, we plot the natural logarithm of the absorption coefficient ($ln(\alpha)$) as a function of the photon energy ($h\nu$). The slope of this linear plot is related to the Urbach energy (E_0). By analyzing the slope of the ln α versus hυ plot (Fig. 7), we can determine the value of the Urbach energy for the ZnO thin films.

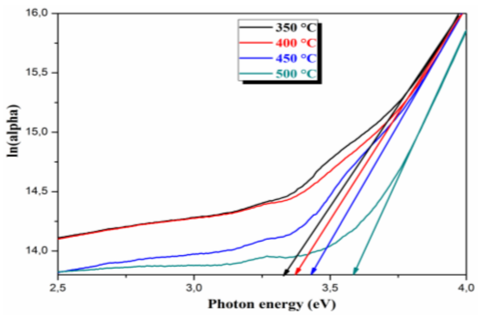


Fig. 7 – Urbach energy diagram of annealed ZnO thin films with photon energy.

It is clear that in Fig. 8 demonstrates a decrease in the Urbach energy or the width of the Urbach tail with increasing annealing temperature. This decrease indicates a reduction in structural disorder within the film as the annealing temperature is raised. Consequently, the improved crystallinity and structural order of the film are reflected in the diminishing Urbach energy. Furthermore, the calculated value confirms that the film exhibits the lowest value of structural disorder when annealed at 500 *°C*. This finding suggests that an annealing temperature of 500 *°C* is optimal for promot-ing maximum crystallinity and minimizing the degree of disorder in the film.

4. CONCLUSION

The ZnO thin films were deposited through thermal evaporation, followed by an annealing treatment in air. The effects of thermal annealing on the structural, morphological and optical properties were investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), contact angle and spectrophotometry.

The XRD analysis revealed an improvement in crystallinity and an in-crease in the crystallite size of the ZnO thin films as the annealing temperature increased. The crystallite size increased from 32 nm to 39 nm, indicating that the heat treatment promoted the growth and alignment of individual crystallites. The films were found to have a Zn single phase with a hexagonal structure. The annealing process induced the oxidation of the deposits, leading to the formation of the desired ZnO phase. The SEM study revealed that the grain size of the ZnO thin films tended to increase with higher annealing temperatures. This observation is consistent with the XRD data, which indicated an increase in crystallite size. The contact angles of the samples were significantly increased, and the surface wettability of the layers changed from hydrophilic to hydrophobic after annealing temperature. The spec-trophotometer data showed a slight increase in optical transmittance as the annealing temperature increased. Additionally, the optical gap of the ZnO thin films de-creased from 3.83 eV to 3.70 eV with increasing anneal-ing temperature. It is worth noting that there was a correlation observed between the optical gap and Urbach energy, indicating that the disorder in the films influenced their optical properties.

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Кореляція між структурними, морфологічними та оптичними характеристиками тонких плівок ZnO, отриманих термічним випаровуванням

W. Darenfad[1], N. Guermat[2], K. Mirouh[1]

[1] *Thin Films and Interfaces Laboratory (LCMI), University of Constantine 1, 25000 Constantine, Algeria*
[2] *Department of Electronics, Faculty of Technology, University of M'sila, PO Box 166 Ichebilia, 28000 M'sila, Algeria*

У цьому дослідженні використовували термічне випаровування для осадження тонких плівок оксиду цинку (ZnO). Потім плівки піддавали відпалу при різних температурах від 350 °C до 500 °C з фіксованим часом відпалу 2,5 години. Товщина плівки була постійною на рівні 300 нм. Морфологічні, оптичні властивості та структурні зміни плівок ZnO досліджували за допомогою методів скануючої електронної мікроскопії (SEM), рентгенівської дифракції (XRD) та спектроскопії у видимому ультрафіолетовому діапазоні (VIS-UV). Картина XRD підтвердила, що плівки ZnO мають гексагональну кристалічну структуру вюрциту. Значення повної ширини на половині максимуму (FWHM) дифракційних піків зменшувалися зі збільшенням температури відпалу, що вказує на кращу кристалічність тонких плівок при вищих температурах. Зображення SEM показують, що розмір зерна тонких плівок має тенденцію до збільшення зі збільшенням температури відпалу. Кути контакту зразків були значно збільшені, а змочуваність поверхні шарів змінилася з гідрофільної на гідрофобну. Залежності VIS-UV показали, що плівки ZnO були прозорими у видимій області. Коефіцієнт оптичного пропускання дещо зростає зі збільшенням температури відпалу. Оптичний зазор (Eg) плівок зменшувався зі збільшенням температури відпалу. Розраховані значення енергії Урбаха показали, що дефекти в плівках ZnO зменшуються з температурою відпалу. Нарешті, було визначено кореляцію між структурними, морфологічними, змочуваними та оптичними властивостями зразків. Спостерігалося, що оптична заборонена зона корелює пропорційно з розміром кристаліту та обернено з енергією Урбаха як функцією температури відпалу.

Ключові слова: Тонкі плівки, ZnO, Термічне випарування, XRD, SEM, Контактний кут.