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The Effect of Deposition Temperature on Solid State Properties of Chemically Deposited Zinc Oxide Thin Films

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ZnO;

Abstract

The optical, structural, surface morphology and elemental composition of chemically deposited ZnO thin films at varying deposition temperatures were studied. The films deposited at room temperature had the highest transmittance of 70.52 %, and films deposited at 70^{o}C had an average transmittance of 38.85 %. The energy gap was between 3.52 and 3.62 eV. The structural properties showed that some of the films were amorphous and some were crystalline, as revealed by the XRD pattern. The diameters of crystals, micro lattice strain, and dislocation density of the crystalline films were measured with the Debye Scherrer. The crystallite size was between 49.1 and 60.91 nm, the micro lattice strain ranged between 6.35 and 8.60 x $10^{-4,}$ and the dislocation density was between 2,69 X 10^{14} and 4.13 X 10^{14} lines per metre cube. The films covered the substrate and were smooth but the films deposited at 40^{o}C, which showed some high particles on the surface as revealed by surface morphology. The elemental compositions of the films showed that zinc and oxygen were present for all the films except the films deposited at 40^{o}C, which had a low percentage of carbon.

Introduction

Zinc Oxide (ZnO) is a binary semiconductor formed from group II and VI elements from periodic table. This material has some unique properties such as a very wide band gap, strong

chemical and thermal stability, a high optical absorption coefficient, a large exciton binding energy (60 meV), and good piezoelectric ,high transmittance in the visible region and high electron mobility of about 2000 $cm^2/(V \cdot s)$ at 80 K (1) .The films of ZnO are applicable to a variety of electronic devices, including gas sensors, light-emitting diodes, laser-diodes, transparent electrodes, photoelectrochemical cells, and optoelectronic devices.(2). ZnO thin films have been deposited by variety of methods, such as thermal evaporation (3–5), sputtering (6,7), plasma-enhanced atomic layer (8),sol gel spin coating (9), spray pyrolysis (10) successive ionic layer adsorption and reacting (11,12) chemical bath deposition (13–17).

Literature survey shows that chemical bath deposition technique (CBD) is the most adopted to deposit ZnO thin films because of its simplicity, economical, large-area deposition capability, low pressure, and temperature requirements(18). In the presence study CBD technique was used to deposit ZnO thin films and influence of deposition temperature was studied on structural, optical, morphological and compositional properties of the films.

Experimental Procedure

Analytical grade Zinc nitrate ($Zn(NO_3)_2$ served as a source of zinc, triethanolamine for a complexing medium, ammonium hydroxide (NH_4OH) for pH adjuster, fluorine-doped tin oxide (FTO) served as the substrates and distilled water served as solvents.. The substrates were cleaned by dipping them in an acetone solution with ultrasonic vibration for 15 minutes. They were then rinsed with de-ionizes water and kept in a desiccator.

ZnO films were coated on FTO substrates using CBD method. The film precursor was formed by mixing 300 ml of ($Zn(NO_3)_2$ at 0.1 M with 100 ml of triethanolamine at 0.1 M in a 500 ml beaker under continuous stirring at room temperature. The pH of the solution was adjusted by adding ammonium hydroxide (NH_4OH) until the pH of the solution was between 10 and 12 to form an ammonium zincate bath. The solution was transferred into five (5) chromatography tanks and a pre-cleaned FTO substrate was vertically inserted in each of the tanks. Four of the thanks were placed in four (4) different water baths heated at 40, 50, 60 and 70^oC, respectively. The fifth tank was not heated and the film was allowed to be deposited at room temperature. The time of deposition of 60 minutes was maintained for all the depositions. At the end of deposition time, the substrates were removed, washed with distilled water and heated in an oven at 200^oC for one hour.

The structural characteristics of the films were measured with Pan Analytical Empryrean X-ray diffractometer Tokyo, Japan and set to produce diffractions at a scanning rate of 2 0/min in the 2 to 70^o at room temperature with a CuKa radiation set at 40kV and 20mA. The crystallites size (D) micro strain (ϵ) of the films and dislocation density were determined according to Equation 1,2 and 3, respectively.

$$D = \frac{k\lambda}{\beta cos\theta} \text{ ……(1)}$$

$$\epsilon = \frac{\beta cos\theta}{4} \text{…….. (2)}$$

$$\delta = \frac{1}{D^2} \text{…….(3)}$$

The nature of films at the surface of substrates was measured with JEOL-JSM 7600F scanning electron microscope (SEM) (Japan), A constant 20 kV was used as the accelerating voltage. To enable comparison research into the variations in the composition of the thin films, all of the SEM micrographs were acquired at the same magnification. The compositional analysis of the films was measured with Energy Dispersive X-ray Spectroscopy.

Avantex UV visible spectrophotometer was used to measure the thin films' optical transmittance in the wavelength range of 239.534 nm to 999.495 nm. The spectrophotometer provided the film transmittance measurements.

The absorbance (A) was calculated from the percentage transmittance (T) according to Eqn. 4

$$A = 2 - logT \text{…….. (4)}$$

The reflectance (R)was obtained from Eqn.5.

$$A + T + R = 1 \text{…….(5)}$$

The absorbance coefficients (α) of the films was calculated according to Eqn.6.

$$\alpha = \frac{2.303A}{t} \text{……(6)}$$

where A is absorbance and t is the thickness of the film.

The photon energy (E) was calculated in eV according to Eqn. 7

$$E = \frac{1243}{\lambda} \text{……(7)}$$

The connection between absorbance coefficient (α) and incident photon energy (hv) is shown in Eqn. 8

$$\alpha h\nu = A\sqrt{h\nu - E_g} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots(8)$$

Results and discussion

The structural pattern of ZnO thin films deposited at varying deposition temperatures displayed in Fig. 1 (a-e). The patterns showed that the ZnO thin films had both polycrystalline and amorphous structures. A similar pattern was observed when ZnO thin films coated by thermal evaporation and annealed at different temperatures (4). The films deposited at room was polycrystalline, at 40^{o}C was amorphous, at 50^{o}C was polycrystalline, at 60^{o}C was amorphous and at 70^{o}C was polycrystalline. The carbon impurity atom detected by energy dispersive spectroscopy could be a factor that caused the irregular pattern of the films deposited at 40^{o}C. The examination of the surface morphology of the films showed that the films deposited at 60^{o}C had an irregular shape and more voids than other films; this may have resulted in the amorphous pattern observed in the XRD results.

The peaks of polycrystalline structure occurred at 2θ = 26.45, 33.64, 37.69, 51.43, 61.46, and 65.41^{o}, indexed with (100), (101), (110), (002), (102), and (112), respectively. The diffraction lines were similar to the values reported in the database of ZnO (JCPDS card no. 89-1397), providing a strong indication for the creation of a hexagonal Wurtzite-type pattern in the synthesized ZnO ((19) .The average crystallite size and micro lattice strain of polycrystalline ZnO films were calculated according to Debye Scherrer equations, and the dislocation density was calculated according to Equation 3 as presented in Table 1. The Table shows that as deposition temperature increased, average crystallite size decreased while micro lattice strain and dislocation density increased. The reduction in crystallite size can be attributed to an increase in the strain as the deposition temperature improved. The obtained crystallite sizes were within the range of crystallite sizes between 9 and 71 nm when thin films of ZnO were formed by the sol-gel method (9). The calculated crystallite size was higher than the value between 29.40 and 29.47 nm in the ZnO films grew on an n-type silicon substrate by thermal evaporation (20) .The difference in method of deposition and substrate used could be a factor in the disparity.

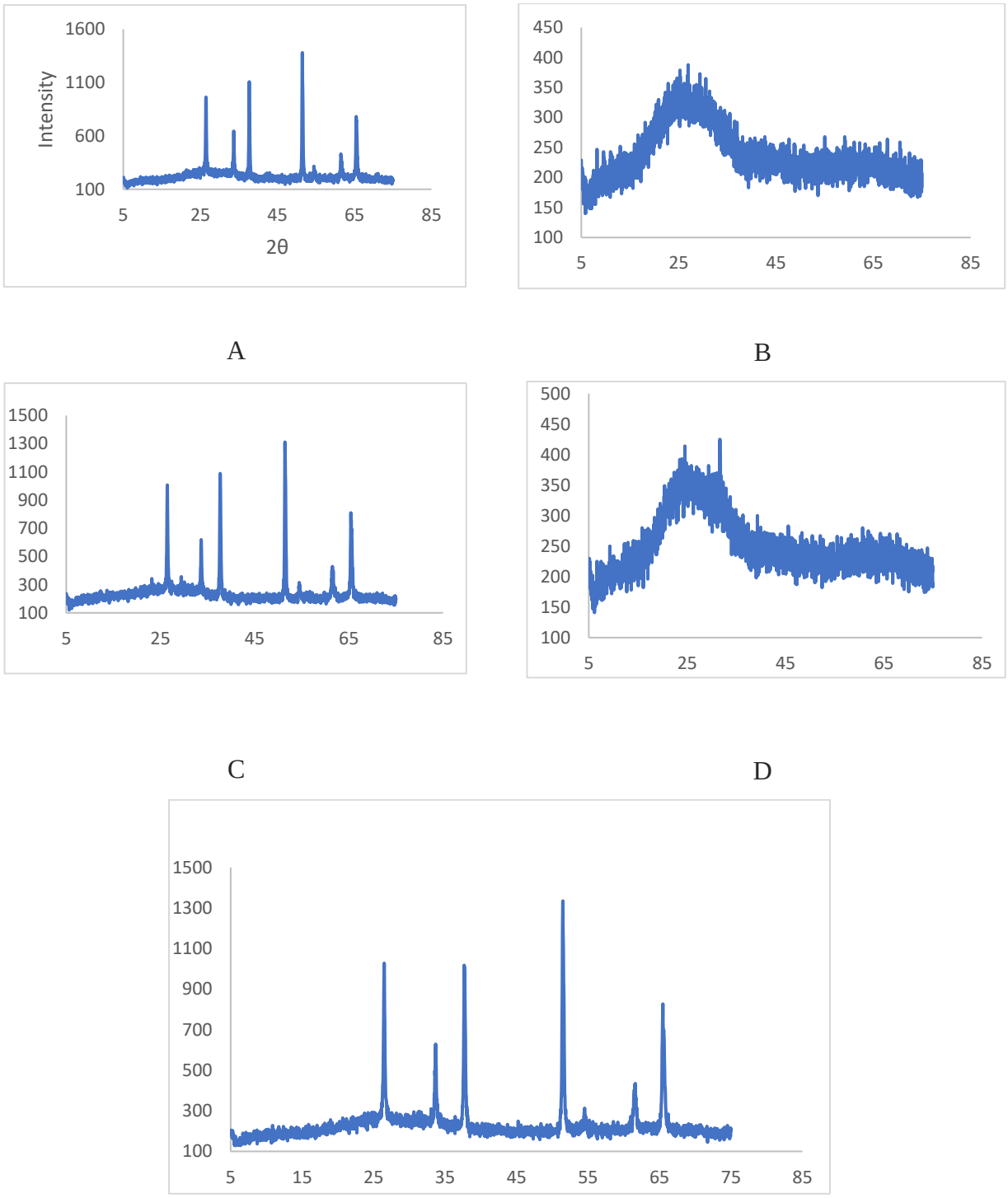
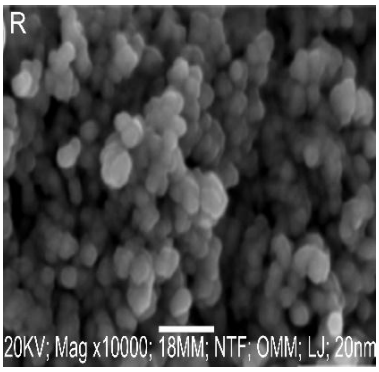


Figure 1 Structural pattern of ZnO thin films deposited at (a) room temperature (b) 40oC (c) 50oC (d) 60oC (e) 70oC

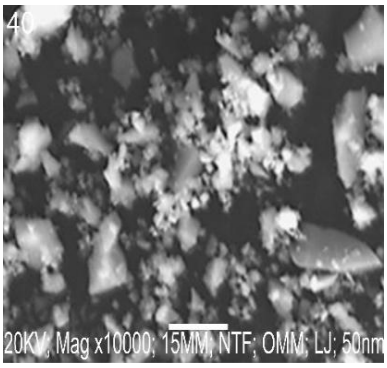
Table 1 Structural Properties of ZnO thin films at different deposition temperatures

Sample	Crystallite size D (nm)	Micro lattice strain ϵ x 10^{-4}	Dislocation density δ x 10^{14} Line/m^2
at room temperature	60.91	6.35	2.69
at 50^{o}C	60.02	7.28	2.78
at 70^{o}C	49.21	8.60	4.13

Also, the surface morphological analysis of zinc oxide films deposited at different temperatures, as presented in Fig. 2(a–e), revealed that the substrates were covered with the films, and the films deposited at room temperature were smooth on the surface of the substrate. The implication of this smoothness was observed in the transmittance of the film. The film deposited at 40 ^{o}C was rough; this may be because of the presence of impurities in the film, as revealed by energy dispersive spectroscopy. The surface morphology of films grew at 50, 60, and 70^{o}C was similar. The increase in the temperature of deposition influences a rise in the firmness, homogeneity, and uniformity of films. The surface morphologies of ZnO films created by bath deposition were similar to the morphologies at surface of CuS thin films coated at different deposition temperatures (18).



A



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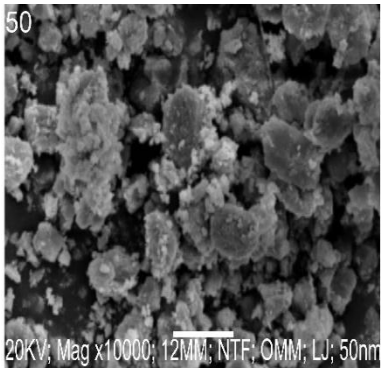
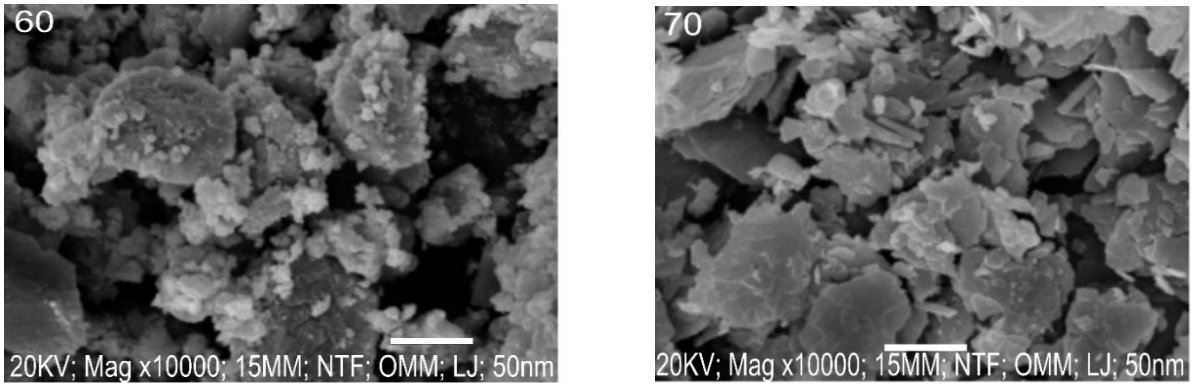


C



D E

Figure 2 SEM images of ZnO thin films deposited at (a) room temperature (b) 40oC (c) 50oC (d) 60oC (e) 70oC

The composition of ZnO thin films deposited at varying depositions, as revealed by energy dispersive spectroscopy in Fig.3 (a–e). The analysis showed that zinc and oxygen made up the film. The atomic percentages of zinc and oxygen were 89.74 and 10.28, 74.78 and 22.00, 54.80 and 45.20, 67.80 and 33.20, and 69.78 and 30.22 for films deposited at room temperature, 40, 50, 60, and 70^{o}C, respectively. This result showed that the percentage of zinc deposited decreased and the percentage of oxygen increased as the deposition temperature rose from room temperature to up to 50^{o}C. The atomic composition of the films deposited at 50^{o}C only was near to the stoichiometric value of ZnO thin films and close to 54.66 and 45.34 % for zinc and oxygen, respectively, as reported in the films of ZnO synthesized by reactive radio frequency magnetron sputtering (21). Although non-stoichiometric ZnO was reported in the EDS of the films deposited by electrospinning technique, the films showed a higher percentage of impurities that arose from the background of the substrate(22). However, the films deposited at 40^{o}C had a low percentage of carbon as an impurity; the source of the impurity may come from the annealing chamber. Carbon impurity atoms were detected in the films of ZnO prepared by the zinc air cell system method, and the source of carbon was traced to either the handling condition of the sample or as a result of annealing in the oven (1). The energy dispersive spectroscopy shows that the films were mainly ZnO, and the smoothness and roughness of the films were affected by the percentage of zinc present in the films, as shown in the SEM images.

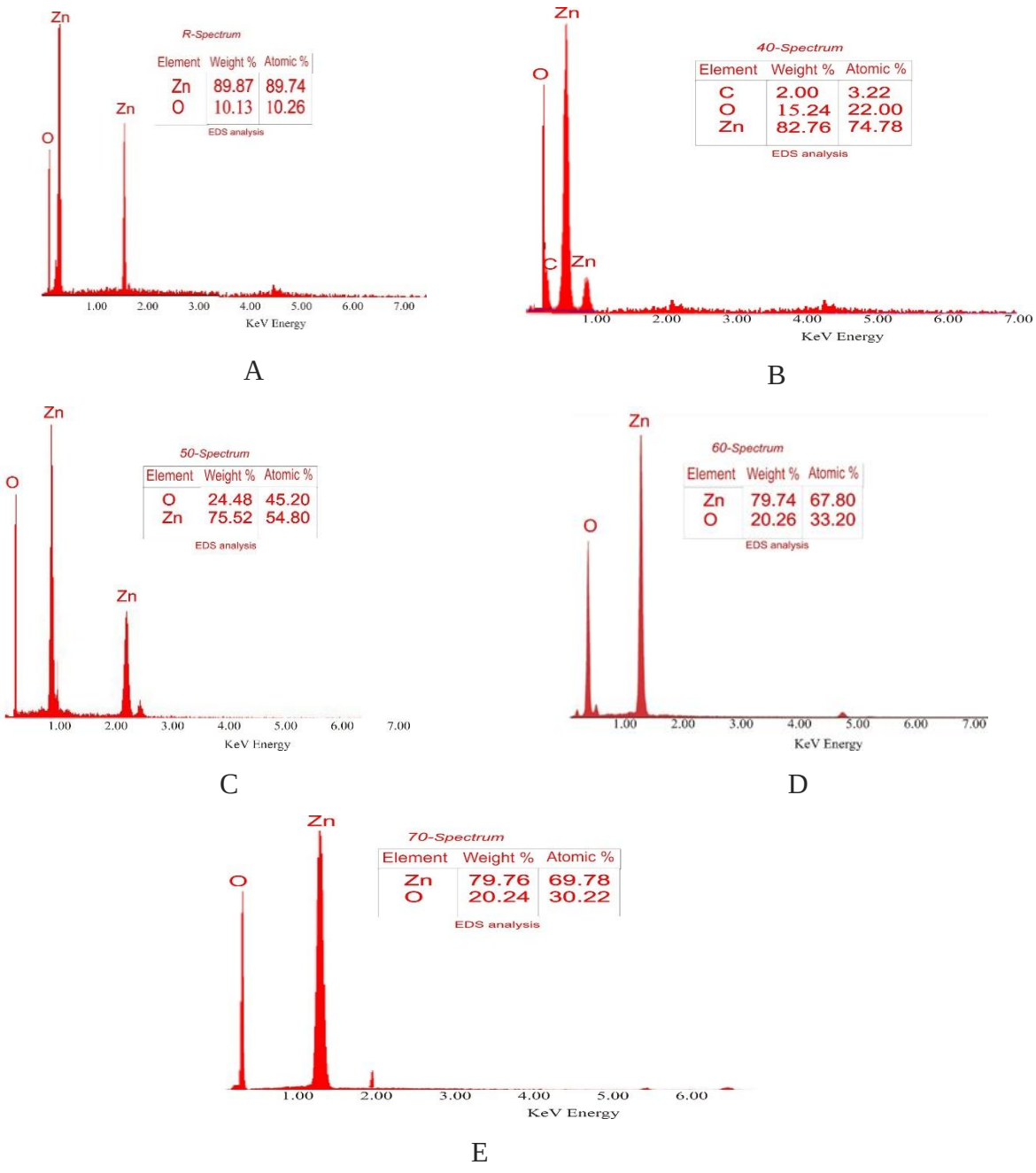


Figure 3 EDS of ZnO thin films deposited at (a) room temperature (b) 40oC (c) 50oC (d) 60oC (e) 70oC

ZnO thin films transmittance spectral is presented in Fig.4, the transmittance was very high in the visible range of electromagnetic region. The transmittance of the films coated at room temperature was between 55.92 and 85.12 % measured between 400 and 700 nm. The film deposited at 40^{o}C had minimum transmittance of 45.39 % at 400.56 nm and maximum

transmittance of 71.84%, the maximum transmittance occurred at wavelength 694.74 nm. The film of ZnO deposited at 50^{o}C transmitted 45.35% minimum and 71.84% maximum measured between 400 and 700 nm. The transmittance range of films deposited at 60^{o}C and 70^{o}C is 51.08 to 85.54 % and 28.97 and 48.72 %, respectively. In general, the average transmittance was between 38.85 % and 70.52 %, which corresponds to the average value between 30 % and 70 % observed by (23). However, the average value of the transmittance of the film deposited at room temperature was highest due to the smooth arrangement of the films on the substrate. However, the transmittance of the film deposited at 70^{o}C gave the least average transmittance because the film had the least particle size of 49.22 nm.

The absorbance spectral of ZnO is presented in Fig.5 showed that maximum absorption occurred in ultra-violent range and continuously decreases in the visible part of electromagnetic spectrum. This agrees very well with the absorbance spectral observed by (24–26).The average percentage of photon adsorption of films deposited at room temperature, 40, 50, 60 and 70^{o}C were 17.20, 25.50, 24.60, 18.10 and 42.60%, respectively. The result revealed that the photon adsorption increases as the deposition temperature increases but the film of ZnO deposited at 40^{o}C had lower adsorption than film deposited at 50^{o}C. The reason could be the presence of carbon impurity atom in the films deposited at 40^{o}C as confirmed by EDS measurements. The films surface morphology revealed that the films roughness jumped up as the deposition temperature improved. This may be a factor causing the reduction in photon adsorption with increasing deposition temperature because light can be transmitted in smooth surface better than on a rough surface; when transmittance is higher the absorbance will be lower and this is similar to the report of (27) . However, the films deposited at 60^{o}C had an absorption of 18.1%, the reason for this could be due to a low scattering effect resulting from the uneven distribution of particle on the substrate which allows higher transmittance of photons. It is also observed from the investigations that the films coated at 70^{o}C had higher average adsorption of 42.6%. because the film transmitted the least photons in the visible region, low porosity of the films as seen in the SEM image could be a reason.

The reflectance spectral of ZnO films as depicted in Fig. 6 revealed that the reflectance started in the UV region at wavelength 315.54 nm and increased slowly to the visible and infrared regions. The reflectance measured in the visible region between 400 and 700nm were between 1.13 and 1.77%; 1.10 and 1.15%; 1.11 and 1.57%; 1.12% and 1.78 %; 0.74 and1.17 % the for films

deposited at room temperature, 40, 50, 60 and 70^{o}C, respectively. It is generally observed that the film exhibited low reflectance between 0.78 and 1.78 % which is similar to the value of the reflectance observed by(28). The low reflectance of the film is an indication that ZnO can transmit a very good percentage of incident radiation to the absorber layer where conversion of solar to electrical energy takes place.

The absorption coefficient of ZnO is measured from absorption coefficient spectral displayed in Fig. 7. It was observed that the adsorption coefficient of all the samples is greater than 10^{6} m^{-1}. This is similar to reported adsorption coefficient of greater than 10^{4} cm^{-1} which indicates the improvement in chance of the occurrence of direct transitions as observed by (29), The adsorption coefficient increases slowly as photon energy increases well. This is evidence of the contact of the films charge carriers with the incident photons that have enough energy for the occurrence of electron transitions (30).

Similarly, the bandgap of the films is extrapolated from Fig.8 and the it was between 3.52 and 3.62 eV. The values are in good agreement with the energy gap between 3.10 and 3.97 eV reported when studying stirring's impact on ZnO thin films that have been chemically deposited (31). The values were higher than the band gap between 3.11 and 3.22 eV in the research by (32) when the physical characteristics of ZnO thin films produced by Sol-gel were examined in relation to the influence of precursor solution and annealing temperature.

The difference in the methods of deposition could be a reason for the higher value because sol-gel method always has a thicker thin film than the chemical deposition method. When the film thickness increases, the adsorption increases and causes low transmittance, which reduces the bandgap. It was observed from the results that the bandgap improved slightly as deposition temperature rises due to increases in clustering of the particles as observed in the SEM morphology, but the films deposited at 40^{o}C exhibited the least band gap energy of 3.52 eV the reason for this could be the presence of impurity atom as seen in the EDS result. Similar results were obtained when ZnO films were coated by pulsed layer method at varying deposition temperatures between 200 and 400^{o}C and band gap rose from 3.3 to 4.19 eV for unannealed films and 3.58 to 3.98 eV after annealing. The increment was attributed to a shift in burstein electron density (23).

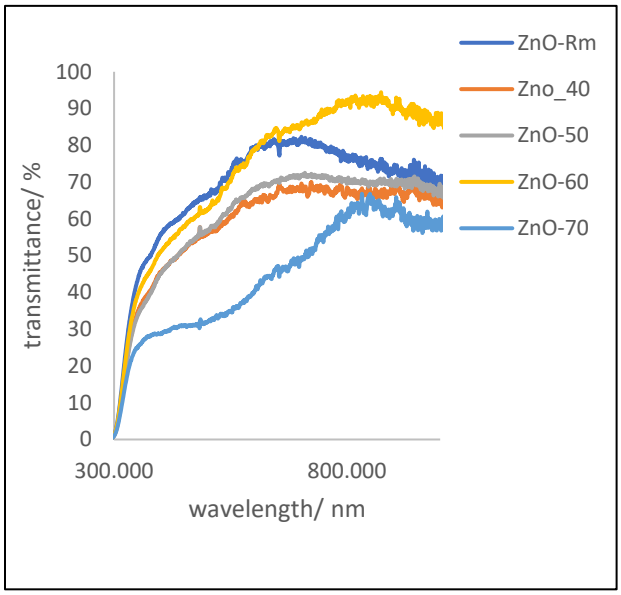


Figure 4: The transmittance spectral

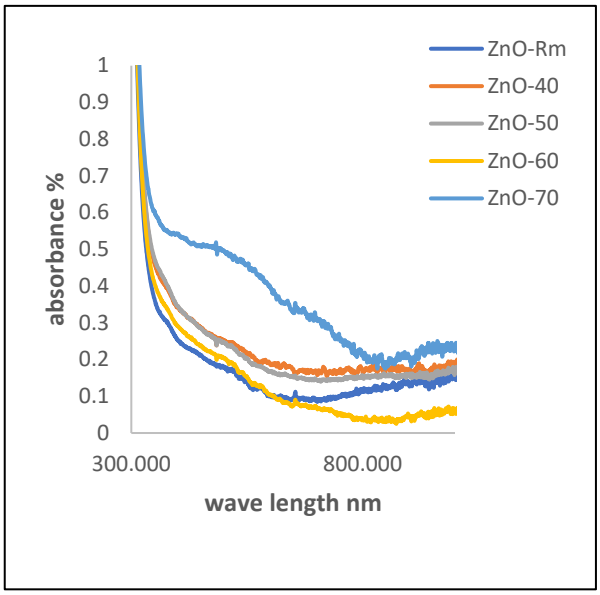


Figure 5: The absorbance spectral

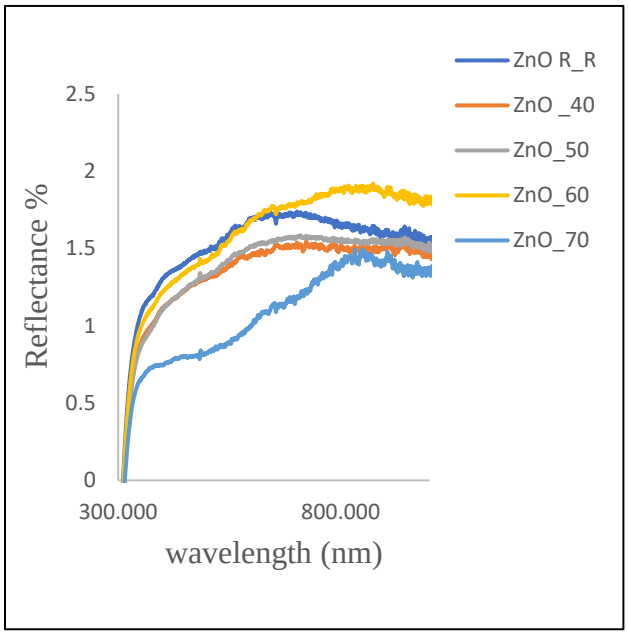


Figure 6: The reflectance spectral

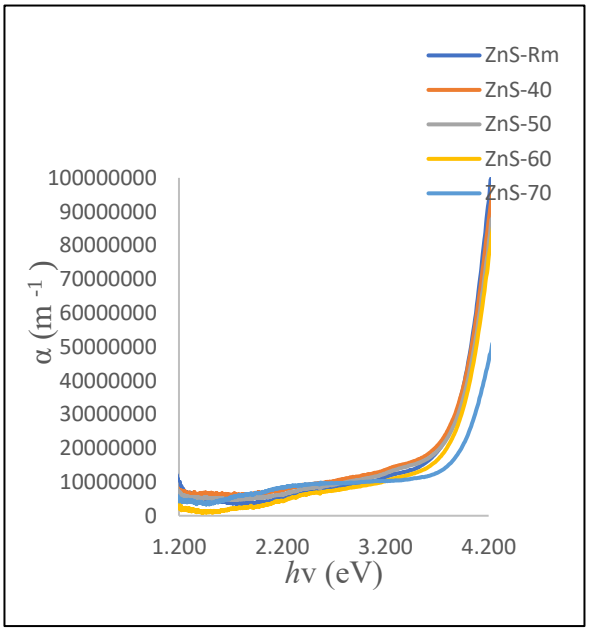


Figure 7: The absorbance coefficient spectral

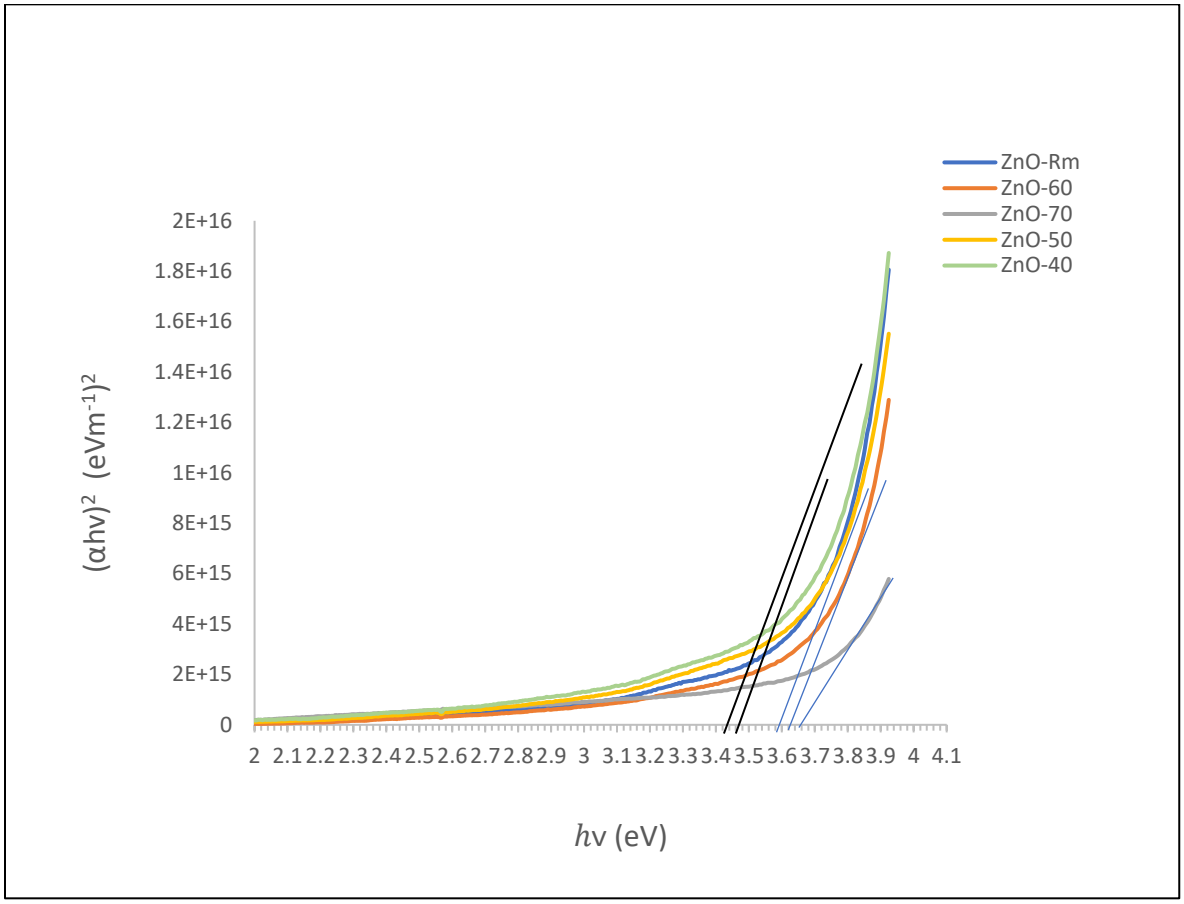


Figure 8: Plot of (α*h*v)2 against photon energy for ZnO at various deposition temperatures

Conclusions

ZnO thin films were successively grew on FTO substrates by chemical bath deposition technique and the effect of deposition temperatures were studied on some solid state properties of the films. The structural properties were measured with XRD and the results showed the films had both amorphous and crystalline pattern, The nature of the films at substrates as revealed by SEM showed that the substrates were covered with the films, and the film deposited at room temperature was smooth on the surface of the substrate while the surface morphology of films deposited at 50, 60, and 70^{o}C was similar. The transmittance of the film deposited room was the highest while the film deposited at70^{o}Chad the least transmittance in the visible part of electromagnetic spectrum.

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