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Optical characterization of chemical bath deposition $Cd_{1-x}Zn_xS$ thin films

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Abstract

$Cd_{1-x}Zn_xS$ thin films prepared by a chemical bath deposition (CBD) method. Cadmium acetate, zinc acetate and thiourea used as a cadmium, zinc and sulfur ions in solution, respectively. Five types of samples were synthesized onto glass substrates with different Cd^{2+} and Zn^{2+} sources molarities in alkaline solution (pH=11) and at a temperature 80 ^oC.

The films' thickness was in the range (102-120) nm and most film's thickness decreased while Zn concentration in solution increased. The transmittance of films increased as Zn concentration increased. All films, however, have high transmittance of 65-85% in the wavelength region (500-900) nm, which is high enough for solar cell applications. The energy band gaps of $Cd_{1-x}Zn_xS$ films calculated and their values were (2.24-3.7) eV. The values of the width of the tails of localized states in the forbidden gap of the films obtained from Urbach plots and varied in the range (0.025-0.11) eV. The optical constants such as, extinction coefficient, refractive index, real and imaginary parts of the dielectric constant are found. The dispersion energy E_d and a single-oscillator energy E_o measured from the single oscillator model below the absorption edge are used to calculate the moments of the optical spectra M_{-1} and M_{-3}.

Keywords: $Cd_{1-x}Zn_xS$ thin films; Chemical bath deposition; Structure composition; Optical energy gap.
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1. Introduction

II-VI semiconductors thin films have attracted considerable attention from the research community because of their widely uses in the fabrication of solar cells and other opto-electronic devices [1, 2].

Cadmium sulfide (CdS) is widely used to fabricate solar cells, but because its value of the band gap, the CdS window layer absorbs the blue portion of the solar spectrum. Cadmium sulfide (CdS) and zinc sulfide (ZnS) form a continuous series of solid solution

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$Cd_{1-x}Zn_xS$ for the whole range of (x) percent. Thus, the energy gap of $Cd_{1-x}Zn_xS$ can be controlled in the range of each binary phase. CdZnS compound has a band gap between CdS (2.42eV) and ZnS(3.66eV) and the value of the band gap depends onto Cd and Zn ratio[3].

The replacement of CdS with the higher energy band gap of $Cd_{1-x}Zn_xS$ alloys has led to a decrease in window absorption losses and has resulted in an increase in the short-circuit current in the solar cell. In addition to CdZnS compound, it is also useful to fabricate p-n junctions without lattice mismatch using quaternary materials such as $CuInGaSe_2$ and $CuInS_2Se_2$[4]. Many others applications of $Cd_{1-x}Zn_xS$ ($Cd_xZn_{1-x}S$) on the nano-dimension have been demonstrated by Kumer and Sankaranarayanan [5].

There are many techniques used to synthesize CdZnS thin films such as co-evaporation, electrodeposition, successive ionic layer adsorption and reaction (SILAR), metalorganig chemical vapor deposition (MOCVD),vacuum evaporation, spray pyrolysis and chemical bath deposition (CBD)[6-12].

Of all of these methods , CBD is preferred due to it is relatively ease , not expensive and suitable technique for the deposition of a high quality, nanostructures and large area films[13].While CdS is less soluble than ZnS, $Cd(OH)_2$ is more soluble than $Zn(OH)_2$ (see Table 1) so ZnS is more difficult to deposit than CdS . Due to the different stability constants of the Cd and Zn precursors, the formation process of CdZnS thin films become more difficult so some researchers have resorted to use of multilayer CdS and ZnS processing [14].

The alkaline deposition of $Cd_{1-x}Zn_xS$ thin films is reported by taking different Cd and Zn sources molarities. Optical transmittance spectra of samples with different ions source molarities have been studied and the values of energy gaps obtained. The Cd and Zn ratios in films has been found from these values and by using Vegard's expression.

Table 1: Values of solubility product at room temperature (R.T) °C [14]

Solid	ksp
CdS	10^{-28}
$Cd(OH)_2$	$2x10^{-14}$
ZnS	$10^{-23.8}$
$Zn(OH)_2$	10^{-16}

2. Mechanism of $Cd_{1-x}Zn_xS$ thin films formation

The reaction procedure for the synthesis $Cd_{1-x}Zn_xS$ thin films may be described by the following steps [15].

Ammonium formation is given by:

$$NH_3 + H_2O \leftrightarrow NH_4^+ + OH^- \quad (1)$$

The method which used to prepare cadmium sulfide (CdS) includes the reaction of cadmium ions (Cd^{2+}) with sulfide ions(S^{-2}). Sulfide ions was found in solution from thiourea as shown in the equations 2,3 below:

$$(NH_2)_2CS + OH^- \leftrightarrow SH^- + CN_2H_2 + H_2O \tag{2}$$

$$SH^- + OH^- \leftrightarrow S^{2-} + H_2O \tag{3}$$

Dissolving cadmium acetate and zinc acetate in water made both cadmium and zinc ions are available in solution indirectly by the formation of complexes of those ions as shown in the equations below:

$$Cd(OH)_2 + 4NH_2OH \rightarrow \left[Cd(NH_3)_4\right]^{2+} \tag{4}$$

$$Cd(OH)_2 + 4NH_2OH \rightarrow \left[Cd(NH_3)_4\right]^{2+} \tag{5}$$

$$Zn^{2+} + 2OH^- \leftrightarrow Zn(OH)_2 \downarrow \tag{6}$$

$$Zn(OH)_2 + 4NH_2OH \rightarrow \left[Zn(NH_3)_4\right]^{2+} \tag{7}$$

$Cd(NH_3)_4]^{2+}$ and $[Zn(NH_3)_4]^{2+}$ are called cadmium and zinc tetra-amine complex ions, respectively .

Each three of these source ions (eqs. 3,5,7) are combined in the reaction mixture leading to the formation of CdZnS as shown in the equation below:

$$(1-x)\left[Cd(NH_3)_4^{2+}\right] + x\left[Zn(NH_3)_4^{2+}\right] + S^{2-} + NH_3 \xrightarrow{PH=11} Cd_{1-x}Zn_xS + wast \tag{8}$$

3. Experimental

In CBD technique, the ions source materials molarities, PH, deposition temperature and time of deposition are greatly affecting on the film's formation. Furthermore, cleaning of the substrates is very important to the initial growth of the grains and then on film's adherent onto the substrate.

Thin films of CdZnS were deposited from the solution by using high purity start materials(Sigma-Aldrich) cadmium acetate $[Cd(CH_3(COO)_2]$ as a Cd^{2+} ions source, zinc acetate$[Zn(CH_3(COO)_2]$ as a Zn^{2+} ions source, thiourea $[SC(NH_2)_2]$ as a S^{-2} ions source in an alkaline solution of ammonia $[NH_3]$ and ammonium acetate $[NH_4(CH_3\ COO)]$ as a buffer solution.

Commercial glass slides, *used* as substrates, were cleaned in HCl, acetone and methanol ultrasonically, and finally, washed again with acetone ultrasonically and hot water before used. After the completion of the cleaning, the glass slides are fixed vertically in the beaker. 50 ml of distilled water put in a beaker and then cadmium and zinc acetate of particular different molarities added (Table2) as a Cd^{2+} and Zn^{2+} ions source slowly under magnetic stirring. NH_3 solution added slowly to the solution for adjusting the pH value 11, which measured by pH meter. DI water added to raise the volume to 100 ml and 5 ml of (0.04M) and then drops of thiourea slowly added to the solution. Finally, the temperature fixed at 80 ^{o}C. The time for the deposition was 100 minutes after achieving constant temperature for all depositions. After that, the samples were taken out and washed by distilled water ultrasonically to remove the loosely adhered particles on the surface of films and finally dried in the air.

Films' thickness varied in the range (102-120) nm which measured by thickness measurement type Filmetric F20 (USA). Room temperature optical transmittance of thin films was carried out using UV-Visible double beam spectrophotometer (type Thermo spectronic) in the wavelength range (300-800) nm. All the parameters of the prepared samples are listed in Table 2.

Table 2: Thin films preparation parameters

Samples	**Molarities mol in 5 ml**			**Films thickness (nm)**
	Cadmium acetate	**Zinc acetate**	**Thiourea**	
S_1	0.04	0.0	0.04	120
S_2	0.04	0.02	0.04	116
S_3	0.04	0.03	0.04	117
S_4	0.02	0.04	0.04	113
S_5	0.00	0.04	0.04	102

4. Results and discussion

All prepared $Cd_{1-x}Zn_xS$ thin films were of good adhesion, smooth, pinhole free, bright yellow orange and the yellowness increases with increasing zinc concentration.

From Table 2, it can be noted that for the three top samples, the films thickness is decreased with increasing zinc acetate molarities and at a fixed ratio of cadmium molarities. Due the low Zn $(OH)_2$ solubility in water, the Zn concentration in thin films will be less, so to prepare ZnS films by CBD method, zinc ions concentration must be enough in the solution and the preparation time must be more. Ubale et.al.[16] noted that the thickness of ZnS thin films which prepared by CBD was affecting by the time of deposition and the pH value . The solubility of $Cd(OH)_2$ in water is higher than $Zn(OH)_2$, thus Cd^{2+} ions concentration are more than Zn^{2+} ions which lead to increase Cd^{2+} coupling with S^{2-} and produce thin films. Due to the different in solubility values between $Cd(OH)_2$ and $Zn(OH)_2$, the thickness of CdS thin films (S_1) are higher than ZnS thin films (S_5) as the same ions source concentration.

The optical transmittance spectra of $Cd_{1-x}Zn_xS$ thin films recorded in the wavelength range 300–800 nm. The wavelength dependence of optical transmittance of the investigated films deposited at different Cd and Zn source molarities is shown in Figure 1.The film's optical transmittances of 65-85% in the 600- 800 nm range which is high enough for solar cell applications. A shift in the optical absorption edge to low wavelengths (blue shift) is apparent among the transmission curves of the five samples. When the Zn^{2+} concentration is increased and Cd^{2+} concentration decreased in the solution, the films became more transparent in wavelength longer than 500nm and the curves shifted towards low wavelength.

From the absorbance data, the absorption coefficient $\boldsymbol{\alpha}$ (ω) at angular frequency of radiation(ω) calculated by using the relationship [17]:

$$\alpha(\omega) = \frac{\ln(1/T)}{d} \tag{9}$$

Where (d) is the film's thickness and T is the optical transmittance.

The energy gap(Eg) calculated from the relationship [18]:

$$\alpha(\omega) = B\frac{(h\nu - E_g)^n}{h\nu} \tag{10}$$

where hυ (or ħω) is the photon energy, B is a constant and n is the power factor and assumes values 0.5, 2, 1.5 and 3 for allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions, respectively.

A plot of $(\alpha h\nu)^2$ vs hν for five samples of $Cd_{1-x}Zn_xS$ thin films prepared at room temperature (RT) is shown in figure 2. From the graph, the values of the direct band gap energy are evaluated. Extrapolating the straight-line portions of the plot $(\alpha h\nu)^2$ vs hν for zero absorption coefficient value give the energy band gap value. The linear nature of the plots at the absorption edge confirmed that $Cd_{1-x}Zn_xS$ is a semiconductor with a direct band gap.

It can also be seen from figure 2 that the energy gap for the films was equal 2.4 eV for zero Zn concentration in solution i.e., the film was CdS and it is increased by increasing Zn concentration in solution – increase in zinc acetate molarities, that means increasing Zn ratio in thin film and at zero Cd concentration, E_g becomes 3.7 eV, i.e. the film became ZnS (Table 3).

The optical band gap for $Cd_xZn_{x-1}S$ thin films deposited by spray pyrolysis technique[1] is varied from 2.41 to 3.32 eV whereas others[2] obtained by using eqn.(10) the value of E_g = 2.445 and 3.75 eV while their values varied between 2.486 and 3.87 eV using the theoretical fit with Elliott- Toyozawa theory. So it is an interesting correlation that the results from both theories of the $Cd_{1-x}Zn_xS$ thin films are in good agreement.

The coefficient B in eqn. (10) may be obtained from the $(\text{slope})^r$ of the linear region of the plot $(\alpha\hbar\omega)^{1/r}$ vs. ħω and its units can be calculated according to cm-1 eV^{1-r}. Thus, the values of B for $Cd_{1-x}Zn_xS$ thin films in figure (2) are calculated and listed in Table 3. They are of the order of 10^9 $cm^{-1}eV^{1/2}$. For amorphous semiconductors such as GeTe and As_2Se_3 where r =2, the value of B is of the order of 10^5 cm^{-1} eV^{-1}. Semiconductors such as Si and Ge which have r =3, their B's value becomes of the order of 10^6 cm^{-1} eV^{-2} while other materials are reported with different values of r [19].

For many amorphous and crystalline semiconductors an exponential dependence of $\alpha(\omega) < 10^4\ cm^{-1}$ may take the Urbach relation[20]:

$$\alpha(\omega) = \alpha_o \exp(\hbar\omega/\Delta E_t), \tag{11}$$

Where α_o is a constant and ΔE_t is an energy characterizing the degree of disorder introduced from defects and grain boundaries and hence it is interpreted as the width of the tails of localized states in the forbidden gap. Figure (3) represents ln α vs ħω in accordance with equation (11) for five samples of $Cd_{1-x}Zn_xS$ thin films. The value of ΔE_t is calculated from the slope of the linear part of the curves and also listed in Table 3.The increase in the values of the energy gap is due to the increase in the concentration of zinc and is accompanied by an increase in the values of ΔE_t except the value 0.04 eV of sample 5, where thin film becomes more crystalline and this is clear from the behavior of transmittance curve (figure4). The values of ΔE_t for ZnS films [21] varied in the range (0.27-0.46) meV versus the substrate temperatures (250-400) °C.

The composition of the ternary $Cd_{1-x}Zn_xS$ estimated from Vegard's expression [22]:

$$E_g(x) = E_g(0) + 0.69x + 0.62x^2 \tag{12}$$

Where $E_g(0)$ is the band gap at x=0. $E_g(0)$ (CdS)= 2.42 eV .

The variation of the energy gaps as a function to **Zn** ratio is shown in figure 4 and the values are summarized in Table 3. Chavhan et.al.[12] synthesized $Cd_{1-x}Zn_xS$ thin films using CBD method with **Zn** ratio in the range ($0.0 \leq x \leq 0.15$). They changed the quantities of the source solution (Cd & Zn ions source) and their results well agreed with the present work. Lee et.al.[23] also used CBD method to form $Cd_{1-x}Zn_xS$ thin films and they got the optical band gap equal to 2.4,2.55, 2.7 eV for Zn content(x) of 0,30 and 50% respectively. Others [2] found that Eg increases in a quadratic manner with Zn content. However, the E_g tunability of the CdZnS films for various Zn content is clearly demonstrated in our results.

Other researchers [10,24-25] have prepared this compound by thermal evaporation technique and obtained high thin film's transmittances and the band gap values limited by Zn concentration. For vacuum evaporated Cd_xZn_{1-x} S films, the value of E_g varied in the range (3.25, 3.01, 2.82, 2.62) eV for x (0.2, 0.4, 0.6, 0.8) respectively[24]. Akyuz and coworkers [26] produced $Cd_x Zn_{1-x}$ S thin films by ultra sonic spary pyrolysis with x values 0.0-1.0 and they noted that the optical gap varied from 3.513eV for ZnS to 2.486eV for CdS. For the same values of x ratio, Baykul and Orhan [2] obtained values of the optical gap in the range 3.75-2.44 eV.

Semiconductors thin films which prepared by CBD method usually have nanocrystalline structure then the energy levels are confined to potential wells of small dimension, the spacing between the energy levels increases as the crystal becomes smaller [27], so the band gap for ZnS appears more than for bulk.

Parts of the dielectric constant(E_r, E_i) have been found by using the relationships [28, 29].

More information about the structural properties can be useful to determine the effect of the molar concentration on the particles size and shapes. X-ray diffraction (XRD) method and scanning electron microscope (SEM) measurements can give these structural properties. In addition, energy dispersive X-ray test can find the elements ratio in the thin films.)

The extinction coefficient k, the reflectance R, refractive index n, the real and imaginary

$$k = \frac{\alpha\lambda}{4\pi} \tag{13}$$

$$R = \frac{(1+R)}{(1-R)} + \left[\frac{4R}{(1-R)^2} - k^2\right]^{1/2} \tag{14}$$

$$n = \frac{1+R^{1/2}}{1-R^{1/2}} \tag{15}$$

$$E_r = n^2 - k^2 \tag{16}$$

$$E_i = 2nk \tag{17}$$

The extinction coefficient and the reflectance of thin films at different ions sources molarities plotted as a function of wavelength in figures (5&6), respectively. In figure 6 it is clearly noted that the reflectance of thin films degrease as Zn concentration increased in visible region of the spectrum. Nadeem and Ahmed[30] reported the reflectance of thermal evaporated ZnS thin film (100) nm thickness less than 20% at wavelength 400-800 nm which agree with our results for ZnS thin films (x=1,sample 5) at the same region of the wavelength. The reflectance behavior in figure 6 matched well with Isah et.al results [14].

The refractive index of $Cd_{1-x}Zn_xS$ thin films is plotted in figure 7 as a function of the wavelength and it is decreased in the visible region (400-800) nm as Zn concentration increasing in the films. Kumar et.al [31] used the envelope method to calculate the values of the refractive index for thermally evaporated $Cd_xZn_{1-x}S$ thin films with x=0,0.2 and 0.4 and they found values in the wavelength range (500-850nm) to be (2.437-2.394), (2.44-2.388) and (2.464-2.336), respectively. The real and imaginary parts of the dielectric constant of $Cd_{1-x}Zn_xS$ thin films different Zn concentration as a function to wavelength are illustrated in figure 8.The figure revealed that the values of real part are higher than the imaginary part for all samples and these values decreased as Zn concentration increased in the films.

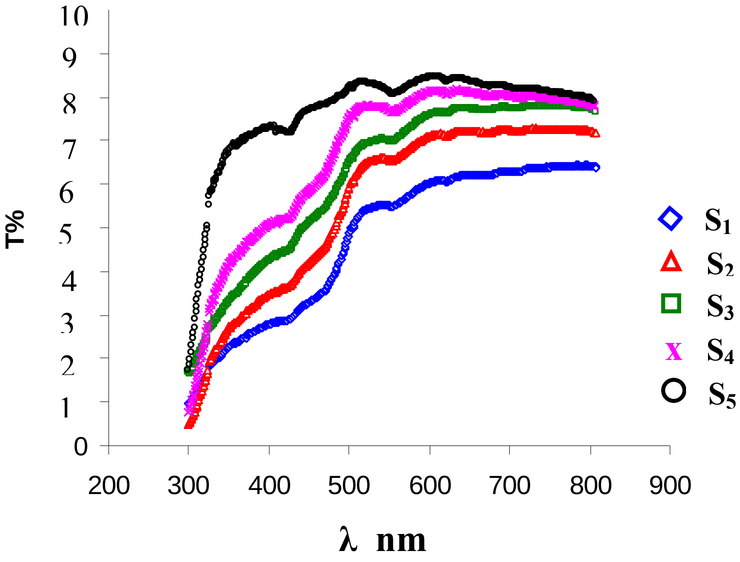

Fig.1: Transmission spectra of $Cd_{1-x}Zn_xS$ prepared thin films

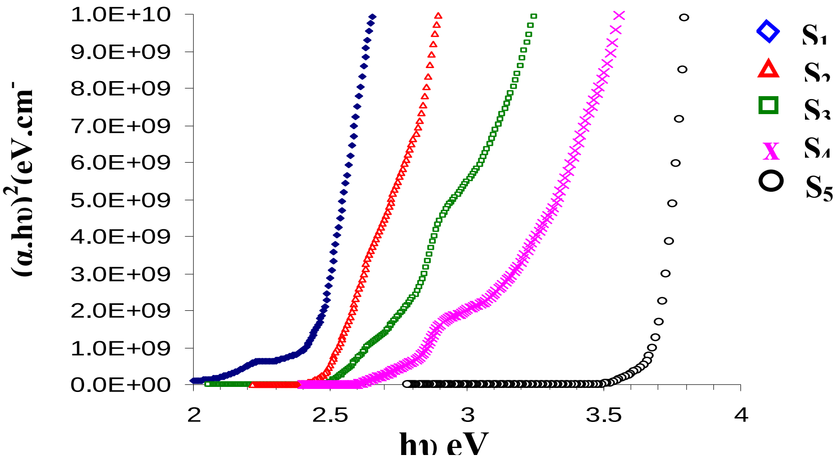

Fig. 2: Plot of $(\alpha\ h\nu)^2$ vs of $Cd_{1-x}Zn_xS$ prepared thin films

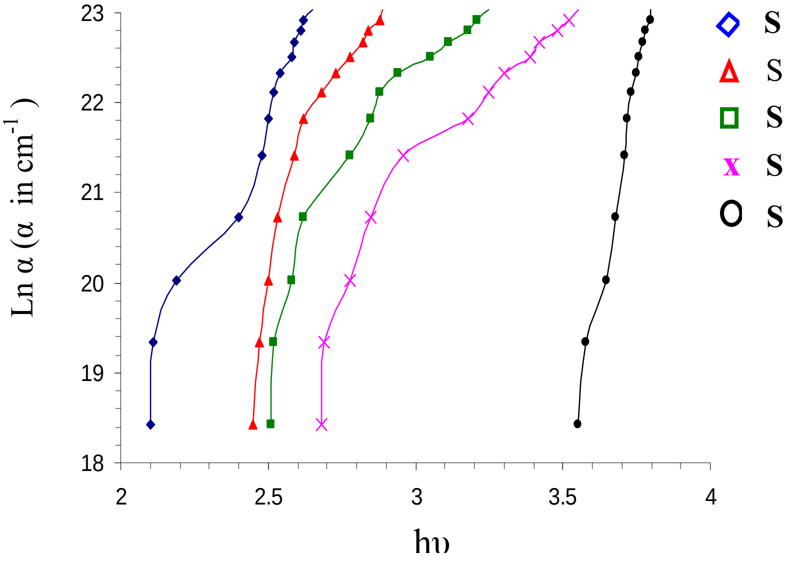

Fig. 3: The Urbach exponential edges of $Cd_{1-x}Zn_xS$ prepared thin films

Table 3: The values of Zn ratio, E_g & ΔE_t and B for the prepared $Cd_{1-x}Zn_xS$ thin film

Samples	**X**	**Compound**	**E_g (eV)**	**ΔE_t (eV)**	**B($cm^{-1}eV^{1/2}$) x10^{10}**
S_1	0.00	CdS	2.42	0.025	4.0
S_2	0.13	$Cd_{0.87}Zn_{0.13}S$	2.50	0.035	2.1
S_3	0.33	$Cd_{0.66}Zn_{0.33}S$	2.70	0.055	1.8
S_4	0.71	$Cd_{0.29}Zn_{0.71}S$	3.20	0.110	2.2
S_5	1.00	ZnS	3.70	0.040	10.0

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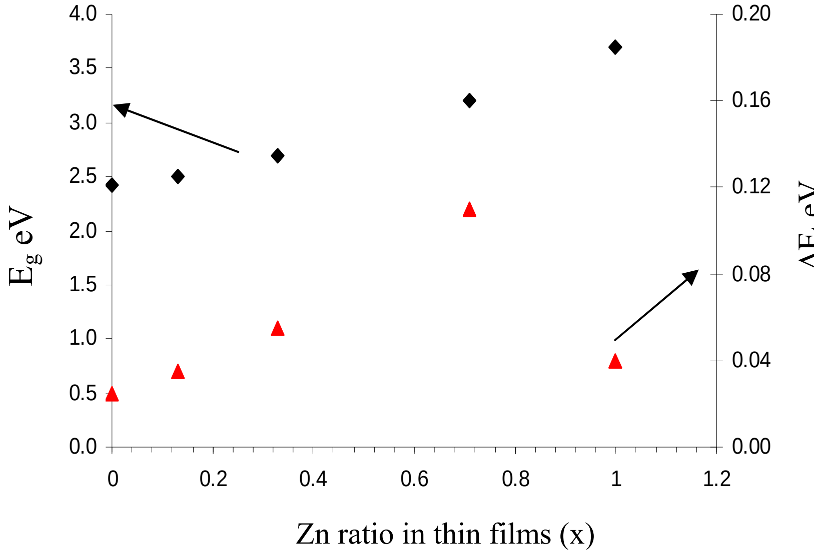

Fig.4: The band gap and tails width vs. Zn ratio in the Cd1$_{-x}$Zn$_{x}$S thin films

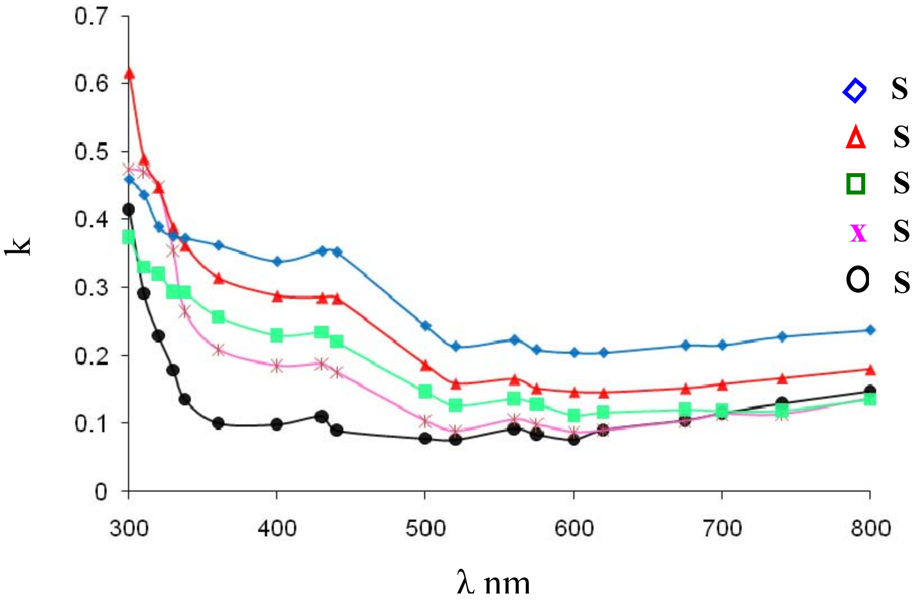

Fig. 5: The extinction coefficient of Cd$_{1-x}$Zn$_{x}$S prepared thin films

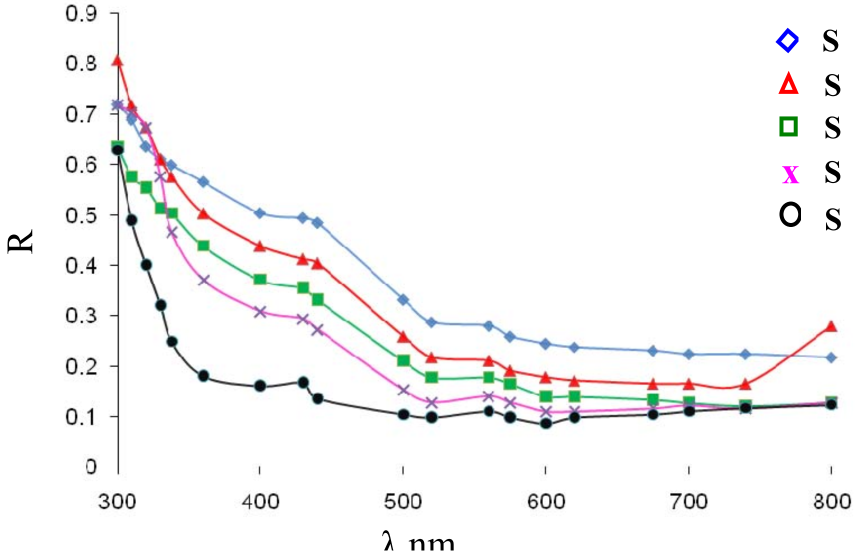

Fig.6: The reflectance of $Cd_{1-x}Zn_xS$ prepared thin films

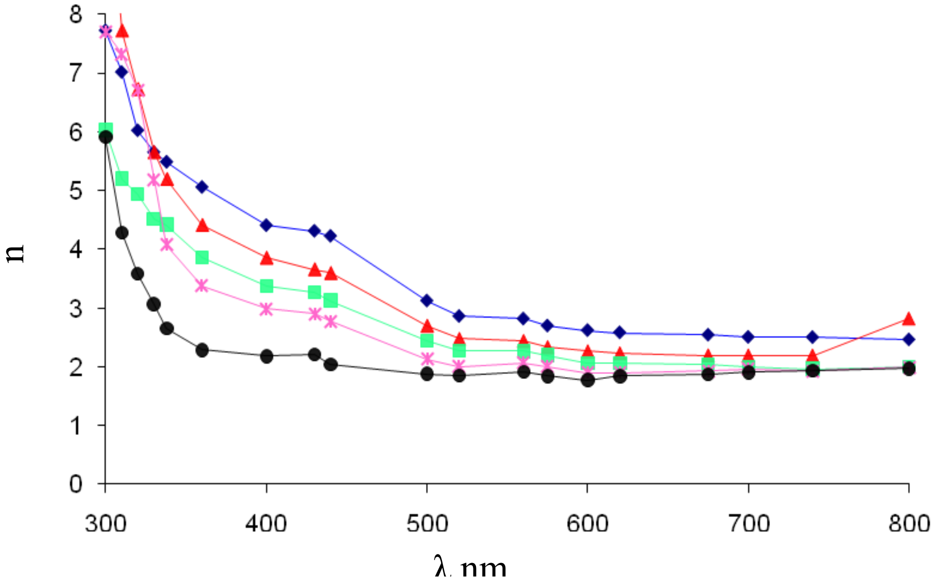

Fig. 7: The reflective index of $Cd_{1-x}Zn_xS$ prepared thin films

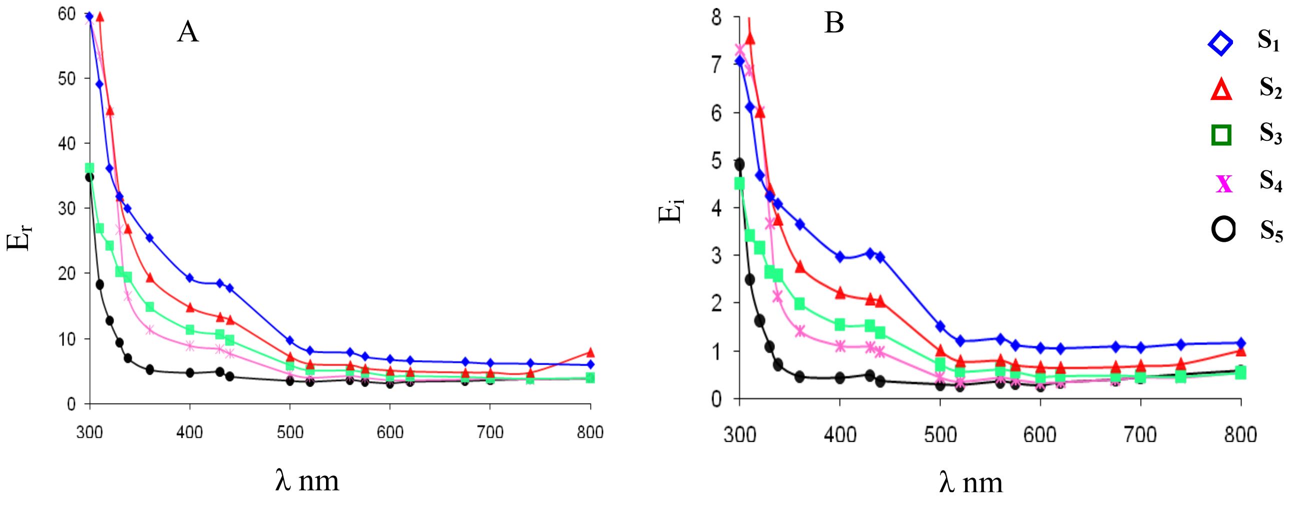

Fig. 8: The real (A) and the imaginary (B) parts of the dielectric constant the prepared $Cd_{1-x}Zn_xS$ thin films

The dispersion data of the refractive index can be calculated by single-oscillator model

The dispersion data of the refractive index can be calculated by single-oscillator model Which developed by Wemple and Didomenico [32] :

$$(n^2-1)^{-1} = \frac{E_o^2-(h\upsilon)^2}{E_d E_o} \tag{18}$$

Where E_o is the energy of the effective dispersion – oscillator and E_d is the dispersion energy, which measures the average strength of inter-band optical transitions. The oscillator energy E_o is an average of optical band gap. The oscillator strength (f) is given by:

$$f = E_o\, E_d \tag{19}$$

Plotting $(n^2\text{-}1)^{-1}$vs $(h\upsilon)^2$ as shown in figure 9 led to obtain E_d and E_o where the curve gradient represented $(E_dE_o)^{-1}$ and (E_o/E_d) determined from intercept on the vertical axis. The moments of optical spectra M_{-1} and M_{-3} can be determined from the following relations [32]:

$$E_o^2 = \frac{M_{-1}}{M_{-3}};\, E_d^2 = \frac{M_{-1}^3}{M_{-3}} \tag{20}$$

For x=0(CdS) we get good agreement results of E_o with others [33, 34].

The obtained M-1 and M-3 moments slightly varied and both decreases as Zn ratio increased in the film. These moments determine the average bond strength and the eqn. (20) indicates a single-oscillator approximation to the dielectric response of these materials. The optical moments are related to the macroscopic quantities like effective dielectric constant,effective number of valence electrons in the investigated material [35].The two moments M-1 and M¬-3 are calculated from the data of Eo and Ed and all are given in Table 4.

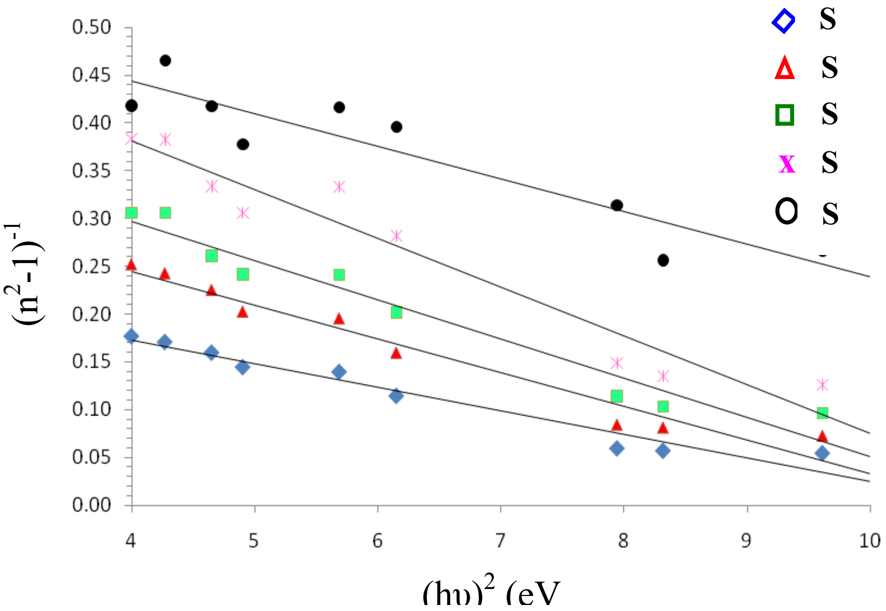

Fig. 9: Plots of $(n^2-1)^{-1}$ vs $(h\upsilon)^2$ of $Cd_{1-x}Zn_xS$ prepared thin films

Table 4: The Wemple-Didomenico oscillating parameters

Sample	**E_o(eV)**	**E_d (eV)**	**f(eV^2)**	**M_{-1}(eV^2)**	**M_{-3}(eV^2)**
S_1	4.12(1.70E_g)	24.25	51.41	5.88	0.34
S_2	4.14(1.65E_g)	17.25	71.41	4.16	0.24
S_3	4.08(1.50E_g)	13.60	55.49	3.33	0.20
S_4	3.89(1.20E_g)	10.26	39.91	2.63	0.17
S_5	2.85(0.77E_g)	6.48	18.46	2.27	0.28

3. Conclusion

In this work, $Cd_{1-x}Zn_xS$ thin films prepared by using chemical bath deposition (CBD) method. All films were a good adherent and homogeneous.

Films thickness decreased with increasing in Zn concentration so the transmission increases. Depending on Cd and Zn ions source **molar concentration,** the values of the direct optical band gaps changed from 2.42 eV for CdS to 3.7 eV for ZnS. The band tailing varies in the range (0.025- 0.11) eV. The optical constants such as, the extinction coefficient, the refractive index and the real and imaginary parts of the dielectric constant are found to depend on the Zn concentration in the films. The dispersion energy Ed and the single-oscillator energy E_o measured and used to calculate the moments of the optical spectra M_{-1} and M_{-3}.

Because of the optical properties of the prepared thin films, they can be used as a window material to make a solar cell. The $Cd_{1-x}Zn_xS$ thin films may be assumed as an ideal alternative material to CdS and ZnS since its composition can be simply controlled.

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