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Influence of Annealing Temperature on the Optical and Structural Properties of Zinc Oxide (ZnO) Thin Film as a Transparent Conductive Oxide Electrode

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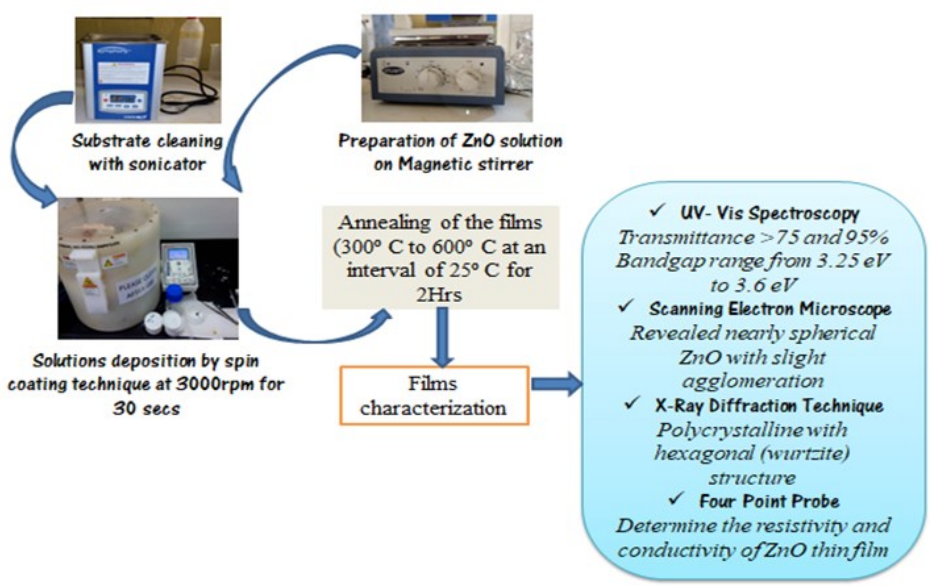
Highlights

- Highest optical transmittance value of 95% was recorded for ZnO thin film.
- The optical band gap energy lies between 3.25 and 3.6eV.
- Absorbance increased with annealing temperature.
- Resistivity decreased with annealing temperature.

Graphical Abstract



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Abstract

This paper presents the modification of ZnO thin film structural and optical properties based on optimized deposition spin-speed and post-deposition heat treatment. ZnO is a semiconductor material in its natural state that have strong n-type conductivity with related flaws such as native defects, interstitial hydrogen and high resistivity. These inherent challenges must be surmounted for pure ZnO to be used as transparent conducting oxide (TCO). ZnO thin films were deposited onto glass substrate by spin coating technique at 3000 rpm for 30 seconds. The deposited thin films were heated at 100 ^oC to evaporate unwanted impurities. The films were then annealed at temperature of 300 ^oC to 600 ^oC at an interval of 25 ^oC for 2 h under ambient temperature. The thin films were characterized and the resistance measurement was performed using four point probe equipment after annealing. Optical, morphology, X-ray diffraction, and electrical properties at ambient temperature condition were reported. Optical properties using UV-Vis spectrophotometer shows that the transmittance of ZnO thin film is >75 and 95% in the visible region of the electromagnetic wavelength. The optical band gap energy ranged from 3.25 eV to 3.6 eV. Scanning electron microscopy (SEM) images reveals the formation of nearly spherical ZnO with slight agglomeration. An appreciable change in its size as the annealing temperatures increases is seen affirming larger size of ZnO at higher annealing temperatures. Increase in ZnO grain size with higher annealing temperatures suggests that crystalline quality of the ZnO improves with the increase in annealing temperature. X-ray diffraction shows that ZnO thin films are polycrystalline and have a hexagonal (wurtzite) structure. The crystallite are oriented with (100) and (101) planes parallel to the substrate surface and current-voltage (I-V) measurement was taken with the use of four point probe with Kiethley measuring source unit to determine the resistivity and conductivity of ZnO thin film.

1. Introduction

Transparent conducting oxides (TCO) serve as substrate on which the other dominant layer components are layered or deposited. TCO for electrode application must exhibit low electrical resistivity and high visible light transparency for photovoltaic devices and display applications [1-4]. Predominantly widely used transparent conducting oxides are thin films of Indium-doped Oxide (ITO) and Fluorine doped Tin Oxide (FTO) in various electronic and optoelectronic devices. However, based on challenge such as high cost of production of ITO's and FTO's call for search for alternative materials for the replacement as transparent conducting electrode. ITO as a transparent electrode in solar cells is an undesirable material for use in low-cost solar cells because of the scarcity of indium and its high deposition cost. ZnO thin films are promising alternative transparent semiconducting oxides because they are inexpensive, non-toxic, have higher resistance against hydrogen plasma and is highly abundant. ZnO is a semiconductor material with a large band gap which has optoelectronic properties and widely used in many applications. However, the main challenges with the use of zinc oxide (ZnO) as transparent conducting oxide are its higher resistance which presents significant challenge for their application although it shows higher light transmittance in the visible region of electromagnetic spectrum. There is a need to reduce the electrical resistivity and the need for low sheet resistance electrode to overcome the flaws or inadequacies and still maintain the higher light transmittance. ZnO, in the pure form or state, offers a high resistance to TCO application. Possibility of using ZnO for large area solar cell transparent electrodes remains a problem to be tackled. Literature reveals that ZnO thin films have been prepared by various

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deposition methods such as spray pyrolysis [5,6], magnetron sputtering [7-11], sol-gel [10,12], pulse laser deposition [13-15], chemical deposition [16-21], thermal evaporation [22-24], simple hydrolysis [25], molecular beam epitaxy [26], laser ablation [27], atomic layer deposition [28], and spin coating [29,30].

Literature reported researches carried out on ZnO as a promising transparent electrode material such as Kim *et al.* [7] reported ZnO/AgNW/ZnO multilayered composite conducting electrode prepared by direct current (dc) magnetron sputtering at room temperature to have thermal stability at 375 ^{o}C, electrical conductivity (8.0Ω /sq), and optical transmittance (> 91% at 550 nm). The solar cells using the electrode they fabricated exhibited a 20% increase in power conversion efficiency when compared to ITO electrode solar cell. Rwenyagila *et al.* [8] reported extremely high resistivity values that cannot be measured for single layered ZnO films for transparent electrode using radio frequency magnetron sputtering of deposition. Rwenyagila *et al.* [9] in their follow up research incorporated Al a mid-layer in radio frequency magnetron sputtering to synthesize ZnO/AI/ZnO film structure having 362 μΩcm, transmittance between 85 and 90% (in the visible region of solar spectrum). 4.72 x 10^{-3} Ω^{-1} with Al thickness of 8nm. Optical band gap energy increases by 0.60 eV for mid layer Al thicknesses 1-10 nm. Ponga *et al.* [18] reported Ga doped ZnO thin films grown through aerosol assisted vapor deposition with resistivity of 7.8 x 10^{-4} Ω cm and carrier concentration of 4.33 x10^{20} cm^{-1}. Afrina *et al*. [31] reported an Al-doped ZnO (AZO) thin films with optical transmittance of 90%, and energy band gap that varies between 3.25 to 3.29 eV. Muslih and Kim [30] investigated zinc oxide thin film that was prepared by spin-coating method at 2000 rpm and annealed at 500 ^{o}C and they reported ZnO thin film with band gap, carrier concentration, mobility, and resistivity of 3.18 eV, 1.21x10^{-19} cm^{3}, 11 cm^{2}/Vs, 2.35 x 10^{-3} Ω.cm, respectively. This research focuses on synthesis of ZnO thin film using spin coating technique at 3000 rpm not previously used and examination of optical property, morphological and structural properties after post-deposition heat treatment. The study is based on deposition at 3000 rpm which is different from spin-speed of thin film deposition encountered in literature. The study of ZnO thin film deposited at 3000 rpm using spin – coating technique will be of interest for potential application in large area electronics and solar cells. This research work presents experimental study of ZnO deposited at spin speeds of 3000 rpm and thermally annealed at temperature regime of 300 ^{o}C to 600 ^{o}C in an interval of 25 ^{o}C under ambient atmosphere at various temperature regimes for development of a low–cost transparent electrode with low resistivity and high transparency ZnO as an alternative material for transparent conducting electrode to replace high cost Indium doped Tin oxide. The optical, morphology, and microstructure of ZnO thin films precursors are examined and reported.

2 Materials and Methods

2.1 Synthesis

The glass substrate used was thoroughly washed with liquid detergent, rinsed in deionized water, ultrasonically cleaned in ethanol, dried inside at 100 ^{o}C in furnace and cooled to room temperature before deposition by spin coating method with a spin coater (model laurel WS-650HZ-23NPP). ZnO precursor solution was prepared from ZnO powder purchased from Sigma-Aldrich, as 0.5g of 99.9% pure ZnO powder was dissolved in 10 ml of ethanol using ultrasonic for 2 h at room temperature without heat. Ethanol as solvent was chosen because it has no characteristic absorption and emission in the visible range of spectrum. The thin films of ZnO were fab-

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ricated by spin –coating method onto glass substrate at 3000 rpm for 30 sec in ambient atmosphere. To improve the properties of the thin films, thermal annealing was carried out at a set temperature regime in Carbolite tubular furnace (Srw 21-501042 type –CT17) for 2 h.

2.2 Characterization

The structural characteristics of ZnO thin film was evaluated by X-ray diffraction technique. Optical properties was examined with a UV-Visible spectrophotometer (Avalight-DH-5-BAL). Surface morphology/ topography was analyzed by scanning electron microscope (SEM model ASPEX 3020). The sheet resistance value measurement of ZnO thin film were made by the four-point probe equipment.

3. Results and Discussion

3.1 Structural Properties

The structural characterization of ZnO thin film was carried out with X-ray diffractometer which was operated at 45kv and 40 mA. The X-ray pattern for the ZnO thin film annealed at 325 ^oC, 425 ^oC, and 525 ^oC is shown in Figure 1(a-d) respectively. The spectra shows a dominant peak at 30.8922^o when ZnO thin film was annealed at 325 ^oC. For comparison, the structural features of ZnO thin films, 2 theta position, height of intensity (Arb.unit), the full width at half maximum (FWHM) of XRD, and d-spacing at 325 ^oC, 425 ^oC, and 525 ^oC are shown in Tables 1 -3, respectively. Peak sharpness of ZnO particles suggests high crystallinity. Figure 1(a)-(d) shows the XRD pattern of ZnO thin films. The peaks observed corresponds to the lattice planes (100) and (101) which are indication of wurtzite hexagonal structure.

3.2 Optical Properties

Optical properties of the annealed zinc oxide was analyzed using UV-Vis spectrophotometer model (Avantes-Avalight-DH-5-BAL) over a wavelength range of 250- 1000 nm in ambient atmosphere. Figure 2 shows optical transmittance of zinc oxide annealed at 325 ^oC, 425 ^oC, and 525 ^oC for 2 h. High transmittance values occurs in the visible region of solar spectrum of wavelength band between 400 to 1000 nm. Comparison shows that the average solar transmittance of the sample annealed at 425 ^oC was greater than 90% in the visible region of solar spectrum (558.109 to 1000 nm). The sample annealed at 325 ^oC has value of transmittance above 85% in the visible region of electromagnetic wavelength. The sample of zinc oxide annealed at temperature of 525 ^oC have transmittance >75% in the visible region of solar spectrum. This high transmittance that lies between >75 and 95% in the visible region of the electromagnetic wavelength are desirable for the optical performance of electrode for solar cell. Absorbance is calculated using Equation (1).

$$A = 2 - log_{10}(\%T) \tag{1}$$

Where, A is absorbance and % T is percentage transmittance

Optical absorption coefficient α is estimated using its relation with the absorbance in Equation (2).

$$\alpha = 2.303\left(\frac{A}{t}\right) \tag{2}$$

Where, A is absorbance and t is the thickness. The absorbance is presented in Figure 3. Optical absorption coefficient versus wavelength graph is shown in Figure 4. The findings obtained from the optical properties shows that post-deposition heat treatment of ZnO thin film improves the migration of electrons from one area to another thereby causing an increase in grain size and decrease in grain boundary. The practical implication of increase of grain size and decrease of grain boundary means that conductivity will improve and more photon will be absorbed. The higher the annealing temperature, the lesser the sheet resistance and the higher the conductivity.

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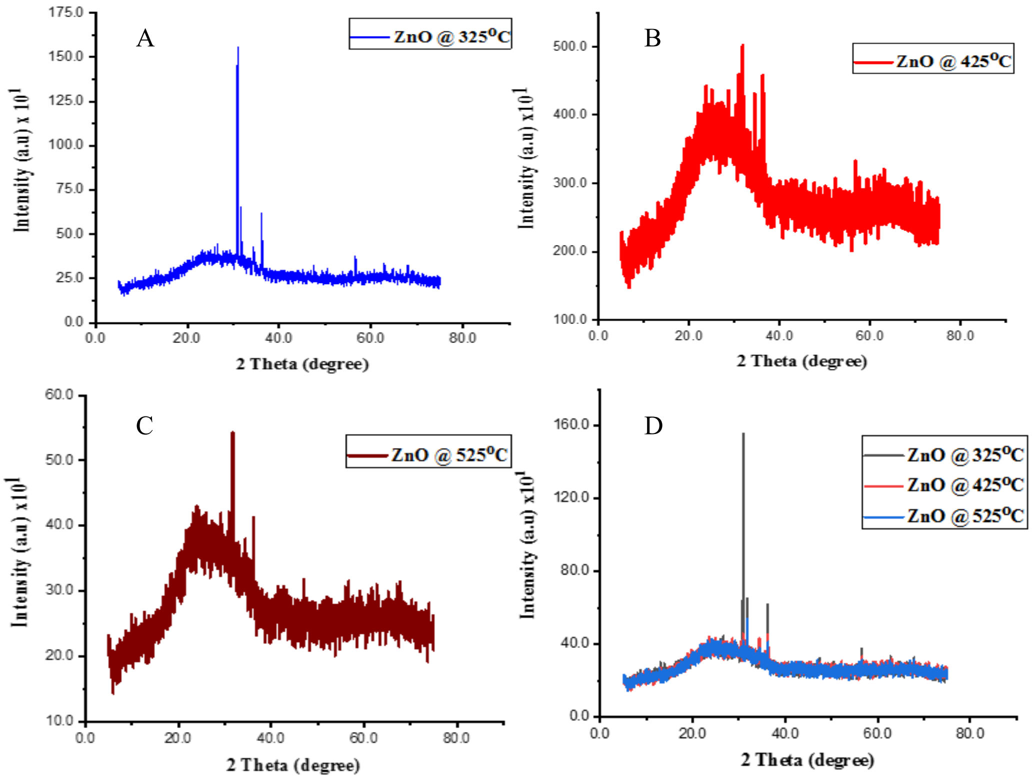

Fig. 1: X-ray diffraction spectra pattern of ZnO annealed at (a) 325 °C (b) 425 °C, (c) 525 °C and (d) combined spectra annealed at substrate temperature

3.3 Optical Band Gap Energy

The band gap was calculated using the Tauc plot method [32] and it was calculated from transmittance data obtained. The energy band gap were obtained from straight line plot of $(\alpha h\nu)^2$ versus $h\nu$ by extrapolating of the line.

$$(\alpha h\nu) = A(h\nu - E_g)^n \tag{3}$$

$$(\alpha h\nu) = A(h\nu - E_g)^{\frac{1}{2}}$$

$$(\alpha h\nu)^2 = A(h\nu - E_g) \tag{4}$$

Where $h\nu$ is the photon energy and A is a constant, optical band gap energy E_g. Where Photon energy intercept at $(\alpha h\nu)^2 = 0$, Equation (4) gives the optical band gap energies of the thin films at different thicknesses. Plot of $(\alpha h\nu)^2$ as a function of photon energy $h\nu$ as shown in Figure 5 The optical band gap energy range from 3.25 eV to 3.6 eV compared to ZnO wide and direct band gap (3.37 eV) at room temperature.

3.4 Morphological properties

Surface morphology was investigated with scanning electron microscope (SEM). SEM surface morphologies images of zinc oxide thin films annealed at 325 °C, 425 °C, and 525 °C, respectively are shown in Figure 6(a-c). Surface morphology of the films consist of spherical grains distributed on the surface. Zinc oxide annealed at 525 °C has large grain size compared with samples annealed at 325 °C and 425 °C. High density of small grain size can be seen in Figure 6(a) specimen annealed at 325 °C. SEM investigation shows that the grain size of the ZnO increased with higher annealing temperatures. It can be said that crystalline quality improves with increase

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Table 1: X-ray diffraction pattern of ZnO thin film annealed at temperature 325 °C

Pos. [2 theta degree.]	Height [cts]	FWHM [2 theta degree]	d- spacing [Å]
30.8922	1185.40	0.1279	2.89465
31.7074	303.42	0.1023	2.82207
34.3867	106.35	0.1535	2.60806
36.1840	346.43	0.1023	2.48254
56.5403	101.52	0.2047	1.62772

Table 2: X-ray diffraction pattern of ZnO thin film annealed at temperature 425 °C

Pos. [2 theta degree.]	Height [cts]	FWHM [2 theta degree]	d- spacing [Å]
30.9128	63.62	0.3070	2.82146
31.7143	127.14	0.2047	2.82146
36.2602	125.92	0.1535	2.47750

Table 3: X-ray diffraction pattern of ZnO thin film annealed at temperature 525 °C

Pos. [2 theta degree.]	Height [cts]	FWHM [2 theta degree]	d- spacing [Å]
31.7080	193.32	0.1023	2.82202
36.1991	104.40	0.1535	2.48154

in annealing temperature. The grain size of ZnO is large at higher annealing temperatures. With reference to Figure 6(a-c), the porosity improved with the increase of annealing temperature. We can deduce that the surface area and pore volume decrease with the increase of annealing temperatures. The pore diameter increases due to increase in the crystal size of ZnO [11]. The practical implication of increase of grain size and decrease of grain boundary means that conductivity will increase and more photon will be absorbed. This result confirms similar observations by Muslih and Kim [30] and Bedia *et al*. [33].

3.5 Electrical properties

Current-Voltage (I-V) measurement of annealed ZnO thin film was performed using four point probe Kiethley 2400 (SMU) equipment. The result is presented in Table 4. The-

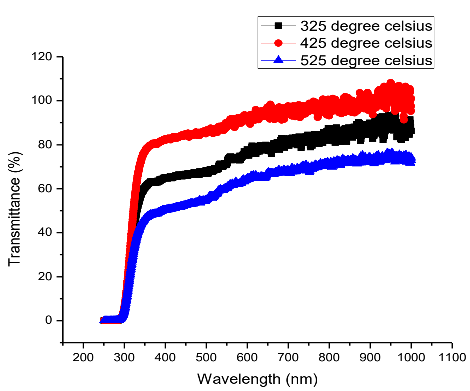

Fig. 2: Optical transmittance of ZnO thin film annealed at (a) 325 °C, (b) 425 °C and (c) 525 °C.

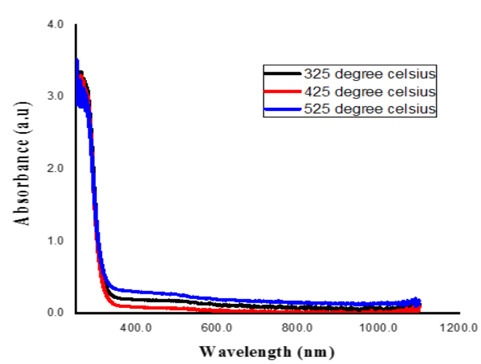

Fig. 3: Absorbance Versus wavelength

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summary of annealed ZnO thin film tabulated in Table 4 shows that increasing annealing temperature results in decrease in resistivity and increases the conductivity. The resistivity was calculated using Equation (5).

$$R = \frac{Ra}{l}(\Omega - cm) \quad (5)$$

Where ρ = resistivity (Ω-cm), R= Resistance (Ω), a = area (sq cm), l= length (cm).
Resistivity and conductivity are related by equation (6).

$$\sigma = \frac{1}{\rho}(Siemens) \quad (6)$$

Where σ = conductivity

The resistivity and conductivity values of the fabricated ZnO thin film were calculated using equations (5) and (6), respectively.

4. Conclusion

ZnO thin film was successfully deposited onto a glass substrate by spin-coating method. Temperature annealing was carried out on the deposited ZnO thin films. Scanning electron microscopy investigation showed that the grain size of the ZnO increased with higher annealing temperatures. It can be said that crystalline quality improves with increased annealing temperature and improve the migration of electrons within the ZnO thin film. Surface grain size increases with increase in

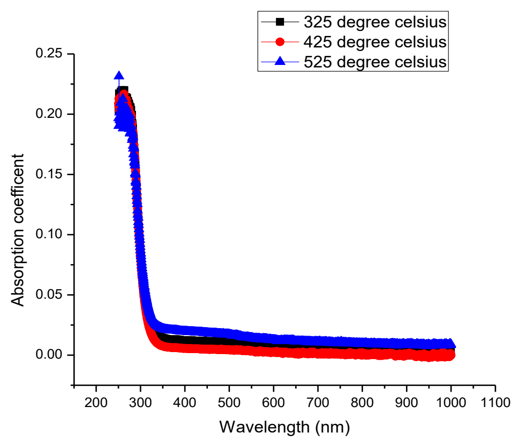

Fig. 4: Optical absorption coefficient of zinc oxide thin film

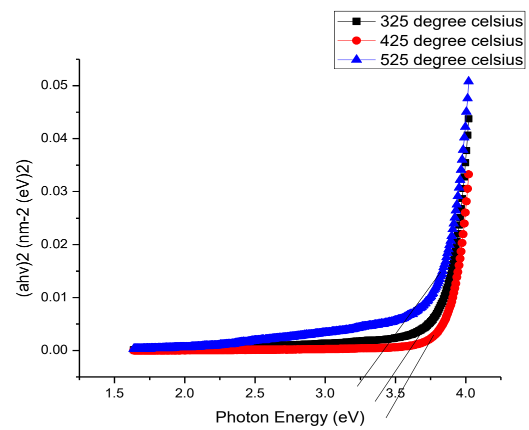

Fig. 5: Tauc plot of band gap energy of ZnO

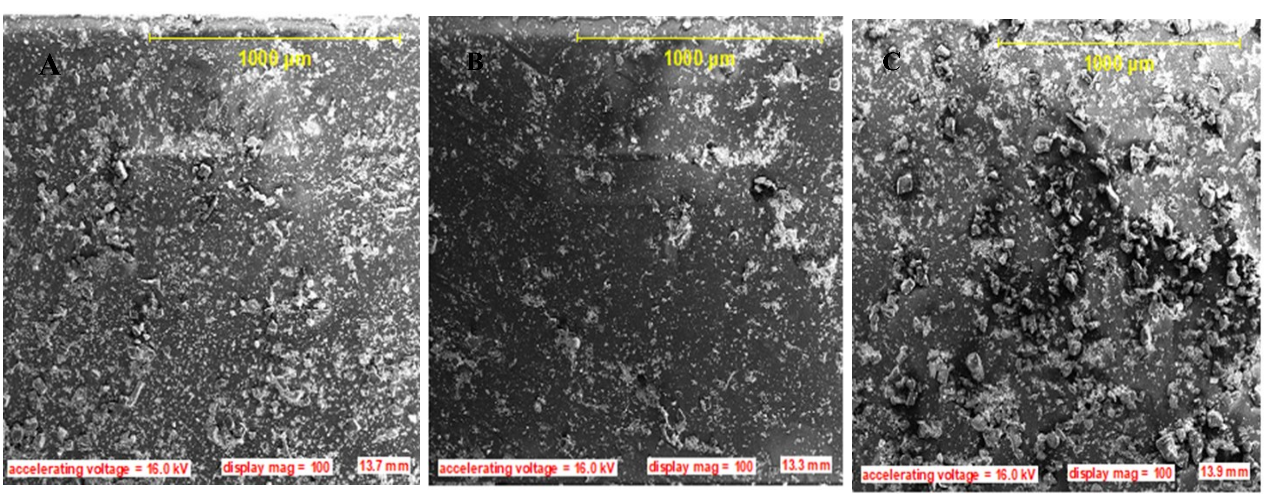

Fig. 6: Surface morphology images of ZnO thin film annealed (a, left) 325 °C, (b, middle 425 °C, and (c, right) 525 °C

Table 4: The sheet resistance of ZnO annealed under different temperatures

Annealed Temperature (°C)	Average sheet resistance Rs, (Ω/sq)	Average resistivity ρ (Ω-cm)	Conductivity (σ) (S/ cm) x 10^{-3}
325	5.2 x 10^{6}	103.8	9.6339 x 10^{-3}
425	2.3 x 10^{5}	4.60	217.39 x 10^{-3}
525	2.1 x 10^{5}	4.20	238.1 x 10^{-3}

annealing temperature. Temperature annealing improves the movement of electrons from one grain to another grain. The optical properties investigated shows transmittance of the ZnO thin film lies between 75 and 95% in the visible region of the electromagnetic wavelength spectrum. This transmittance within this range is desirable for the optical performance of very good electrode material. Band gap energy was obtained for each ZnO thin film annealed at different temperature. The optical band gap energy ranges from 3.25 eV to 3.6 eV compared to ZnO wide and direct band gap energy between 3.2 to 3.37 eV at room temperature. Absorbance of ZnO thin film increased with increase in annealing temperature. The crystal properties examined by X-ray diffraction reveal the crystallinity and structure of the film. It was observed to contain Wurtzite (100) and (101) oriented crystallites and corresponded well to typical crystal structure orientation of ZnO. ZnO thin film optical properties depicts it will be suitable as Transparent Conducting Oxide in electronic structures such as Solar cell, light emitting devices fabrication, and components to replace the expensive ITO and FTO. Further research work is ongoing on study of Argon gas effect on annealing process of ZnO thin film and introduction of mid layer between ZnO structures.

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Conflict of Interest

The authors declare that there is no conflict of interest.

Authors' Contributions

BSW conceived, designed, investigated, wrote and funded the study while **BSA** and **OOA** analyzed data and contributed to writing.

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