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Effects of Deposition Time and Temperature on the Optical properties of Chemically Deposited ZnO thin film

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Abstract
Zinc Oxide thin film was successfully deposited using chemical bath deposition method. The optical and morphological measurements were reported using ultra-violet visible spectrophotometer and Scanning Electron Microscope respectively. The elemental composition was investigated using Energy Dispersive X-ray and the result showed peaks corresponding to the two main elements (Zinc and Oxygen) only, signifying the purity of the deposited films. The optical property was observed to depend on the deposition times and temperature. The films' percentage transmittance was observed to reduce with increasing deposition time, with the film sample deposited for 30 min exhibiting the highest percentage transmittance of 82.7% among the samples deposited for the same temperature. Reduction in the deposition temperature to 60 ^{o}C increased the films' transmittance to the highest value of 84.8%. The direct band gap energy values increased from 3.47 to 3.51 eV when the deposition time was increased from 30 to 60 min and reduced to 3.46 eV when the deposition temperature was reduced from 70^{o}C to 60^{o}C. Urbach energy was deduced from its exponential relationship with photon energy and the result showed that the value increase with increasing deposition time but decreased with deposition temperature. These results from this study revealed that ZnO thin film possessed the required properties suitable for its application as transmission conducting oxide in solar cells.

Keywords: Band gap energy, Urbach energy, Chemical bath deposition method, Transmittance, SEM

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Introduction:

Over the past century, Zinc oxide (ZnO) has received extensive attention as a result of its interesting optical, chemical, and electrical properties, as well as its non-toxic, low-cost, and abundant constituents. ZnO thin film has a direct wide band gap energy of 3.37 eV at room temperature and a large exciton binding energy of 60 meV (Osanyinlusi *et al.*, 2016). The large band gap of ZnO allows for higher breakdown voltages, sustenance of large electric fields, high temperature, and high-power operation. ZnO thin films have been widely employed in several modern applications including, surface acoustic wave systems, varistors, gas sensors, solar cell transparent contact fabrications and UV laser (Tangade *et al.*, 2020).

Among the different techniques that have been used in preparing ZnO thin films are; thermal evaporation (Memarian, 2017), chemical vapor deposition (CVD) (Carlsson, *et al.,* 2010), vapor-liquid-solid (VLS) (Redwing, *et al.,* 2015), chemical bath deposition (Chai, 2009), successive ionic layer adsorption (SILAR (Patil, *et al*, 2017) and spray pyrolysis (Sahay, *et al*, 2008). However, most of these techniques mostly require special equipment, complex process mechanism, or high temperatures, which are unfavorable for industrialization (Seshan, 2002). Chemical bath deposition technique has been adjudged to be the most attractive deposition method for the production of uniform thin films over large areas because of its simplicity, convenience and freedom from vapor deposition associated with other

high-temperature fabrication techniques (Chai, 2009).

A thin film comprises of one or more layers of material with thickness in the order of nanometer to several micrometers. Thin film deposition is essential in different applications requiring deposition of thin layer on a substrate or on existing layers (Ohring, 2001).

The preparation of thin films using chemical bath deposition (CBD) method involves immersing a cleaned substrate in a heated solution containing precursor metal salts and other additives like the complexing agent. A controlled chemical reaction will bring about the deposition of the thin films on the substrate. This method can be employed to deposit metal oxides from aqueous solutions, usually alkaline and contain a base (as a source of hydroxide), a metal ion and a complexing agent to control the hydrolysis of the metal ion (Winkler *et al.*, 2018). The properties of chemically deposited thin films depend largely on the major process parameters such as bath temperature, deposition time and complexing agents (Gonzalez-Chan, *et al.*, 2019).

Some previous research works ascertained that variations in the deposition time and temperature have potential effect on the optical properties of ZnO thin films. For instance, Siregar *et al.*, (2023) reported that with the increase in deposition time of ZnO thin films prepared by electroplating technique, the values of transmittance decrease. They associated this outcome to continuous coating of the substrate with more ZnO atom leading to increase in thickness as the dipping time increases. This resulted in more frequent collisions between light and ZnO particles making it more difficult for light to pass through. They also observed that the band gap energy reduced with prolonged deposition time. Ungula *et al*, (2024) also recently investigated the influence of deposition time on ZnO nanorod deposited unto GZO seed layer using CBD technique. They discovered a declining of percentage transmittance with prolonged deposition time and attributed the behaviour to enhanced film's density with longer deposition time. Some other researchers have shown that deposition temperature has a significant impact on the optical properties of semiconductor thin films. Ke *et al.*, (2014) examined the effects of bath temperature on the properties of ZnS thin films and reported that the thickness of the films significantly increase with increased deposition temperature from 50 ^{o}C to 90 ^{o}C. They also reported an initial decrease in band gap energy from 4.06 to 3.93 eV as deposition temperature increased from 50 ^{o}C to 70 ^{o}C, and later increased to 4.03 eV at 90 ^{o}C deposition temperature. To the best knowledge of the researcher, little reports are available regarding the effects of deposition temperature on optical properties on ZnO thin film. This study therefore aimed at investigating the influence of deposition time and temperature on the optical properties of ZnO thin films.

Materials and Methods

Experimental Details

Chemical bath deposition technique was used to deposit ZnO thin films unto a commercially available glass slides with dimension 25.4 mm×76.2 mm. Before use, the glass slides were ultrasonically washed using acetone, isopropanol and distilled water, in order to remove dusts and other contaminants. Firstly, 50 ml 0.1 M zinc acetate dihydrate ($C_4H_6O_4Zn.H_2O$) solution was prepared using distilled water inside a 100 ml beaker and stirred for 20 min using magnetic stirrer in order to obtain a homogenous solution. Under continuous stirring, ammonia solution was added in drop, turning the solution milky but become colourless with excess ammonia. The pH of the bath was adjusted to 12.3 using an earlier prepared 4 M sodium hydroxide (NaOH) solution. Distilled water was afterward added to the solution to make up 100 ml. The pre-cleaned glass slides were vertically dipped into the solution and the temperature maintained at 70 °C under continuous stirring using magnetic stirrer with heater. The thin films were subsequently removed after 30, 45 and 60 min, rinsed with isopropanol in order to remove loosely attached particles and thereafter air-dried. Following the same procedure, another ZnO thin film sample was prepared for 60 min with the bath temperature maintained at 60°C in order to examine the impact of varying deposition temperature. Finally, all the prepared film samples were annealed at 200°C for 2 hours in order to enhance the structural quality and adherence to substrate. The samples were labelled ZnO-1, ZnO-2 and ZnO-3 for films deposited at 70°C for 30, 45, 60 min respectively and ZnO-4 for film deposited at 60°C.

The transmission and absorption measurements were carried out using Uv-vis spectrophotometer (SPECORD 200 PLUS double beam Uv-vis spectrophotometer). Surface morphology and elemental composition were studied using scanning electron microscopy coupled with energy dispersive X-ray analysis (EDX) spectrometer (JEOL JSM 7600F Field emission SEM).

Results and Discussion

Elemental Analysis

Energy Dispersive X-ray (EDX) analysis was used to examine the elemental compositions of the thin film. Figures 1 (a and b) shows the EDX spectra for the ZnO-3 and ZnO-4 thin film samples. The peaks of the spectra are quite distinct and show the presence of the two major elements; zinc and oxygen. There are no observed peaks associated with any other element, which signifies the purity of the prepared films. The atomic percent of each element was calculated using their respective percentage weight. The calculated atomic percent are, 49.42% (Zn) and 50.58% (O) for the ZnO-3 sample as well as 45.79% (Zn) and 54.21% (O), for ZnO-4 sample, indicating Zn/O ratio of 0.98 and 0.85 respectively. This result show that the prepared ZnO film possess compositional ratio that is approximately close to the stoichiometric composition within the reasonable experimental error.

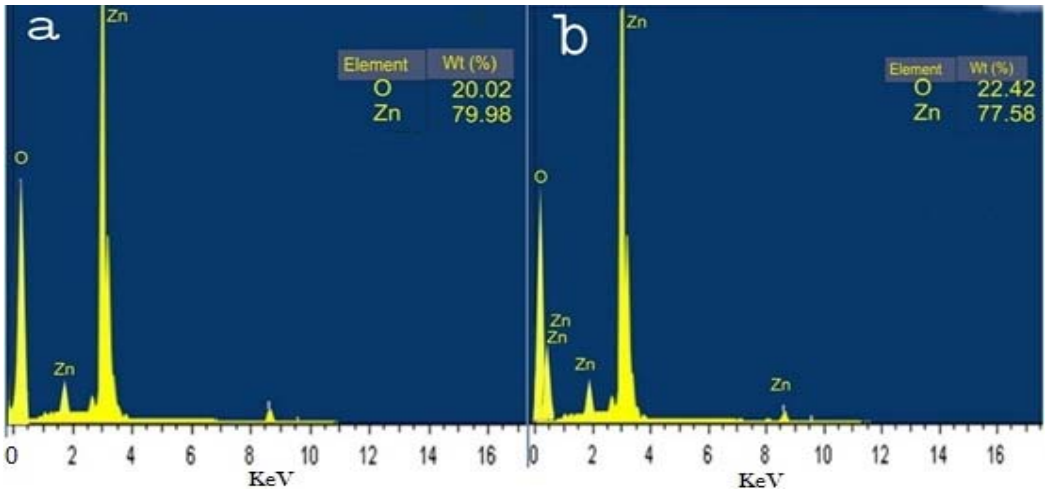

Figure 1: EDX spectra of (a) ZnO-3 and (b) ZnO-4 thin film sample.

Surface Morphology

The surface morphology of the prepared films was investigated using Scanning Electron Microscopy (SEM). Figure 2 (a and b) presents the SEM micrographs for the ZnO-3 and ZnO-4 thin film samples. It can be observed from the figure that the surface of the ZnO-3 film sample appears smooth with inhomogeneous grains of different shapes. The film covers the substrate well with no visible crack or pore. For the ZnO-4 sample, the surface looks more porous but compacted, and the grains were observed to be of almost equal in size with spherical shape.

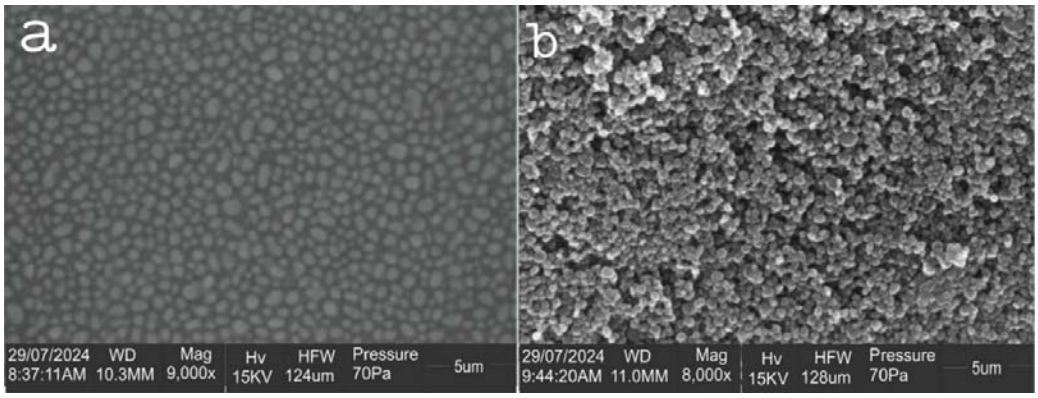

Figure 2: SEM micrograph of (a) ZnO-3 and (b) ZnO-4 samples.

Optical Properties

The optical properties of the prepared ZnO thin film samples were determined from transmittance measurements taken in the 300-700 nm wavelength range. Figure 3 shows the plot of transmittance against wavelength from where the maximum visible transmittance of 82.7%, 78.1%, 73.3%, and 84.8% were respectively observed for the ZnO-1, ZnO-2, ZnO-3, and ZnO-4 samples. These observed values of maximum transmittance are similar to the value reported by Khelladi *et al*., (2013), and are within the acceptable transmittance values suitable for application as transmission conductor oxide in optoelectronic devices like solar cells (Cisneros-Contreras *et al.*, 2023). It is clear from the figure that deposition time exhibits significant impact on the optical transmittance, with an increase in deposition time resulting in the reduction of percentage transmittance. The observed reduction in transmittance may be attributed to the enhancement of film's thickness as the deposition time increases (Hariech *et al*., 2022). The Figure also revealed that ZnO-4 sample prepared at a reduced temperature of 60°C exhibits the highest transmittance within the visible region (84.8%) among the prepared film samples. This value is significantly higher when compared with ZnO-3 sample prepared for the same time but at different temperature of 70°C (73.3%). Similar occurrence has been earlier reported by Kim *et al*., (2020) for ZnO thin films prepared using CBD method, and this may be attributed to the increase in the film's growth rate at high temperatures resulting in thicker films (Chen *et al*., 2024).

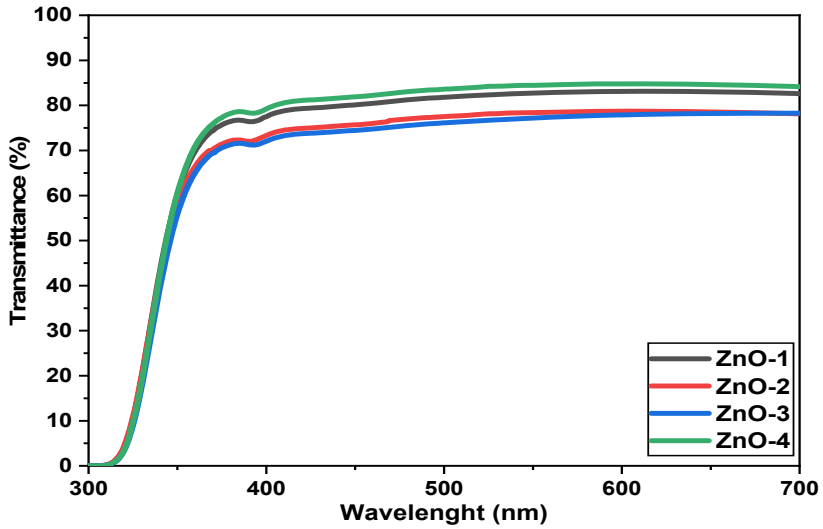

Figure 3: Transmittance spectra for the prepared ZnO thin film samples.

The absorption spectra for the deposited ZnO thin film samples measured between 300 and 700 nm wavelength range is presented in Figure 4. It is observable from the figure that all the samples display low absorption values, and remains almost constant all through the visible wavelength. It is clear from the figure that the film sample with the highest deposition time and lowest transmittance (ZnO-3) has the highest absorbance, and this agrees with the result of Abdulrahman *et al*., (2017). Variation in deposition temperature is seen to also affect the film's absorption property, as the film samples deposited for the same time but at different temperature (ZnO-3 and ZnO-4) exhibited significant difference in light absorption. The film sample deposited at 60 °C and with the highest transmittance (ZnO-4) presents lower absorption of 0.10 when compared to the sample deposited at 70 °C with absorption value of 0.19 which aligns with the work of Abdulrahman *et al*., (2017).

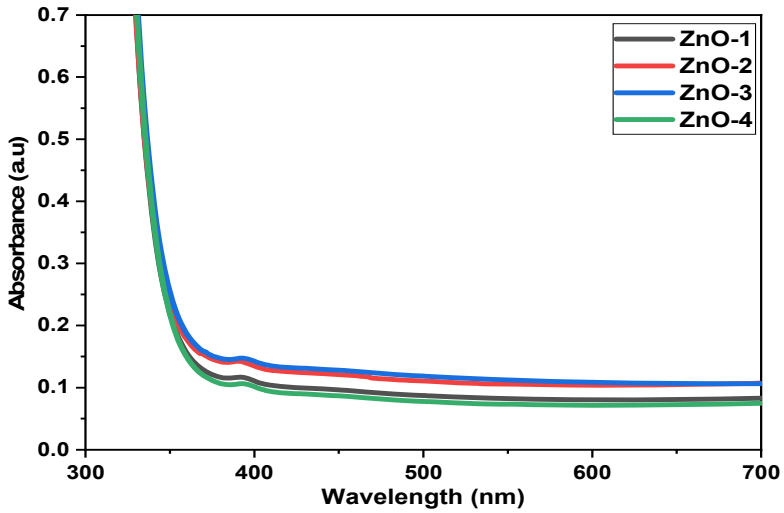

Figure 4: Absorbance spectra for the prepared ZnO thin film samples.

The Lambert equation which relates the absorption coefficient (α) with the film's light absorption (A) and thickness (t) has been employed in determining the film's absorption coefficient (Inbanathan *et al*., 2021);

$$\alpha = \frac{2.303A}{t} \qquad (1)$$

The plot of absorption coefficient against light wavelength for the prepared samples has been displayed in Figure 5. From the graph it can be seen that all the film samples exhibit absorption coefficient values in the order of 10^4 cm^{-1}. The spectra shows that the film deposited at a lower temperature presents the lowest value of absorption coefficient across the entire wavelength, which follows the trend observed in the absorbance spectra. From the figure, the film sample deposited at a lower temperature exhibited the lowest absorption coefficient. This can be attributed to differences in surface morphology. At higher deposition temperatures, film surface becomes more uniform, bringing about improved light interaction and higher absorption coefficients. In contrast, at lower temperatures film surfaces are comparatively rougher with an increased light scattering phenomena and a reduction in the absorption coefficient (Kumar *et al*., 2023). These results correlate with the result from the scanning electron microscope.

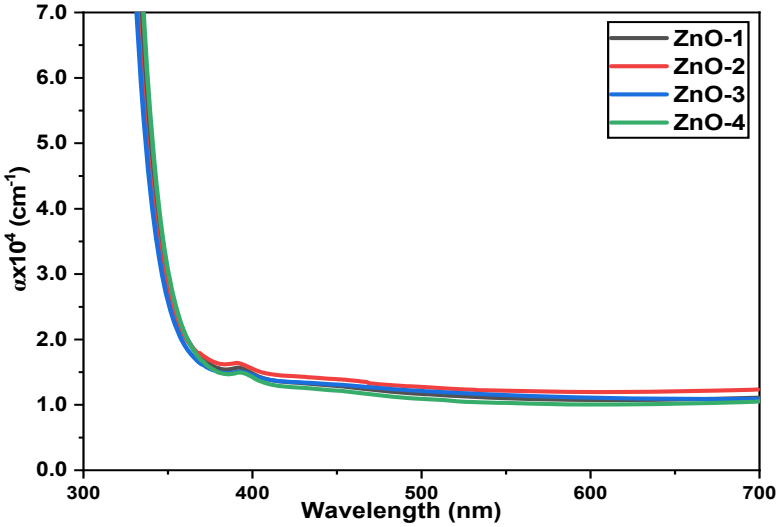

Figure 5: Absorption coefficient spectra for the prepared ZnO thin film samples.

Band gap energy is a very important parameter in determining the possibility of semiconductor materials application in optoelectronic devices. In the determination of thin films' band gap energy (E_g), the Tauc's relation (equation 2) has been commonly adopted (Olasanmi and Mukolu, 2023).

$$(\alpha h\nu)^{\frac{1}{p}} = \boldsymbol{B}(h\nu - E_g) \quad (2)$$

here, B represents the transition probability constant which depend on the effective mass of the material's charge carriers, ν is the photon frequency, h is the Plank's constant and E_g the band gap energy. The parameter p signifies the power factor of the transition mode which depends on the nature of the material (either crystalline or amorphous) and the photon transition. In case of allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions, p takes values as 1/2, 2, 3/2 and 3 respectively. For the ZnO thin films prepared in this study which is a direct transition compound, the value of p is ½ and the band gap energy is estimated by extrapolating the linear portion of the curve of $\alpha h\nu^2$ against $h\nu$ to $\alpha h\nu^2$ = 0, as shown in figure 6. The values of the band gap energy as deduced from the plot have been displayed in table 1.

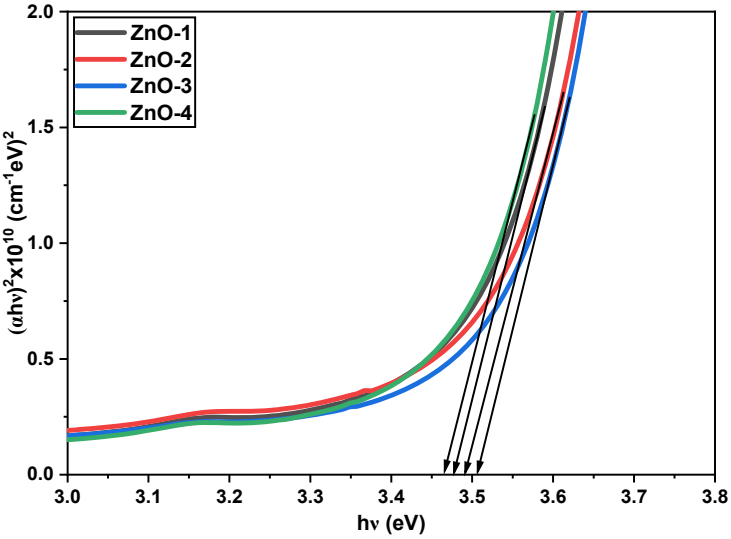

Figure 6: Plot of $(\alpha h\nu)^2$ against $(h\nu)$.

Table 1: Band gap energy and Urbach energy values for the ZnO thin film samples.

Samples	Band gap energy E_g (eV)	Urbach Energy meV
ZnO-1	3.47	176
ZnO-2	3.49	182
ZnO-3	3.51	181
ZnO-4	3.46	173

Table 1 shows that the band gap energy ranges between 3.46 eV and 3.51 eV; a range of band gap energies similar to the values obtained by Purohit *et al*., (2015) for ZnO thin film prepared by radio frequency sputtering method. The reported band gap energy values are slightly greater than the band gap energy of the bulk ZnO (3.37 eV) which may be due to two phenomena (i) axial strain effect from lattice deformation as suggested for ZnO films (Ong *et al.*, 2002), or (ii) change in semiconductor carriers' density (Amakali *et al*, 2020). From the table, it is observed that the band gap energy increases with increasing deposition time; an outcome that has been previously reported by Ghorannevis *et al*., (2016). The same behaviour have earlier been reported for ZnS thin film prepared by chemical bath method, and may be as a result of the decrease in defects (Chabou *et al.*, 2019). The result also confirms that with reduction in deposition temperature, the band gap energy is also affected. From the table, by

comparing the results of the two film samples deposited at the same time but different temperatures, it is clear that the thin film sample deposited at lower deposition temperature exhibits lower band gap energy. Thus, reduction in deposition temperature results in the reduction of the band gap energy value. This range of band gap energy reported in this study has place ZnO as a promising material for transparent conducting oxide in **solar cells**. **(**Chouhan *et al.*, 2021**)**.

The absorption coefficient (α) within the low photon energy range is assumed to be exponentially related to the incident photon energy (hν) and can be expressed by the following equation (Sta *et al.*, 2014);

$$\alpha = \alpha_o e^{h\nu/E_U} \quad (3)$$

where α_o is a constant and E_U refers to the Urbach energy or the width of Urbach tail. Using equation 3, a graph of ln(α) against (hν) was plotted for the deposited ZnO thin film samples and presented in Figure 7. The slope of the linear fits made to the linear part of the plot gives the inverse of the Urbach energy ($1/E_U$) and the values of the Urbach energy is presented in Table 1. It can be seen that increase in deposition time increases the value of Urbach energy; a result that is in agreement with the work of Suryavanshi *et al*., (2018). The observed outcome may be attributed to the elongated time of deposition, which allows for the formation of more defect states and leading to the broadening of the Urbach tail (Suryavanshi *et al*. 2018).

It can also be seen that the reduction in deposition temperature brought about a reduction in the Urbach energy. Similar observation has previously been reported by Ali *et al*. (2016). Defects such as oxygen vacancies, zinc interstitials, and grain boundaries which usually occur at high temperatures are known to promote the broadening of Urbach tail. Hence, a lower deposition temperature results in the reduction of defects formation with an attendant narrowing of the Urbach tail and reduction of the Urbach energy (Jamal *et al*., 2019).

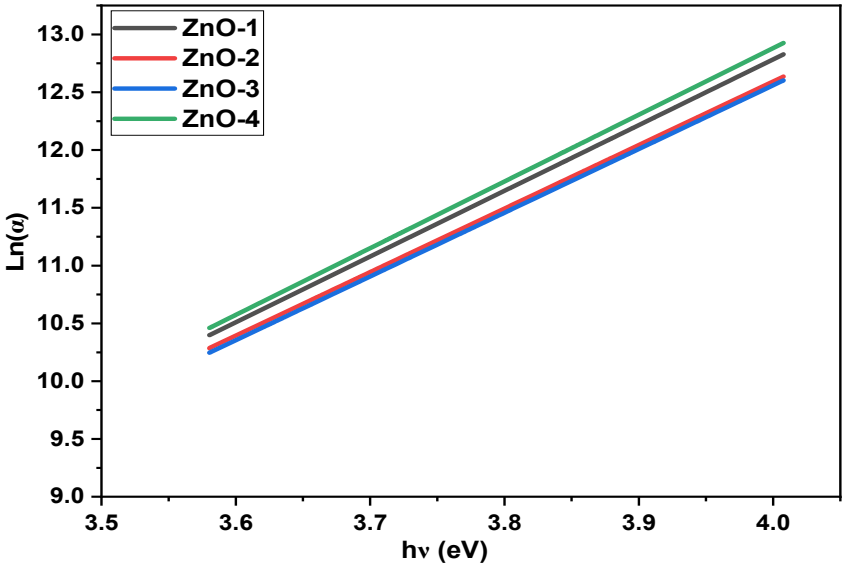

Figure 7: Ubarch energy (E_U) spectra for the prepared thin film samples.

The skin depth (δ) is a parameter that describes the depth in a particular medium at which the radiation intensity reduces to 1/e (about 37%) of its value at the surface (Osanyinlusi *et al.*, 2023). The Skin depth (δ) of the prepared thin film samples has been determined from its inverse relationship with the absorption coefficient (α) as represented by the following simple equation (Sharma *et al*., 2020);

$$\delta = \frac{1}{\alpha} \quad (4)$$

Figure 8 shows the dependence of skin depth on the photon energy for the prepared ZnO thin film samples.

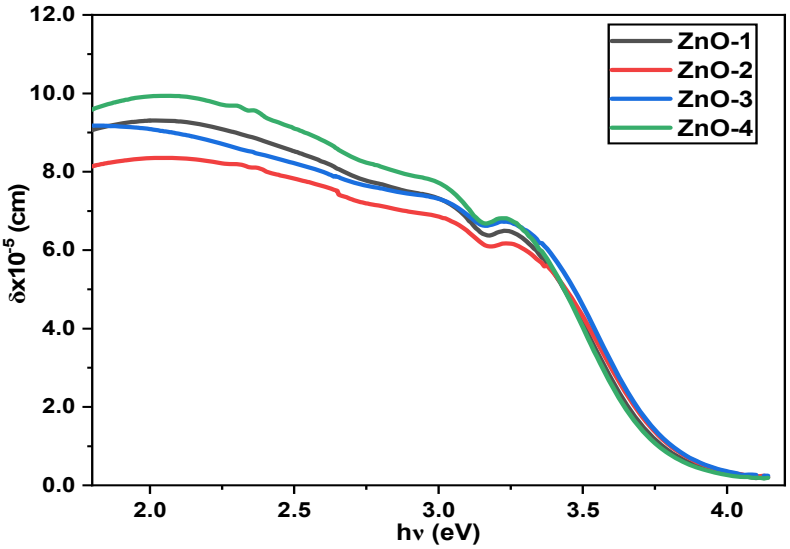

Figure 8: Skin depth spectra for the prepared ZnO thin film samples.

From the graph, it is clear that the skin depth decreases with increasing photon energy up to 4.0 eV where the skin depth value converges to approximately zero. This photon energy value at which skin depth converges to zero is referred to as the cut-off energy ($\mathbf{E_{cut\text{-}off}}$) and the corresponding wavelength is known as the cut-off wavelength ($\boldsymbol{\lambda_{cut\text{-}off}}$). For the prepared ZnO thin film samples, the $E_{cut\text{-}off}$ is 4.0 eV and the corresponding wavelength is 310 nm. It is observed from the Figure that the skin depth reduces with increasing deposition time from 30 to 60 min. The ZnO-2 sample deposited for 45 min exhibited the lowest value of skin depth, which may be as a result of its highest value of absorption coefficient. It is visible from the graph that ZnO-4 thin film sample with lower deposition temperature possess the highest value of skin depth. The occurrence may also be due to the slower growth kinetics of the thin film at reduced temperature, resulting in more porous films with lower density, enabling deeper light penetration into the material, thus increasing the skin depth (Langlet *et al.*, 2005). The **extinction coefficient** (**k**) is an important optical parameter for materials and depicts the degree of light absorption and scattering as it passes through the material. Extinction coefficient is determined from its relation with absorption coefficient (α) and light wavelength (λ) as (Osanyinlusi, 2020).

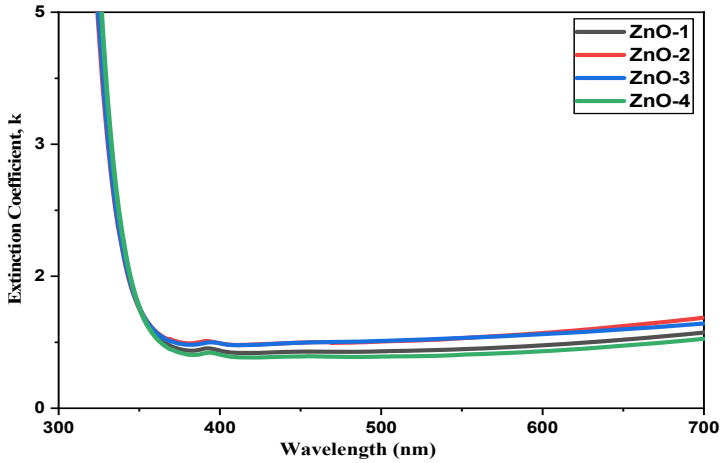

Figure 9: Extinction coefficient against wavelength for the prepared ZnO thin film samples.

$$k = \frac{\alpha\lambda}{4\pi} \quad (5)$$

Figure 9 shows the dependence of extinction coefficient with light wavelength. It was observed from the graph that the value of extinction coefficient increases with increase in deposition time. The Figure reveals that the reduction in deposition temperature also affects the value of the extinction coefficient, with the film deposited at a reduced temperature of 60ºC possessing the lowest value of extinction coefficient.

Conclusion

In this study, the variation of deposition time and temperature on their optical properties has been examined. ZnO thin films have been prepared using the chemical bath deposition technique. EDX image confirmed the presence of zinc and oxygen with their atomic percent very close to the stoichiometric composition. The study reveals that the percentage transmittance reduces as the deposition time increase, with the ZnO-1 sample deposited for 30 min having the highest transmittance value of 82.7% among the films deposited at 70 ºC. Lower deposition temperature also has significant effect on the transmittance values, as the film deposited at lower temperature of 60 ºC possesses higher transmittance when compared with the one prepared at 70ºC. The band gap values of the prepared ZnO films were found to be in the range of 3.46 and 3.51 eV which is slightly above that of the bulk. Increase in deposition time was observed to increase the band gap energy while reduction in deposition temperature decreases the band gap. The outcome of this study has revealed that the prepared ZnO thin films have the potential for application as transparent conductive oxides in solar cells.

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