Effect of Annealing Temperature on Optical and Structural Properties of EDTA Mediated Solution Grown Zinc Selenide Thin Films

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Abstract:

This work reports successful deposition of Zinc Selenide thin films on microscopic glass substrates of dimension (25.4 mm x 76.2 mm x 1.2 mm) using solution growth method in alkaline medium in the presence of EDTA as complexing agent. The bath composed of molar solution of zinc acetate ($Zn(CH_3CO_2)_2 \cdot 2H_2O$) as source of Zn^{2+} ion, freshly refluxed sodium selenosulphate as source of Se^{2+} ion and ammonium hydroxide as pH adjuster. Five samples of ZnSe thin films were fabricated at room temperature (300 K). The four of the deposited samples were heat – treated in an electric oven at temperatures of 373 K, 473 K, 573 K and 673 K respectively. Film thickness obtained by gravimetric method ranged from 435.15 nm to 744.05 nm. Film thickness was found to increase as annealing temperature increases. Optical properties measured with Spectrophotometer showed that the absorbance is high within UV region but decreases as wavelength increases. Transmittance of the films are found to be low within UV region but increases as wavelength increase. Reflectance of the films is low which suggest the usefulness of the deposited film in antireflective coating. Absorbance was found to increase as temperature increases while transmittance to decreases as temperature increases. The energy band gap result obtained ranged between 2.42 eV – 2.94 eV. We observed that the energy band gap decrease as annealing temperature increase. Refractive index ranged between 1.14 and 2.60. The optical results obtained show that ZnSe thin films could be used for solar energy application and optoelectronics devices. Average crystallite sizes obtained using Scherrer's formula are between 7.44 nm and 8.05 nm.

Keywords:

Solution Growth Method, Zinc Selenide, Annealing Temperature, Optical Properties, Structural Properties

1. Introduction

The ability of field of material science and engineering to develop new materials with astonishing combination of chemical, physical and mechanical properties has transformed our present world. The demands for thin film based materials are on the increase because of its enormous industrial applications. Thin film technology is the basic of astounding development in solid state electronics.

The usefulness of the optical properties of metal films, and scientific curiosity about the behavior of two-dimensional solids has been responsible for the immense interest in the study of science and technology of thin films [1]. Thin film studies have directly or indirectly advanced many new areas of research in solid state physics and chemistry which are based on phenomena uniquely characteristic of the thickness, geometry, and structure of the film [2,3]. Thin film materials have already been used in semiconductor devices, wireless communications, telecommunications, integrated circuits, rectifiers, transistors, solar cells, light-emitting diodes, photoconductors, light crystal displays, magneto-optic memories, audio and video systems, compact discs, electro-optic coatings, memories, multilayer capacitors, flat-panel displays, smart windows, computer chips, magneto-optic discs, lithography, micro electromechanical systems (MEMS), and multifunctional emerging coatings, as well as other emerging cutting technologies [1,2,3,4].

In all the thin film materials making advancement in the last few years, Zinc Selenide (ZnSe) thin films have shown great potentials in the field of science and technology due to their properties. ZnSe is n – type semiconducting material belonging to II – VI family of semiconducting materials and has wide band gap of about 2.70 eV at room temperature [5,6]. The major merits of zinc – based materials in comparison to other IIB elements in forming II – VI semiconducting material are that it is non – toxic, readily available and eco – friendly. Zinc selenide crystallizes either in cubic (zinc blende) or hexagonal (wurtzite) structures [5]. Figure 1 shows the crystal structure of the cubic and hexagonal phases of zinc selenide system.

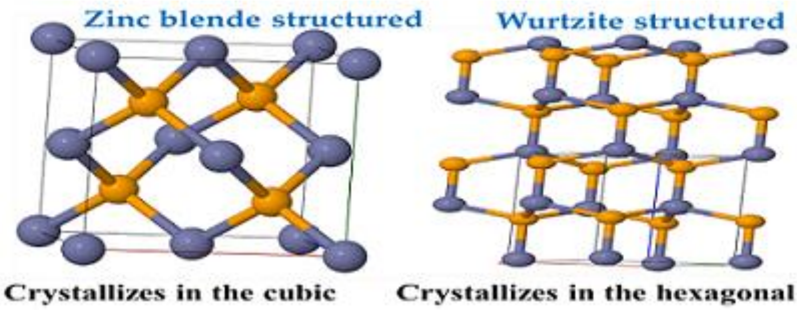

Figure 1. *Crystal structural phases of ZnSe system (VESTA Software).*

Several physical and chemical methods of deposition have been used to deposit ZnSe thin films by various researchers which include; chemical bath method [5,6,7,8], photo – assisted chemical bath techniques [9,10], pulsed laser deposition [11], thermal evaporation [12,13], inert gas condensation [14], electrodeposition [15,16,17], vacuum deposition techniques [18], electron beam evaporation [19].

In this work, we prepared thin films of ZnSe by solution growth techniques at room temperature in the presence of disodium ethylenediamine tetraacetate (EDTA) as a complexing agent. The films obtained were annealed at different temperature so as to determine the effect of annealing temperature on the optical and structural properties of ZnSe thin films.

2. Experimental Details

Zinc selenide was deposited in the reaction of solution containing zinc acetate dihydrate $(Zn(CH_3CO_2)_2).H_2O$, Sodium selenosulphate (Na_2SeSO_3). Disodium ethylenediamine tetraacetate $(Na_4(C_{10}H_{16}N_2O_8))$ commonly known as EDTA, ammonium solution (NH_4OH), and distilled water in a beaker. EDTA as complexing agent slowed down the precipitation of the zinc ions in the mixture. Ammonium solution stabilizes the pH of the mixture. Deposition of five samples of zinc selenide thin films were carried out using 100 ml glass beaker at an average room temperature of 300K. For a sample deposition, 30 ml of zinc acetate dihydrate, was measured, transferred into the beaker, 10 ml of sodium selenosulphate was added to the beaker. The mixture was stirred with a magnetic stirrer for 10 minutes. After that, 15 ml of EDTA was added and stirred for 10 minutes. Followed by addition of 5 ml of ammonium solution. The solution was stirred for 10 minutes, then 20 ml of distilled water was added. The final solution was stirred for 30 minutes using a magnetic stirrer to have a homogenous mixture. Glass substrate was inserted vertically into the beaker with the help of a synthetic foam. Five beakers were prepared in the same way. The substrates were allowed to stay in the bath for constant time of 24 hours and temperature of about 300 K was maintained throughout the deposition period. At the end of 24 hours, the substrates were removed, rinsed with distilled water and dried in open air at room temperature of (300 K). Four of the grown films were annealed at temperatures of 373 K, 473 K, 573 K and 673 K respectively.

Table 1. *Optimization of Zinc Selenide (ZnSe) at Room Temperature.*

Temp. (K)	$Zn(CH_3CO_2)_2$		Na_2SeSO_3	NH_4OH	EDTA		H_20	Dip Time (hrs)
	Vol (ml)	mol	Vol (ml)	Vol (ml)	Vol (ml)	Mol	Vol (ml)	
300	30.00	0.4	10.00	5.00	15.00	0.05	20	24
373	30.00	0.4	10.00	5.00	15.00	0.05	20	24
473	30.00	0.4	10.00	5.00	15.00	0.05	20	24
573	30.00	0.4	10.00	5.00	15.00	0.05	20	24
673	30.00	0.4	10.00	5.00	15.00	0.05	20	24

3. Results and Discussions

3.1. Thickness Measurement

Thicknesses (t) of the deposited thin films were evaluated using the gravimetric method given by [20,21,22] equation (3.1)

$$t = \frac{\Delta m}{\rho A}$$

where Δm is the mass gain of the film. A is the surface area of the deposited film and ρ is the bulk density of the material film. The mass gains of the deposited films were obtained by finding the difference in mass between the mass of the glass substrate and the film after deposited and the mass of glass substrate before deposition. Bulk density of 5.27 g/cm^3 for ZnSe was used in evaluating film thicknesses. Figure 2 shows the plot of thickness of the films plotted against annealing temperature. From the figure, the thickness of the films increases as annealing temperature increases. This is due to the increase in the crystallinity of the ZnSe at high temperature. This result is in line with results obtained by [23,24]. Peak thickness 744.05 nm was

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obtained for film annealed at 673 K while the least thickness of 435.15 nm was observed for the as – grown film.

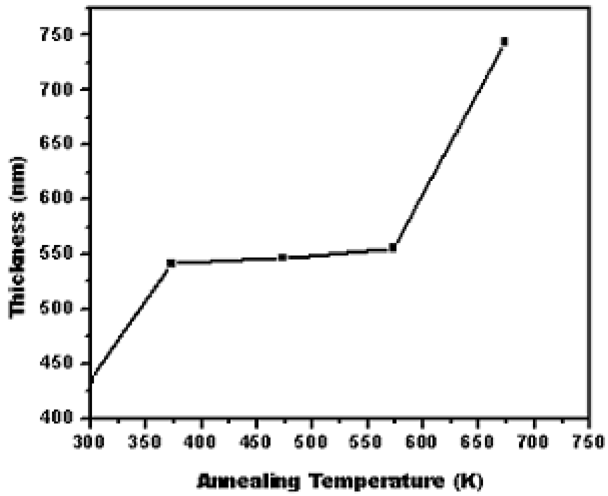

Figure 2. *Plot of thickness (nm) against annealing temperature for Zinc selenide thin films.*

3.2. Optical Analysis

Optical characterization of the films was carried out using 752W UV – VIS – NIR Grating spectrophotometer at wavelength interval of 300 nm to 1100 nm. Absorbance values of the films were obtained using the spectrophotometer within the wavelength range of 300 nm and 1100 nm. Other optical properties such as transmittance, reflectance, refractive index, extinction coefficient and energy band gap were evaluated using the following equations as obtained from literatures.

Transmittance of the film was evaluated using equation (3.2) given by [25,26].

$$T = 10^{-A}$$

Reflectance was obtained using the expression in equation (3.3) as given by [27,28].

$$R = 1 - (A + T)$$

Refractive index of the films was calculated using equation (3.4) as given by [29,30].

$$\eta = \frac{(1 + \sqrt{R})}{(1 - \sqrt{R})}$$

The absorption coefficient (α) was calculated from the transmittance and thickness values using the equation (3.5) as given by [31,32,33].

$$\alpha = \frac{1}{t} In\left(\frac{1}{T}\right)$$

Extinction coefficient was obtained using equation (3.6) as given by [34,35].

$$k = \frac{\alpha\,\lambda}{4\pi}$$

The energy band gap was estimated using Tauc’s model given in equation (3.7) as given by [36,37].

$$(\alpha hv)^n = \beta\left(hv - E_g\right)$$

Where β is a constant, $n = 2$ for direct band gap. The energy band gaps of the films were obtained by extrapolating the straight portion of the plot of $(\alpha hv)^2$ against the photon energy (hv) at $(\alpha hv)^2 = 0$.

Figure 3(a) shows the graph of absorbance of the films plotted against wavelength. From the graph, the absorbance of ZnSe thin films decreases as wavelength increase and decreases slightly along the NIR region. Also, the graph shows an increase in absorbance as annealing temperature increases. The as – grown thin film of ZnSe has absorbance that ranges from a peak value of 71.4 % at 300 nm to minimal value of 17.93 % at 1100 nm. ZnSe film annealed at 373K has absorbance peak of 91.48 % at 300 nm and minimal value of 19.64 % at 1100 nm. ZnSe film annealed at 473K has absorbance peak of 87.10 % at 300 nm and minimal value of 21.05 % at 1100 nm. ZnSe film annealed at 573K has absorbance peak of 85.65 % at 300 nm and minimal value of 21.48 % at 1100 nm. ZnSe film annealed at 673K has absorbance peak of 99.55 % at 300 nm and minimal value of 29.15% at 1100 nm.

Figure 3(b) shows the graph of transmittance of the films plotted against wavelength. From the graph, the transmittance of ZnSe thin films increases as wavelength increase. Also, the graph shows a decrease in transmittance as annealing temperature increases. The as – grown thin film of ZnSe has transmittance ranging from a peak value of 66.19 % at 1100 nm to minimal value of 19.34 % at 300 nm. ZnSe film annealed at 373K has transmittance peak of 63.62 % at 1100 nm and minimal value of 13.30 at 300 nm. ZnSe film annealed at 473K has transmittance peak of 61.59 % at 1100 nm and minimal value of 13.45 % at 300 nm. ZnSe film annealed at 573K has transmittance peak of 60.98 % at 1100 nm and minimal value of 13.91 % at 300 nm. ZnSe film annealed at 673K has transmittance peak of 51.12 % at 1100 nm and minimal value of 10.03 % at 300 nm.

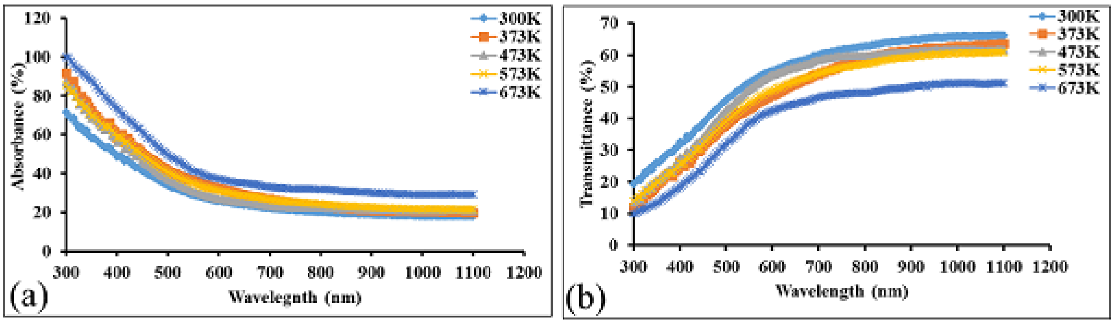

Figure 3. *Plot of (a) Absorbance, (b) Transmittance against wavelength for Zinc Selenide thin films at different annealing temperature.*

Figure 4(a) shows the graph of reflectance of the films plotted against wavelength. From the graph, the reflectance of ZnSe thin films increases as wavelength increase along the UV and some parts of VIS up to 500 nm. Beyond 500 nm, the reflectance of the films slightly decreases. Also, the graph shows a decrease in reflectance as annealing temperature increases within UV and some parts of VIS up to a wavelength of 500 nm. Beyond this wavelength, the reflectance of the films increases as annealing temperature increases. The as – grown thin film of ZnSe has reflectance ranging from a peak value of 15.89 % at 1100 nm to minimal value of 9.28 % at 300 nm. ZnSe film annealed at 373K has reflectance peak of 16.74 % at 1100 nm and minimal value of 2.50 % at 320 nm. ZnSe film annealed at 473K has reflectance peak of 17.36 % at 1100 nm and minimal value of 1.14 % at 305 nm. ZnSe film annealed at 573 K has reflectance peak of 17.54 % at1100 nm and minimal value of 0.41 % at 300 nm. ZnSe film annealed at 673 K has reflectance peak of 19.74 % at 1100 nm and minimal value of 1.08 % at 360 nm.

Figure 4(b) shows the graph of refractive index of the films plotted against wavelength. From the graph, the refractive index of ZnSe thin films increases as wavelength increase along the UV and some parts of VIS up to 500 nm. Beyond 500 nm, the refractive index of the films slightly decreases. Also, the graph shows a decrease in refractive index as annealing temperature increases within UV and some parts of VIS up to a wavelength of 500 nm. Beyond this wavelength, the refractive index of the films increases as annealing temperature increases. The as – grown thin film of ZnSe has refractive index ranging from a peak value of 2.32 at 1100 nm to minimal value of 1.88 at 300 nm. ZnSe film annealed at 373 K has refractive index peak of 2.38 at 1100 nm and minimal value of 1.38 at 320 nm. ZnSe film annealed at 473K has refractive index peak of 2.43 at 1100 nm and minimal value of 1.24 at 305 nm. ZnSe film annealed at 573K has refractive index peak of 2.44 at 1100 nm and minimal value of 1.14 at 300 nm. ZnSe film annealed at 673K has refractive index peak of 2.60 at 1100 nm and minimal value of 1.23 at 355 nm.

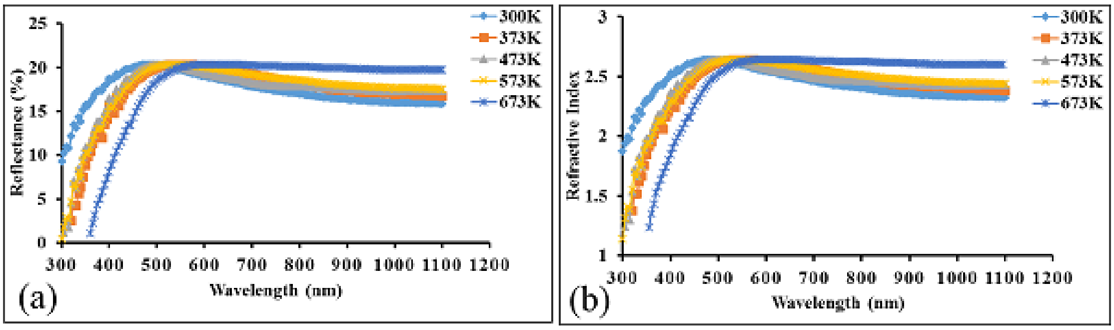

Figure 4. *Plot of (a) Reflectance (%) and (b) refractive index against wavelength for Zinc Selenide thin films at different annealing temperature.*

Figure 5(a) shows the graph of extinction coefficient of the films plotted against wavelength. From the graph, the extinction coefficient of ZnSe thin films decreases as wavelength increase and remained almost the same along the NIR region. Also, the graph shows an increase in extinction coefficient as annealing temperature increases. The as – grown thin film of ZnSe has extinction coefficient that ranges from a peak value 5.68×10^{-2} at 300 nm to minimal value of 1.42×10^{-2} at 1100 nm. ZnSe film annealed at 373K has extinction coefficient peak of 7.28×10^{-2} at 300 nm and minimal value of 1.56×10^{-2} at 1100 nm. ZnSe film annealed at 473K has extinction coefficient peak of 6.93×10^{-2} at 300 nm and minimal value of 1.68×10^{-2} at 1100 nm. ZnSe film annealed at 573K has extinction coefficient peak of 6.82×10^{-2} at 300 nm and minimal value of 1.71×10^{-2} at 1100 nm. ZnSe film annealed at 673K has extinction coefficient peak of 7.95×10^{-2} at 300 nm and minimal value of 2.32×10^{-2} at 1100 nm.

Figure 5(b) shows the plotted graphs of $(\alpha h\upsilon)^2$ against photon energy $(h\upsilon)$ for the different annealing temperature. The direct band gap energy was extrapolated at the axis of the photon energy $(h\upsilon)$ where $(\alpha h\upsilon)^2 = 0$. The as – grown ZnSe film has band gap energy of 2.94 eV. ZnSe film annealed at 373 K has band gap of 2.86 eV. ZnSe film annealed at 473 K has band gap of 2.75 eV. ZnSe film annealed at 573 K has band gap of 2.55 eV and ZnSe film annealed at 673 K has an energy band gap of 2.42 eV. This show that the energy band gap of ZnSe thin films deposited decreases as temperature increases. The values of energy band gap obtained correspond to values obtained by other researchers given in Table 2 below. According to [24], materials with these ranges of energy band gap are good for light emitting diodes (LEDs), photosensor, reflecting dielectric mirrors and other visible luminescent devices. [31] suggested that ZnSe films of energy band gap between 2.40 eV and 2.80 eV are good material for solar cell buffer layer. Figure 6 shows the variation of energy band gap with annealing temperature. The energy band gap is found to decrease as temperature increases. This is because of increase in the interatomic spacing as a result of increase in the amplitude of the atomic vibrations caused by thermal energy. This increase in interatomic spacing causes a decrease in the potential seen by the electrons with the ZnSe films which in turn makes the energy band gap to reduce. This is similar to results obtained by [38,39].

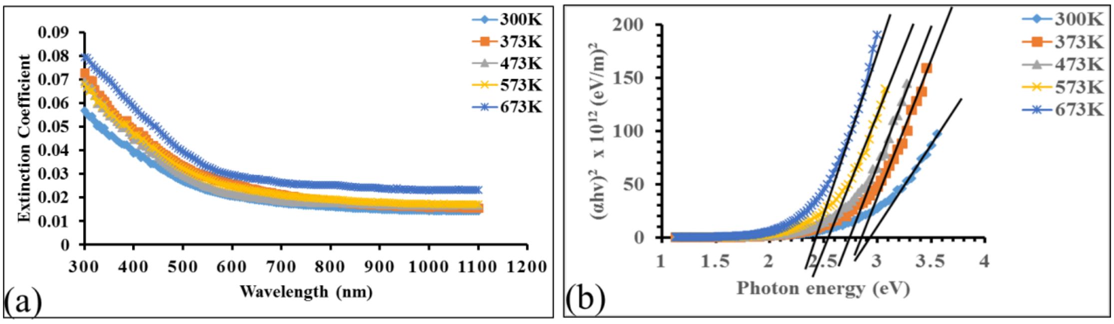

Figure 5. *Plot of (a) extinction coefficient and (b) (αhv)2 against photon energy against wavelength for Zinc Selenide thin films at different annealing temperature.*

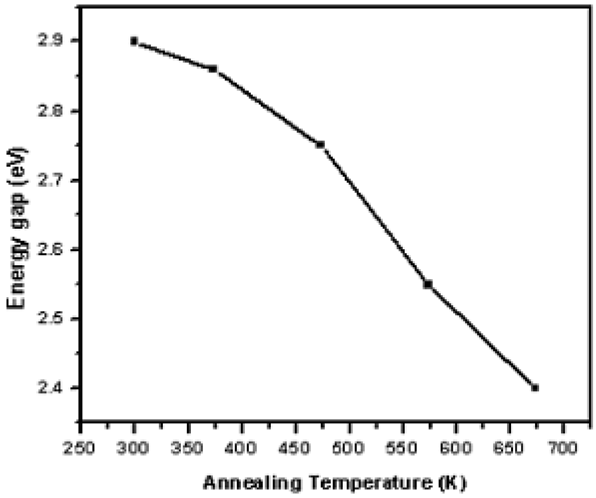

Figure 6. *Plot of Energy band gap against Temperature.*

Table 2. *Corresponding energy band gap from other researchers.*

Researchers	**Band gap (eV)**
Our result	**2.42 – 2.94**
[40]	2.12 – 2.49
[41]	2.82
[42]	2.70 ± 0.02
[23]	2.02 – 2.48
[31]	2.80
[43]	2.65
[44]	2.79 – 2.40
[24]	2.95 – 3.30

3.3. X – ray Diffraction Analysis

Figure 7 shows the x – ray diffractograms of as – grown and annealed ZnSe thin film deposited obtained using Cu – Kα radiation with wavelength ($\lambda = 1.5406Å$). The diffractograms obtained show that films crystallizes in the cubic phase of ZnSe with JCPDS file number 37 - 1463. The broadening of the diffraction peaks revealed that the film is polycrystalline in nature. For as – grown ZnSe and ZnSe annealed at 473K, the peaks obtained were 26.56 °, 31.25 °, 44.71 °, 53.41 ° which correspond to the miller indices of (111), (200), (222) and (311). Additional peak of 64.76 ° corresponding to (400) was observed in the diffractogram of ZnSe thin film annealed at 473 K. Crystallite size of the film was obtained using Scherrer‘s formula given in equation (9) by [45,46.47].

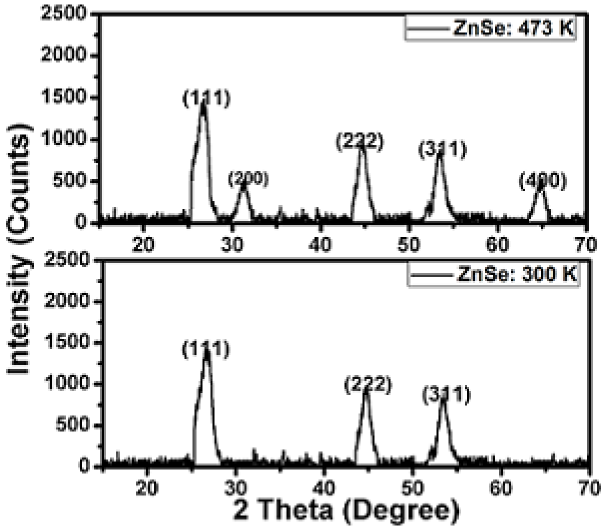

Figure 7. *Diffractograms of as – grown (300 K) and annealed (473 K) ZnSe thin films.*

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

where D is the crystallite size, is the wavelength of x – ray radiation (Cu), is the full width at half maximum intensity of peaks in radians and θ is the Bragg diffraction angle. The crystallite size of the films were evaluated using Scherrer’s formula as given by (48,49) The crystallite size of the films is between 7.44 nm – 8.05 nm. The slight increase in the crystallite size and appearance of additional peak showed that increase in annealing temperature increases the crystalline nature of the films. This result is in line with x – ray pattern obtained by [50,51].

4. Conclusion

We have successfully deposited thin film of ZnSe thin films using chemical deposition approach. Five thin films were deposited using the same bath parameters such as concentration of the solution, pH, temperature of the baths, and time of depositions. After deposition, four of the films were annealed at four different temperatures of 100 $°C$ (373K), 200 $°C$ (473K), 300 $°C$ (573K), and 400 $°C$ (673K) respectively. The as – grown and the four annealed thin films were subjected to optical analysis using spectrophotometer and structural analysis using diffractometer. Absorbance values of the films were obtained at wavelength range of 300 nm to 1100 nm. Other optical properties were evaluated using existing formulations outlined in the literature review. The results obtained show that annealing temperature affects the optical properties of ZnSe thin films.

The optical properties show that ZnSe thin films deposited have high optical absorption within ultraviolet (UV) region and visible light (VIS) region and low absorption within the near infrared (NIR) region, also absorbance is found to increase as annealing temperature increase. The transmittance of the films are low within UV and VIS region but increases to its maximum value within NIR region. Transmittance of the films decreases as annealing temperature increases. The reflectance of the films are generally low for all the films which suggest the possibility of using the films for anti – reflective coating to shed of UV radiations. The refractive index values of the films are low within UV and VIS region but increases to peak values within NIR region. This result shows that infrared radiation travels faster in the ZnSe thin film samples more that UV and VIS component of electromagnetic radiation. The extinction coefficient of the films decreases as wavelength increase but increases as annealing temperature increases. Energy band gap obtained is between $2.42\ eV$ and $2.94\ eV$. The energy band gap decreases as annealing temperature increases. Structural analysis of the films confirmed that ZnSe is in nanocrystalline form with crystallite size ranging between 7.44 nm – 8.05 nm.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this article.

Author Contributions

Conceptualization: E.N.J.; O.N.L.; Methodology: E.N.J.; O.N.L.; Software: E.N.J.; O.N.L.; Validation: E.N.J.; O.N.L.; Formal analysis: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Investigation: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Resources: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Data Curation: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Writing – original draft preparation: O.N.L.; Writing – review and editing: E.N.J.; O.N.L.; N.I. E.; O.I.O..; Supervision: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Project administration: E.N.J.; O.N.L.; N.I.E.; O.I.O.; Funding acquisition: E.N.J.; O.N.L.; N.I.E.; O.I.O.

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