



Williamson-hall analysis and absorption spectrum fitting in the estimation of crystallite size and band gap energy of CdZnS thin films

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ABSTRACT

CdZnS ternary thin film was successfully prepared from cadmium sulphide (CdS)/zinc sulphide (ZnS) bilayer and its subsequent post thermal annealing via chemical bath technique. X-ray diffraction measurement indicates that the samples presented a crystalline wurtzite phase with (002) plane as the preferred orientation. Williamson-Hall (W-H) analysis was employed to estimate the crystallite size and the microstrain. The result showed that the crystallite size decreases with increasing ZnS layer deposition times. Using the Tauc model, the absorption spectrum fitting (ASF) procedure was used to determine the optical band gap energy. The values of the band gap energy range between 3.61 and 3.66 eV, with the values increasing with ZnS layer deposition times.

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1. INTRODUCTION

Ternary compounds such as CdZnS which belong to the II–VI semiconductor group have attracted a lot of attention in many applications as a result of their tunable physical properties, like the lattice parameter and band gap. By substituting CdS with CdZnS which has a wider band gap as window layer in solar cells, decrease in window absorption losses which leads to an increase in the short circuit current and open circuit voltage has been addressed [1].

In order to enhance the performance of CdZnS thin films, numerous interests have been deployed into investigating the properties of these thin films prepared by different methods. CdZnS thin film has been prepared by different techniques such as; spray pyrolysis [2], simple ultrasonic radiation method [3], solution growth method [4], vacuum evaporation [5] and chemical bath deposition [6]. In this study, CdZnS thin film has been grown

through post thermal annealing of bilayer CdS/ZnS via chemical bath route.

XRD structure analysis is largely accepted because of its accurate assessment of the crystallographic structural parameters like; crystallite size, micro-strain, and dislocation density [7]. Increase in the diffraction peak width and intensity as well as the shifting in the peak position has been associated with the crystallite size and lattice strain. Crystallite size is a measure of the size of a coherently diffracting domain while Lattice strain is referred to the degree of distribution of lattice constants resulting from crystal imperfections [8]. A perfect crystal would extend in all directions to infinity, and as a result, no crystal is perfect because of their finite size [9]. The result of this deviation from perfect crystallinity is the widening of the diffraction peaks. In order to determine the variations in the crystallite size and the microstrain in the measured XRD pattern for the prepared film samples, Williamson–Hall method has been adopted. Williamson-Hall (W-H) analysis is a simplified integral breadth

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method where both size-induced and strain-induced broadening is deconvoluted by considering the peak width as a function of 2θ [9].

Study of an optical materials requires deeper knowledge of their optical band gap energy and hence the optical susceptibility. The band gap energy determination is somewhat complicated due to the valence and conduction bands surface atoms edges of the valence which are not sharp and the tail states complicate the definition of the true optical gap [10, 11]. The most commonly used procedure for estimating band gap energy of materials involves evaluating the optical absorption coefficient and applying the Tauc method. This method has been widely used for different binary and ternary semiconducting materials [12]. In determining the absorption coefficient, measurement of the film's absorbance, the reflectance and film thickness are necessary [13]. Another technique for estimating the band gap energy known as absorption spectrum fitting (ASF) has been proposed. In this procedure, band gap energy can be estimated with the measurement of film's absorbance only, and without information about the film's thickness and reflectance.

The present work therefore is carried out to accomplish two objectives; the first is to estimate the crystallite size and lattice strain of CdZnS using W-H analysis and the second is to determine the optical band gap of CdZnS thin film using ASF procedure.

2. EXPERIMENTAL DETAILS

The commercially available glass slide of dimension 25.4 mm x 76.2 mm x 1.2 mm was used as substrate in this study. Prior to use, the substrates were cleaned in an ultrasonic bath using methanol, acetone and distilled water, after which they were dried in air. 50 ml Cd $(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (0.2 M) and 50 ml $\text{CS}(\text{NH}_2)_2$ (0.5 M) as well as 50 ml ZnSO_4 (0.2 M) and 50 ml $\text{CS}(\text{NH}_2)_2$ (0.5 M) were used in the preparation of the CdS and ZnS baths respectively. The complexing agent used is ammonia (NH_3) and the preparation pH been 10.5. The detailed thin film preparation procedures were earlier published somewhere [14]. Thermal annealing of the films was done at 673 K for 1 h using Muffle furnace (model SXL) and they were labeled as CdS/ZnS-10, CdS/ZnS-15 and CdS/ZnS-20, for films with ZnS layer deposition times of 10, 15 and 20 min respectively. ZnS thicknesses were chosen in order to have a minima overall thickness of the alloy so as not to compromise its photon transmission capacity. The x-ray diffraction pattern was detected using PANalytical multipurpose (X'Pert-Pro) utilizing Cu α radiation ($\lambda = 0.15406$ nm). The optical transmittance was carried out using Avantes-SAI-07086751 model UV-Vis spectrophotometer.

3. RESULTS AND DISCUSSION

3.1. PHASE IDENTIFICATION

Figure 1 presents the X-ray diffraction pattern for the deposited film samples. All the thin films exhibit peaks that closely resembles the (100), (002), (101), (110) and (200) reflections of the hexagonal CdZnS structure which corresponds to the JCPDS-card No.49-1302 data [15]. The X-ray spectral revealed a preferred orientation at the (002) plane for all the films with no observed structural transformation as the ZnS deposited time increases. The detailed discussion of the X-ray spectra as well as

the structural parameters obtained from (110) and (002) planes only has been previously published elsewhere [14].

3.2. NELSON-RILEY PLOT ANALYSIS

Nelson-Riley plot is a very effective process used to estimate the corrected values of lattice constants. The corrected lattice constants were determined in order to ensure accurate characterization of material crystal structure, as it take cognizance of factors like temperature effects, strain, and defective samples. These corrections assist in obtaining inherent lattice parameters that are not affected by external influences and offer uniformity across various measurement techniques and conditions. The Nelson-Riley plot signifies a plot between the calculated lattice constants 'a' and 'c' for all the various planes and their estimated error functions [16, 17].

The lattice constants 'a' and 'c' for each of the peaks were calculated using Equations (1) and (2) respectively, while their error functions were estimated from Equation (3) [17–19]. Figures 2 and 3 shows the Nelson-Riley plots for the lattice constants 'a' and 'c', and their estimate corrected lattice constants are presented in Table 1.

$$a = \frac{\lambda}{2\sin\theta} \sqrt{\frac{4}{3}(h^2 + hk + k^2) + \left(\frac{a}{c}\right)^2 l^2}. \quad (1)$$

$$c = \frac{\lambda}{2\sin\theta} \sqrt{\frac{4}{3}\left(\frac{c}{a}\right)^2 (h^2 + hk + k^2) + l^2}. \quad (2)$$

$$f(\theta) = \frac{1}{2} \left(\frac{\cos^2\theta}{\sin\theta} + \frac{\cos^2\theta}{\theta} \right). \quad (3)$$

The values of the corrected lattice constants are very close to that of the bulk CdZnS (0.4136 nm and 0.6689 for 'a' and 'c' respectively). Furthermore, the lattice constant values are observed to decrease with increasing ZnS deposition times and this can be attributed to the small ionic radius of Zn (0.74 Å) which when introduced into the Cd structure (0.94 Å) causes shrinkage in the lattice constant [20].

3.3. WILLIAMSON HALL (W-H) ANALYSIS

In X-ray diffraction analysis, broadening of peaks is due to both the crystallite size and the presence of non-uniform strain in the material [19]. The strain induced (β_s) and size induced (β_D) peak broadening are related by Equations (4) and (5) respectively [21].

$$\beta_s = 4\epsilon \tan\theta. \quad (4)$$

$$\beta_D = \frac{k\lambda}{D_{hkl} \times \cos\theta}, \quad (5)$$

where k is a constant with a value of 0.94, ϵ is the microstrain, θ is the diffraction angle. D_{hkl} is the crystallite size of a particular crystal plane (hkl), λ is the wavelength of incident X-ray and for CuK α radiation = 0.15406 nm.

It can be inferred from these equations that β_s vary linearly with $\tan\theta$ and β_D varies inversely with $\cos\theta$. Assuming that the crystallite size and microstrain contributions to the peaks broadening are independent of each other and both have a Cauchy-like profile, Equations (4) and (5) can be brought together resulting

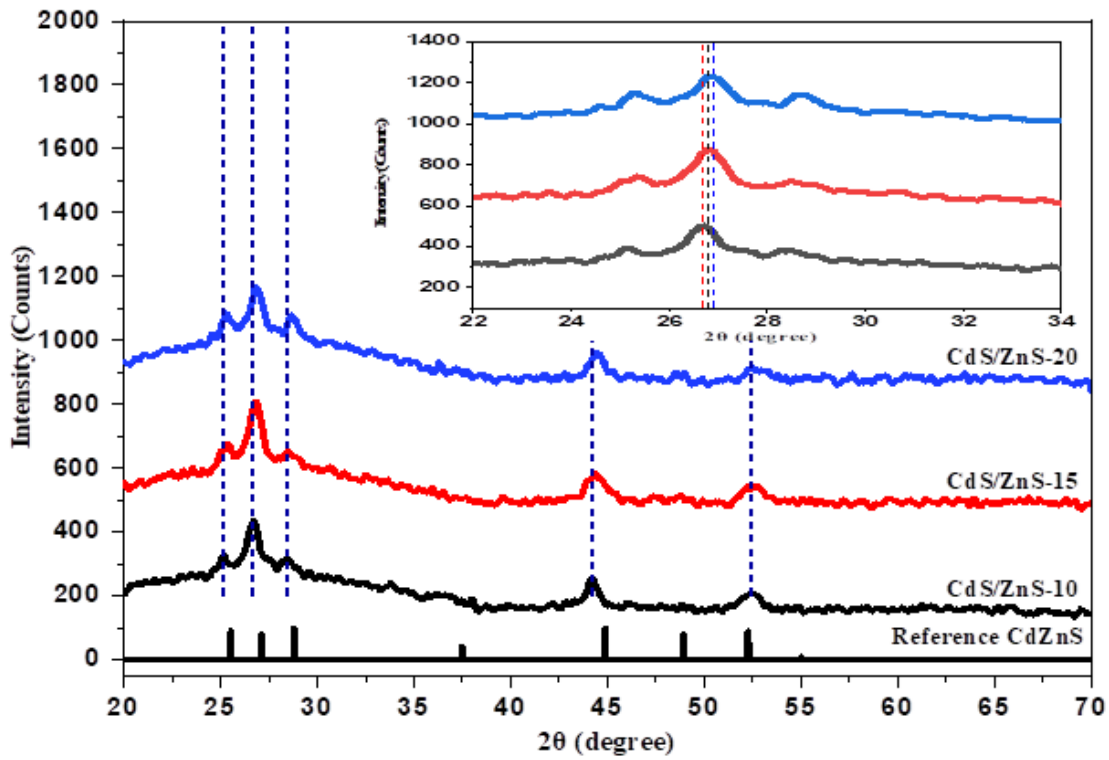


Figure 1. XRD (X-ray Diffraction) spectral for the prepared CdS/ZnS-10, CdS/ZnS-15 and CdS/ZnS -20 samples [14].

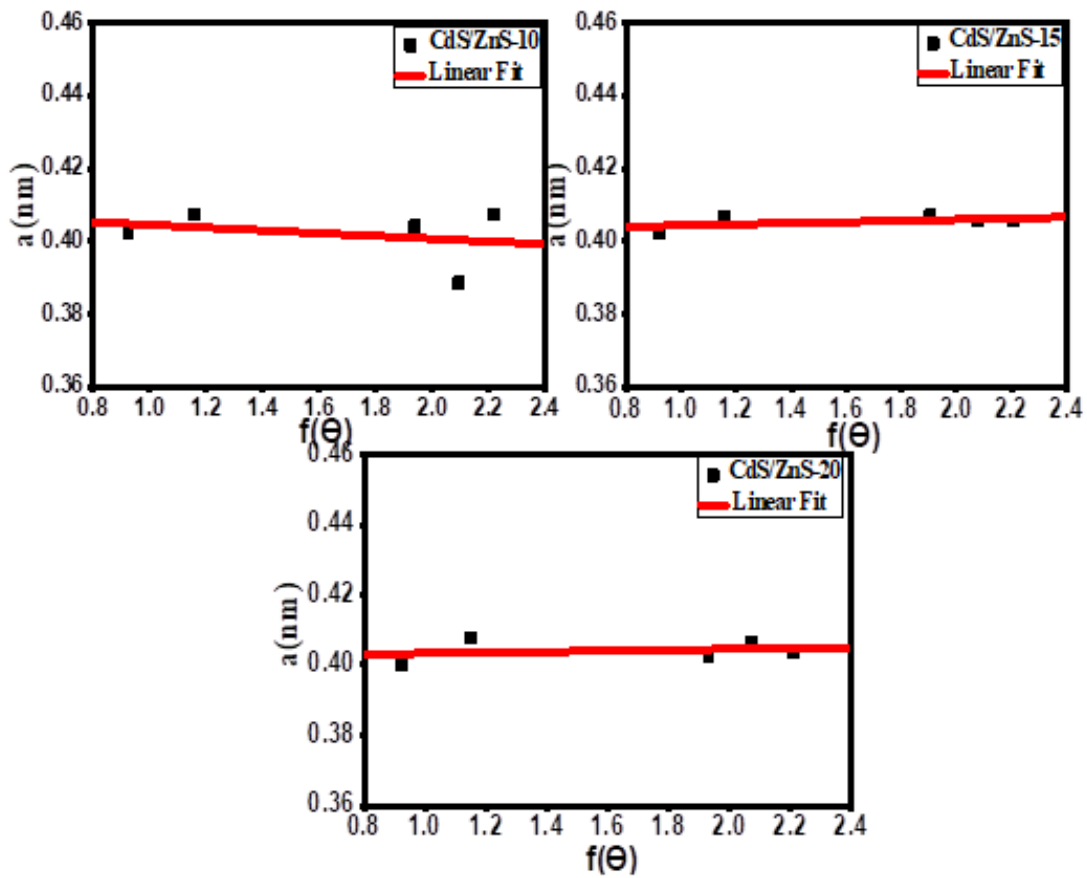


Figure 2. A Nelson – Riley plot for the corrected values ‘a’.

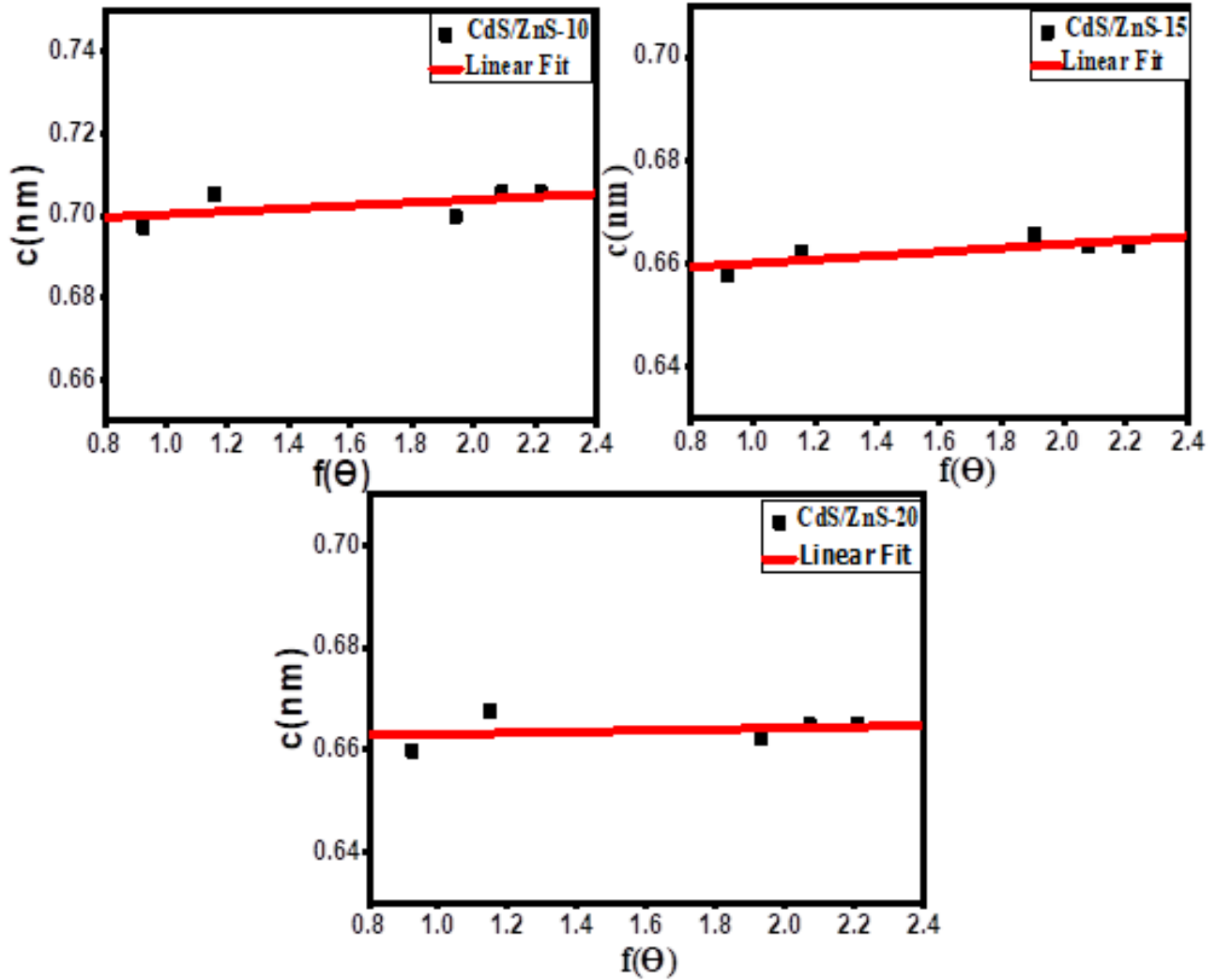


Figure 3. A Nelson–Riley plot for the corrected values ‘c’.

Table 1. Structural parameters of the CdS/ZnS -10, CdS/ZnS -15 and CdS/ZnS -20 thin films showing corrected lattice constant.

Samples	<i>hkl</i>	2θ (deg.)	$D(\text{\AA})$	$a(\text{nm})$	$a(\text{corrected})$ (nm)	$c(\text{\AA})$	$c(\text{corrected})$ (nm)	$F(\theta)$
CdS/ZnS-10	100	25.21	3.5284	0.4074	0.4083	0.7057	0.6968	2.2189
	002	26.66	3.3410	0.3886		0.7057		2.0936
	101	28.50	3.1294	0.404		0.6998		1.9408
	110	44.20	2.0475	0.4072		0.7053		1.1568
	112	52.47	1.7422	0.4025		0.6972		0.9252
CdS/ZnS-15	100	25.34	3.5120	0.4055	0.4027	0.6633	0.6566	2.2079
	002	26.79	3.3239	0.4055		0.6633		2.0765
	101	28.94	3.0828	0.4069		0.6656		1.9031
	110	44.40	2.0387	0.4067		0.6624		1.1538
	112	52.71	1.7349	0.4022		0.6579		0.9194
CdS/ZnS-20	100	25.34	3.5120	0.4037	0.4021	0.6648	0.6619	2.2079
	002	26.86	3.3166	0.4065		0.6648		2.0716
	101	28.63	3.1144	0.4028		0.6624		1.9309
	110	44.51	2.0335	0.4077		0.6675		1.1498
	112	52.50	1.7416	0.3999		0.6598		0.9249

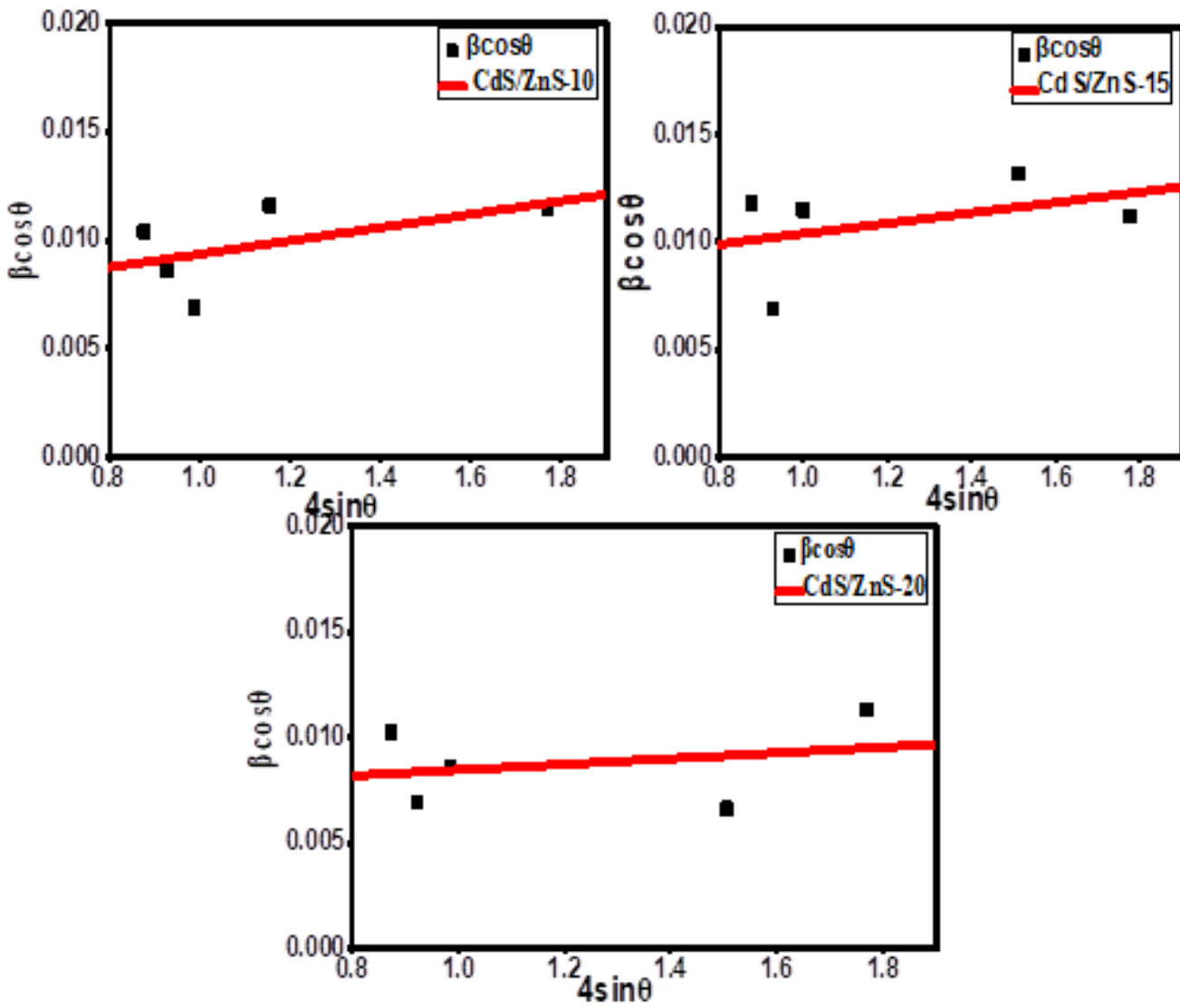


Figure 4. Williamson-Hall plot ($\beta, \cos\theta$ against $4\sin\theta$) of synthesized samples.

Table 2. Crystallite Size, Micro Strain and Energy gap for the prepared CdZnS samples.

Sample	Crystallite Size D (nm)	Micro Strain $\epsilon \times 10^{-3}$	Energy gap E_g (eV)
CdS/ZnS-10	19.24	7.55	3.61
CdS/ZnS-15	19.23	7.50	3.63
CdS/ZnS-20	15.37	9.35	3.66

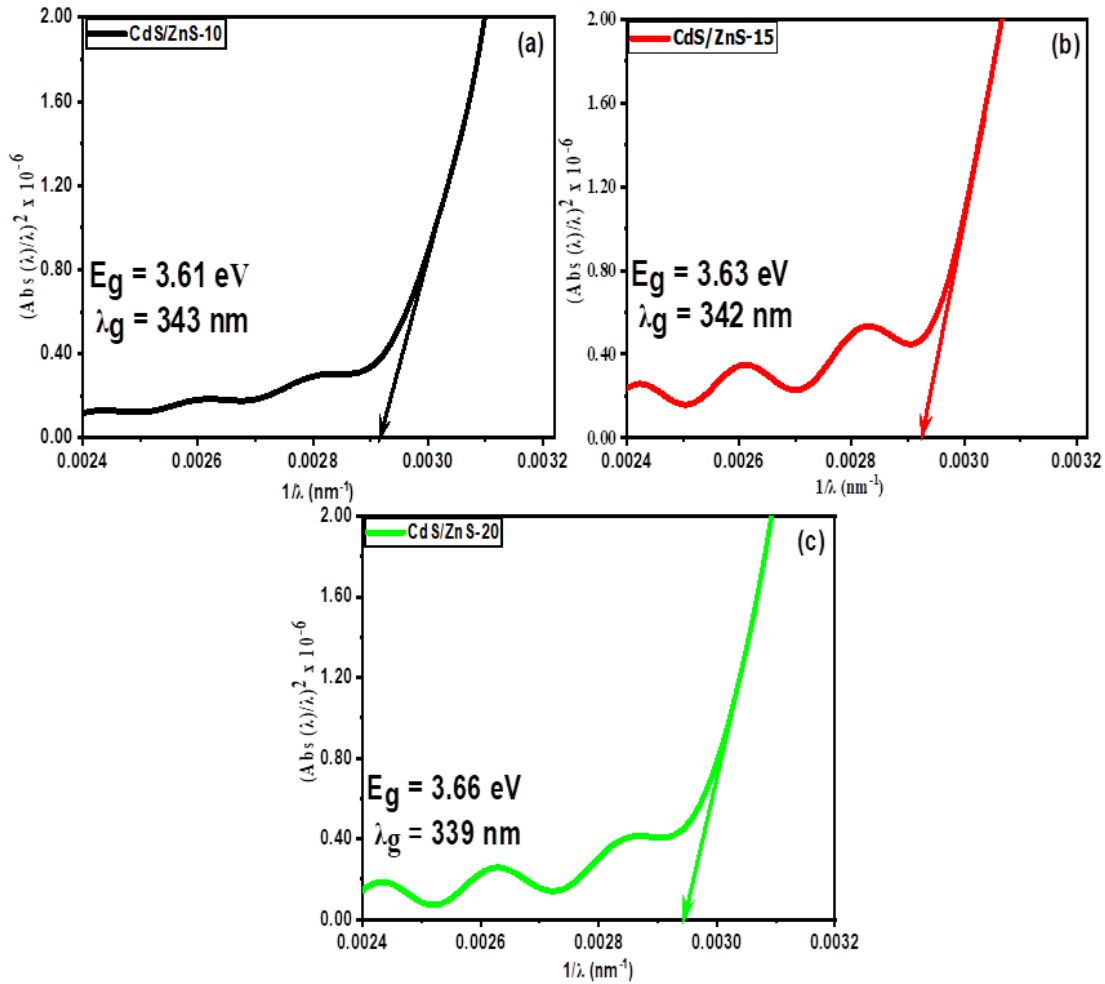


Figure 5. Absorption spectrum fitting Plots for the prepared CdZnS thin film samples.

in what is known as the Williamson and Hall (W-H) equations [21].

$$\beta = \frac{k\lambda}{D\cos\theta} + 4\epsilon \tan\theta. \quad (6)$$

Rearranging Equation (6), we have

$$\beta\cos\theta = \frac{k\lambda}{D} + 4\epsilon\sin\theta. \quad (7)$$

A linear fitting applied to a graph of $\beta\cos\theta$ against $4\epsilon\sin\theta$ (Figure 4) gives a slope corresponding to the value of the microstrain and an intercept which gives the value crystallite size. The micro strain values as determined from the plot are presented in Table 2. From the result it is observed that the crystallite size increase while the microstrain decreases with increasing ZnS deposition times. Similar reduction in crystallite size with an attendant increase in microstrain has previously been reported by Zakria *et al.* [22] for CdZnS thin films prepared by Close Space Sublimation process (CSS), using CdS and ZnS as the starting materials.

3.4. ABSORPTION SPECTRUM FITTING (ASF)

In absorption spectrum fitting, only the measurement of film's absorbance without the need for additional measurement like

film's thickness and reflectance are considered. Absorption spectrum fitting (ASF) involves the transformation of the Tauc's equation to an analogous equation having a function of wavelength using $\nu = \frac{c}{\lambda}$. The method have previously been employed by Ghobadi *et al* [23] to estimate the band gap energy of CdSe thin films prepared by chemical bath method. Recently, Bhogi *et al* [24] used the method to determine the band gap energy of $\text{Li}_2\text{O-SrO-B}_2\text{O}_3\text{-MnO}$ quaternary glass system.

$$\alpha h\nu = B(h\nu - E_g)^p. \quad (8)$$

$$\frac{\alpha hc}{\lambda} = B\left(\frac{hc}{\lambda} - \frac{hc}{\lambda_{gap}}\right)^p, \quad (9)$$

where h , c and λ_{gap} are the planks constant, velocity of light and the wavelength corresponding to optical gap value respectively, p is the power factor of the transition mode and B is the proportionality coefficient which depend on the probability of optical transitions.

$$\frac{\alpha(\lambda)}{\lambda} = B(hc)^{x-1} \times \left(\frac{1}{\lambda} - \frac{1}{\lambda_{gap}}\right)^p. \quad (10)$$

By using the Beer-Lambert's law given as; $\alpha(\lambda) = \frac{2.303 \text{ Abs}(\lambda)}{t}$ where t and α are thickness and absorp-

tion respectively, it is possible to rewrite Equation 4 as follows:

$$\frac{\text{Abs}(\lambda)}{\lambda} = \left[\frac{Bt(hc)^{p-1}}{2.303} \right] \times \left(\frac{1}{\lambda} - \frac{1}{\lambda_{gap}} \right)^p \quad (11)$$

$$\frac{\text{Abs}(\lambda)}{\lambda} = K \times \left(\frac{1}{\lambda} - \frac{1}{\lambda_{gap}} \right)^p + K_2, \quad (12)$$

where $K = Bt(hc)^{p-1}/2.303$ and K_2 is a constant that takes into account the reflection. In the case of allowed direct transition, $p = 1/2$. Equation (7) can therefore be rewritten in this form. Thus, a plot of $(\text{Abs}(\lambda)/\lambda)^2$ against $1/\lambda$ is presented in Figure 5. The values of λ_{gap} can be deduced by extrapolating the linear portion of the curve at $1/\lambda = 0$. The values of the λ_{gap} as determined from the plot signify the wavelength which corresponds to the direct band gap energy. Thus, the band gap energy (in electron volt) for the prepared thin film samples can be calculated using $E_g = 1239.83/\lambda_{gap}$ [25] and are presented in Table 2. It can be observed that the value of the band gap energy of the ternary thin film compound increase with increasing ZnS deposition times. Widening of CdZnS band gap energy with increasing ZnS deposition time can be attributed to the introduction of more ZnS compound with higher band gap energy into the structure as its deposition time increase. This assertion confirms that the band gap of CdZnS ternary compound thin films can be tuned to a desired value with the adjustment of the ZnS layer deposition times and subsequently, the thickness.

4. CONCLUSION

Ternary CdZnS thin film semiconductor was prepared by post-thermal annealing of CdS/ZnS bilayer with increasing ZnS layer deposition times using chemical bath deposition technique. The structural properties of the prepared thin films were investigated using the x-ray diffraction analysis and the result revealed a hexagonal structure with the crystalline size decreasing as ZnS deposition time increase. The lattice constant was determined using the Nelson-Riley plot analysis and the result showed that the lattice constant decreased with increasing ZnS layer deposition times. The optical energy gap was determined using the absorption spectrum fitting procedure (ASF). The values of the band gap energy ranges between 3.61 and 3.66 eV, and it is observed to increase with increasing ZnS deposition time. The outcome of this study suggested that CdZnS compound thin film can find usefulness as buffer layer in photovoltaic cells.

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