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Analysis of Zinc Sulphide (ZnS) and Cadmium Sulphide (CdS) thin films deposited by improved solution growth method at 300K

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Abstract

We successfully deposited good quality semi conducting thin films of zinc sulphide (ZnS) and cadmium sulphide (CdS) on glass slides at 300K and pH values of 8, 10 ***and*** 12 ***by improved solution growth method. Ethylenediamine-tetra acetate (EDTA), another complexing agent, with pH oppose that of bath constitutions was added to bath reagents to enhance control and stabilize the deposition pH at different values suitable for film growth. X-ray diffraction method was used to confirm the depositions. Electron micrographs of the films reveal uniform surface deposition. Absorbance (A) spectra data were measured by a single beam spectrophotometer at wavelength range*** 200nm ***to*** 900nm***. Other optical and solid state properties were calculated using the data and compared with other deposited thin films. Average optical and solid state properties of ZnS thin films include absorbance ranging from*** 0.049 to 0.110***, transmittance*** 0.776 to 0.893***, refractive index 1.63 to 2.02, film thickness*** 0.035 ***to*** 0.056***μm and band gap*** 2.30 ***to*** 2.62 $\pm$0.05***eV. For CdS thin films, the absorbance ranges from 0.067 to 0.080, transmittance*** 0.832 ***to*** 0.857***, refractive index*** 1.76 ***to*** 1.84***, film thickness*** 0.002 ***to*** 0.017***μm and band gap*** 1.62 ***to*** 2.25 $\pm$0.05***eV. Films with refractive index lower than 1.9 could be used in antireflection coatings, eyeglass coating and solar thermal control coatings. Those with refractive index greater than*** 1.9***, could be useful in construction*** *of poultry production, anti dazzling coatings and thin films solar cells.*

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1.0 Introduction

Solution growth method is a simple, cheap and convenient technique of depositing high quality and reproducible compound semiconductor thin films [3, 4, 12, 13, and 22]. Films produced by this technique have comparable structural and optoelectric properties to those produced using other sophisticated thin film deposition techniques such as chemical vapour deposition and sputtering. The method has added advantages of depositing films with good adhesion and uniform thickness on large surface areas and shapes of both metallic and non-metallic substrates. The technique has also been applied in producing emerging materials for solar cells, protective coating and solar thermal controls in buildings. It is now being adopted by some industries [7, 12, 13, 20, 22].

Some works have been reported on the preparation of metal chalcogenide films using various deposition techniques. Basu and Pramanik [1] deposited cobalt sulphide films using solution growth method with thioacetamide as complexing agent. Ilenikhena and Okeke [12] produced cadium sulphide thin films using solution growth technique with ammonia. Osuji [22] deposited cadium selenide and cadium sulphide tin films using chemical bath technique with triethanol amine (TEA).

Ezema and Okeke [8] deposited palladium sulphide thin films using solution growth method with di-sodium thiosulphate. Ilenekhena and Okeke [13] also used solution growth technique with ammonia to produce cobalt sulphide thin films.

A problem associated with the solution growth method is the combinations of the various bath constitutions to obtain the pH for deposition. The combinations could be tedious and wasteful before obtaining the suitable deposition pH value. This could be improved by adding another complexing agent with pH oppose that of bath constituents to the bath mixture to enhance control and stabilize the deposition pH at different suitable values for film deposition.

This paper reports the deposition of high quality semi conducting films of zinc sulphide (ZnS) and cadmiun sulphide (CdS) on glass slides by improved solution growth method at 300K and pH of 8, 10 and 12. Ethylenediamine-tetra acetate (EDTA), with pH oppose that of the bath constitutions was added to bath reagents to enhance control and stabilize the deposition pH at different values suitable for film deposition. The optical and solid state properties obtained for the deposited thin films were calculated and compared with some other deposited films. Possible applications of the deposited thin films were also discussed.

2.0 Theory

The absorbance (A) of a semi conducting film is the common logarithm of the reciprocal transmittance (T), that is:

$$A = log_{10}(T^{-1}) \qquad (2.1)$$

From equation (2.1) the absorbance (A) and transmittance (T) are related [7] by:

$$T = 10^{-A} \qquad (2.2)$$

The spectra reflectance (R), absorbance (A) and transmittance (T) have a relationship which allows for conservation of energy [16]:

$$A + T + R = 1 \qquad (2.3)$$

At normal reflection, the reflectance (R) and the refractive index (n) of semi-conductors are related [10, 18] by:

$$n = (1 + R^{1/2}) / (1 - R^{1/2}) \qquad (2.4)$$

The transmittance (T), coefficient of absorption (α) and distance (dμm) of the film traversed are related [7, 23] by:

$$T = \exp(-\alpha d) \qquad (2.5)$$

From equation (2.5) for a unit distance traversed:

$$\alpha = In(T^{-1}) \times 10^{6}\ m^{-1} \qquad (2.6)$$

The coefficient of absorption (α) is also related to extinction coefficient, k and wavelength of radiation, λ [28] by:

$$\alpha = 4\pi k/\lambda \qquad (2.7)$$

According to Pankove [23] and Cho et al, [5] the complex dielectric constant is:

$$\in_c = (n + ik)^2 \qquad (2.8)$$

where the real dielectric constant is:

$$\in_r = n^2 - k^2 \qquad (2.9)$$

and the imaginary dielectric constant:

$$\in_i = 2nk \qquad (2.10)$$

The optical conductivity (σ_o) of the film is given by:

$$\sigma_o = \alpha nc/4\pi \qquad (2.11)$$

where c is the velocity of light. The electrical conductivity σ_e of the film [9] is giving by:

$$\sigma_e = 2\lambda\, \sigma_o/\alpha. \qquad (2.12)$$

Near the absorption edge, the dependence of absorption coefficient (α) on photon energy ($h\nu$) [6, 18] is giving by:

$$\alpha = A(h\nu - E_g)^{\gamma\Psi} \quad (2.13)$$

where E_g is the bandgap, $\gamma = \frac{1}{2}$ for allowed direct transition and $\Psi\gamma = \frac{3}{2}$ for forbidden direct transition. Value of $\gamma = 2$ for allowed indirect transition and $\gamma = 3$ for forbidden indirect transition. The constant $A = 3.38 \times 10^7\ n^{-1}\ (m_e/m_o)^{1/2}\ E_g/h\nu$ where m_e is the effective electron mass and m_o is the free electron mass for direct allowed transition. The plots of α^2 against hν is a straight line with a deviation in the region of absorption edge. Extrapolation to the point where $\alpha^2 = 0$ gives the energy gap. The thickness (t) of the films based on the optical method for transmittance (T) of light through a weak absorbing film on non-absorbing substrate [11, 23] is giving by:

$$t = \text{In}[(1 - R)^2/T]/\alpha \quad (2.14)$$

3.0 Experimental details

The deposition of zinc sulphide (ZnS) and cadmium sulphide (CdS) thin films on glass slides were successfully carried out using solution growth technique at room temperature of 300K and pH of 8, 10 and 12. The glass slides were cleaned by degreasing them in concentrated nitric acid (HNO_3) for 3 days, thoroughly washed in detergent solution, rinsed in distilled water and dried in air. The cleaned glass surfaces provided nucleation centres for growth, good adhesion and uniform deposition of the films. Reaction baths were 50ml glass beakers containing different molar solutions and volumes of deposition bath constituents. The bath constituents for the deposition of zinc sulphide (ZnS) thin films were zinc chloride ($ZnCl_2$) as source of (Zn^{2+}), thiourea [$(NH_2)_2CS$] as a source of sulphide ions (S^-) in the presence of sodium hydroxide, ammonia (NH_3) as complexing agent while ethylenediaminetretra acetate (EDTA) (another complexing agent) was used to vary pH values of the deposition solutions. Distilled water was added to raise the volume of the bath solutions to a certain level. For the deposition of cadmium sulphide (CdS) thin films, cadmium chloride -1 - water ($CdCl_{2*}\,H_2O$) was used to replace the zinc chloride ($ZnCl_2$) in a similar reaction bath. Details of bath constituents for the preparation of metal sulphide (YS) thin films are shown in table 1.The symbol Y represents Zn and Cd in the deposition of ZnS and CdS respectively. The complexing agent ammonia (NH_3) formed complex ions with Y^{2+}. It slowly released Y^{2+}, ensured ion by ion condensation of Y^{2+} and S^{2-}, controlled the growth rate of the deposited thin films and eliminated spontaneous precipitation of the chemical reagents in the bath. The solution baths were stirred with a glass rod and their initial pH values noted at room temperature of 300K. A cleaned glass slide was suspended in each reaction bath for 24 hours. After deposition time, the coated glass slides were rinsed with distilled water and dried in air. Pretest runs were carried out to determine the optimum deposition parameters such as deposition time, pH and volumes of bath constituents. The most probable reaction equation for the deposition of (ZnS) thin films is

$$ZnCl_2 + NH_3 + (NH_2)_2CS + 2NaOH \rightarrow ZnS\downarrow + NH_3 + CH_2\,N_2 + 2H_2O + 2NaCl$$

The basic reaction equation for deposition of CdS thin films is

$$CdCl_{2*}\,H_2O + NH_3 + (NH_2)_2CS + 2NaOH \rightarrow CdS\downarrow + NH_3 + 3H_2O + 2NaCl + CH_2\,N_2$$

Table 1: Bath constituents for preparation of ZnS and CdS thin films

Initial Bath pH	0.3M $YCl_2(H_2O)^r$ Vol. (ml)	5.0M NH_3 Vol. (ml)	1.0M TEA VoL.(ml)	2.5M NaOH Vol. (ml)	0.8M $(NH_2)_2CS$ VoL. ml	H_2O Vol (ml)	0.2M EDTA Vol (ml)
8	8	3	2	2	7	19	23
10	7	4	3	3	8	16	20
12	6	5	4	4	9	13	15

The symbols Y = Zn and r = O for $ZnCl_3$, Y = Cd and r = 1 for $CdCl_{2*}H_2O$

The absorbance (A) spectra data of the deposited thin films where obtained using a computerized single beam spectrophotometer (Pharmacia LKB Biochrom 4060) was used to obtain the absorbance spectra data of deposited thin films in the ultraviolet -visible - near infrared (UV - VIS - NIR) regions at wavelength range of 200nm to 900nm. The reference and coated glass slides were mounted on a rotating holder at the reference and sample compartments respectively and scanned to obtain the absorbance spectra data. Other optical and solid state properties were obtained from the spectra data by calculations based on the theory. Structural characterization of the films was determined with x- ray diffraction technique using Diano cooperation x-ray diffractometry (model XRD 2100 E*) and copper target (CuKα) with wavelength 1.540502 *Å*, current 30*mA* and voltage 45 kV. The surface microstructure of the films was viewed using electron microscope at magnification x 100.

4.0 Results and analysis

The absorbance spectra data of the deposited zinc sulphide (ZnS) and cadmium sulphide (CdS) thin films were obtained by direct measurement and displayed in fig. 1 and 2 respectively. They were used to determine the other optical and solid-state properties the deposited thin films based on theory. The thin films have high absorbance for wavelengths lower than 300nm and low absorbance for wavelength range 350 – 850nm. Figure 1 shows that zinc sulphide (ZnS) thin film produced at pH of 8 has negative absorbance at wavelength range of 400nm to 500nm and at 700nm and 800nm with a minimum of -0.020 at 500nm. Similarly, the film produced at pH of 12 has negative absorbance at 400nm to 500nm and at 700nm with a minimum of 0.037 at 400nm. The films produced at pH of 10 have positive absorbance except at 850nm where it is 0.001. Fig 2 also shows that cadmium sulphide thin films have similar absorbance spectra. The films produced at pH of 8 have negative absorbance in the wavelength range 300nm to 650nm and at 850nm. It has a minimum absorbance of -0.024 at wavelength of 550nm. Films produced at pH of 12 has negative absorbance at 300nm, 450 – 500nm and at 850nm with a minimum of 0.04 at 300nm. Film produced at pH of 10 has positive absorbance spectra except at 350nm and at 850nm with a minimum of 0.014 at 850nm. The spectra transmittance (T) and reflectance (R) of zinc sulphide and cadmium sulphide thin films were calculated from equations (2.2) and (2.3) and shown in Figures 3 and 4 respectively. Both films have low transmittance for wavelengths lower than 300nm and high transmittance for wavelength range 300nm to 850nm. Films with negative absorbance have transmittance greater than 1.00 while those with positive absorbance have transmittance lower than 1.00. The transmittance of zinc sulphide thin films varies from 0.312 to 0.508 for wavelength lower than 300nm and from 0.805 to 1.089 for wavelength range 350 to 850nm. Film produced at pH of 12 has the highest transmittance of 1.089 at 400nm. For cadmium sulphide thin films, the transmittance varies from 0.339 to 0.401 for wavelength lower than 300nm and from 0.891 to 1.054 for wavelength range 350nm to 850nm. The films produced at pH of 12 also have the highest transmittance of 1.054 at 850nm. Figures 3 and 4 also show that both thin films exhibited high reflectance (R) of 0.182 to 0.203 for wavelengths lower than 300nm. The reflectance varies from –0.052 to 0.101 for zinc thin film and from –0.031 to 0.059 for CdS thin films in the wave length range of 350 to 850nm. The films with negative absorbance also exhibited negative reflectance. The thin films of ZnS produced at pH 0f 12 and CdS produced at pH of 8 and 12 with high transmittance and low reflectance characteristics could be employed in antireflection coatings for solar thermal devices and eye glass coatings to reduce solar reflectance, increase the transmittance and improve their efficiencies. The high transmittance and low reflectance properties of CdS thin film produced at pH of 10 in the visible region are desirable characteristics for ideal solar control glazing to avoid glare problems [17] and could be employed in solar thermal control coatings. The thin films of ZnS produced at pH of 8 and 10 with relatively lower transmittance and higher reflectance could be useful in construction of poultry houses to allow enough infrared radiation to the warm the very young chicks during the day.

This could also reduce the cost of energy consumption through the use of stoves, heaters, electric bulbs, etc. and the hazards associated with them while at the same time protecting the chicks from ultraviolet radiation. The application of solar energy as a source of heat in chick brooding is environmentally acceptable and promotes sustainable development [21]. Solar energy technologies are also applicable to egg incubation and to the drying of chicken manure [19]. These thin films could also be used in photocells [22], thin film solar cells [14] and in anti dazzling coatings for car windscreens and driving mirrors to reduce the dazzling effects of light at night. Refractive index (n) was computed from equation (2.4). The variation of refractive index (*n*) with photon energy for ZnS and CdS thin films are shown in figures 5 and 6 respectively. Both films exhibited high refractive index (*n*) for photon energies higher than 4.14 eV and low refractive index (*n*) values for photon energy range 1.46 eV to 4.14*eV*. For zinc sulphide thin films, values of refractive index vary from 2.29 to 2.64 for photon energy higher than 4.14eV and from 1.12 to 1.92 for photon energy range 1.46*eV* to 4.14*eV*. In the case of cadmium sulphide (CdS) thin films, Figure 6, values of refractive index vary from 2.55 to 2.63 for photon energy higher than 4.14*eV* and from 1.00 to 1.73 for photon energy range 1.46*eV* to 4.14*eV*. The films of both ZnS and CdS with very low refractive index values could find useful applications in antireflection coatings in agreement with the results of Brinker and Harringston [2] and Petit and Brinker [25]. Such films with refractive index lower than 1.9 could be employed to antireflect photovoltaic from 0.36 to 0.04 and improve the transmittance of glass from 0.91 to 0.96. The coefficient of absorption was calculated from equation (2.). The variation of coefficient of absorption (α) with photon energy for the zinc sulphide (ZnS) and cadmium sulphide (CdS) thin films is shown in figures 7 and 8 respectively. The magnitude of the coefficient of absorption $\alpha = 10^6$ m^{-1} is within the α range 10^6 to 10^7 m^{-1} for semi-conductor thin films suitable for polycrystalline thin film solar cell [15]. The band gaps of the films were obtained from equation (2.13). The coefficient of absorption method is the simplest and perhaps the most direct method of determining the band gap of semiconductor. Values of band gap for the deposited zinc sulphide films vary from 2.30 to 2.62 $\pm$0.05eV. For the cadmium sulphide films, the band gap varies from 1.62 to 2.25 $\pm$0.05eV. These results compare well with the values of 3.30*eV* for ZnO, 2.42 to 2.82*eV* for CdS and 1.74*eV* for CdSe films [6] and could be used thin film solar cells. Optical conductivities of the films were computed from equation (2.11). Both films have good photo response with average optical conductivity (σ_o) of 10^{13} s^{-1}. The magnitude of average electrical conductivities obtained from equation (2.12) for both thin films is 10^{-1} (ohm-cm) $^{-1}$ within the electrical conductivity range 10^{-12} to 10^2 (ohm – cm)$^{-1}$ for semiconductors [24, 26, 27]. Film thickness was calculated from equation (2.14) The thickness of ZnS thin films varies 0.035 to 0.056µm. For CdS thin films, the thickness varies from 0.002 to 0.017µm. Other properties determined include extinction coefficient (*k*) from equation (2.7), real dielectric constant ($\in_r$) from equation (2.9) and imaginary dielectric constant ($\in_i$) from equation (2.10). Average optical and solid-state properties at wavelength of 550*nm* for ZnS and CdS are shown in Tables 2 and 3 respectively. The x-ray diffraction patterns of the uncoated glass and deposited films on glass are shown in Figure 9 and Figure 11 for zinc sulphide (ZnS) and cadmium sulphide (CdS) thin films respectively. The diffraction patterns reveal diffraction peaks at some 2 – theta (2θ) values. Electronmicrographs of the films at magnification of x100 shown in Figures 10 and 12 for zinc sulphide (ZnS) and cadmium sulphide (CdS) respectively reveal their uniform surface deposition.

Table **2: Average optical and solid-state properties for zinc sulphide thin films**

pH	T	n	k x 10^{-2}	α x 10^6 (m^{-1})	$\in_r$	σ_o x 10^{13} s^{-1}	σ_e (ohm-cm)$^{-1}$	t (µm)	E_g $\pm$ 0.05 (eV)
8	0.776	2.02	1.11	0.254	4.07	1.22	0.53	0.045	2.30
10	0.789	1.98	1.04	0.237	3.92	1.12	0.52	0.035	2.62
12	0.893	1.63	0.45	0.113	2.66	0.44	0.43	0.056	2.30

Table 3: Average optical and solid-state properties for cadmium sulphide thin films

pH	T	n	k x 10^{-2}	α x 10^{6} (m^{-1})	$\in_r$	σ_o x 10^{13} (s^{-1})	σ_e (ohm-cm)$^{-1}$	t (μm)	E_g ±0.05 (*eV*)
8	0.857	1.76	0.62	0.143	3.10	0.58	0.44	0.002	1.62
10	0.832	1.84	0.81	0.184	2.40	0.81	0.48	0.002	1.95
12	0.847	1.79	0.73	0.166	3.22	0.71	0.71	0.017	2.25

5.0 Conclusion

We successfully deposited good quality semiconductor thin films of zinc sulphide (ZnS) and cadmium sulphide (CdS) on glass slides at 300K and pH values of 8, 10 and 12 with improved growth characteristics by improved solution growth method. Ethylenediamine-tetra acetate (EDTA) with pH oppose that of bath constituents was added to bath constitutions to enhance control and stabilize deposition pH at different values suitable for film growth. X-ray diffraction method was used to confirm the depositions. A single beam spectrophotometer (Pharmacia LKB Bichrom 4060) was used to obtain the spectra absorbance data. Other optical and solid-state properties of the films such as transmittance, refractive index, extinction coefficient, optical conductivity, electrical conductivity, film thickness, coefficient of absorption, energy gap, etc. were calculated based on theory. The films with refractive index lower than 1.9 could be used in antireflection coatings, eyeglass coatings and solar thermal control coatings. Those with refractive index greater than 1.9 could be useful in poultry production, antidazzling coatings and solar cells.

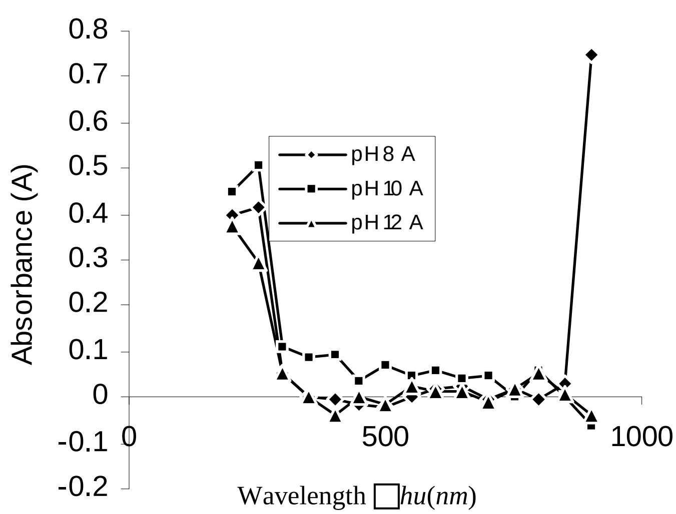

Figure 1: Absorbance (A) spectra for ZnS thin films produced at 300K

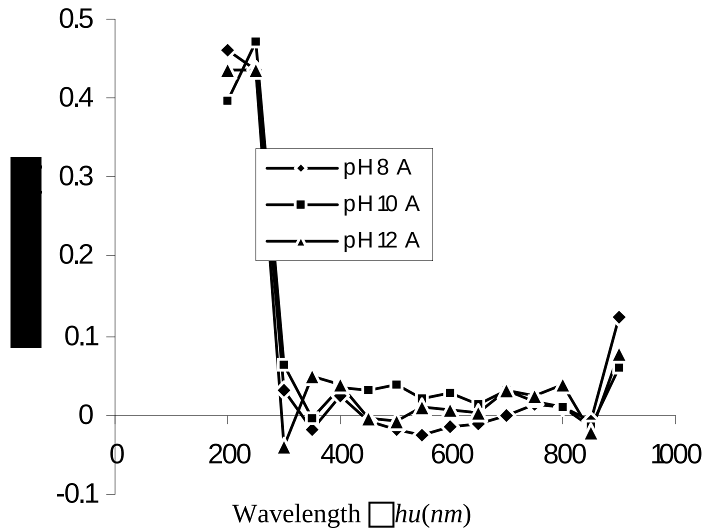

Figure 2: Absorbance (A) spectra for CuS thin films produced at 300K

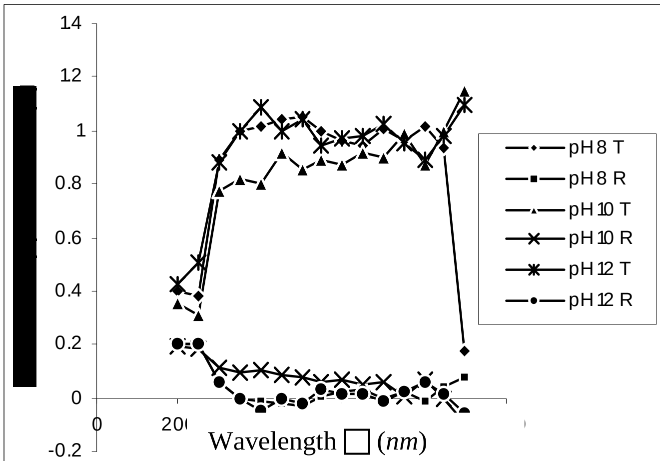

Figure 3: Transmittance (T) - Reflectance (R) for ZnS thin films produced at 300K

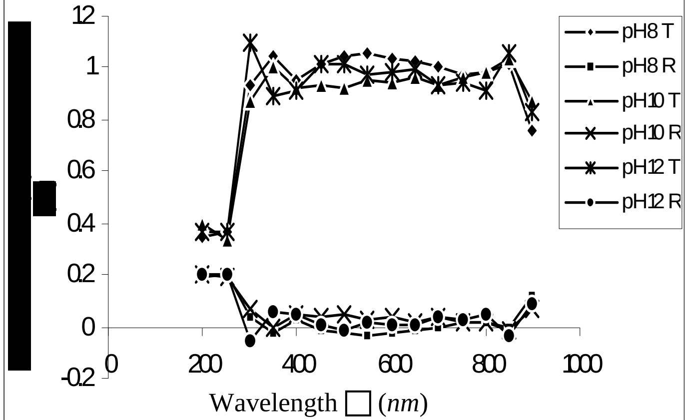

Figure 4: Transmittance (T) - Reflectance (R) spectra for CuS thin films produced at 300K

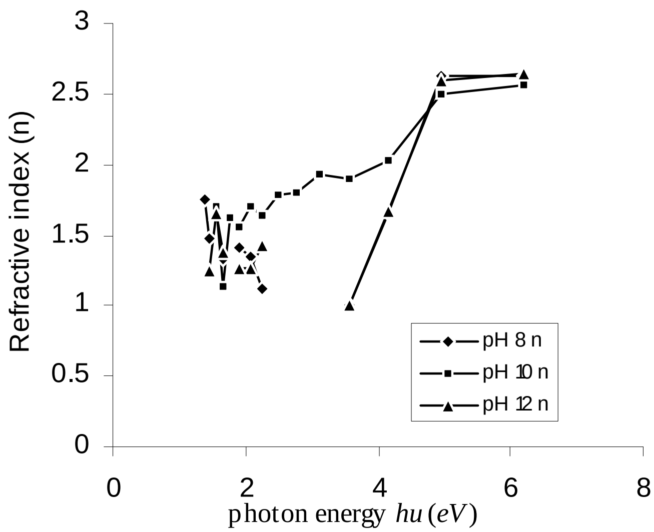

Figure 5: Variation of refractive index (n) against photon energy for ZnS thin films produced at 300K

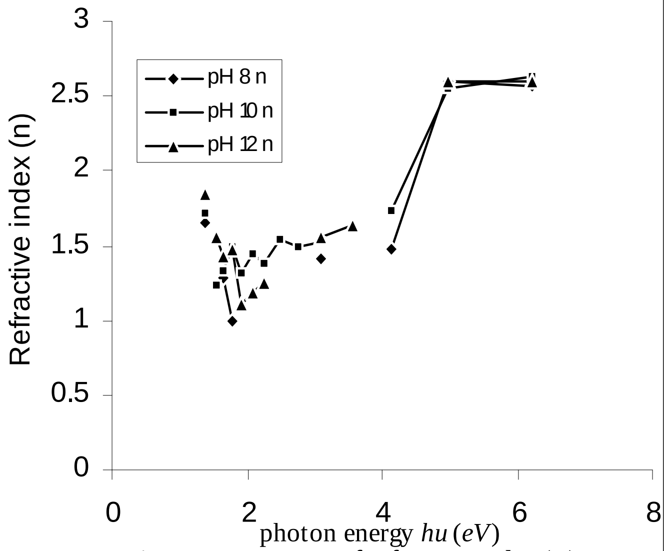

Figure 6: variation of refractive index (n) against photon energy for CuS thin films produced at 300K

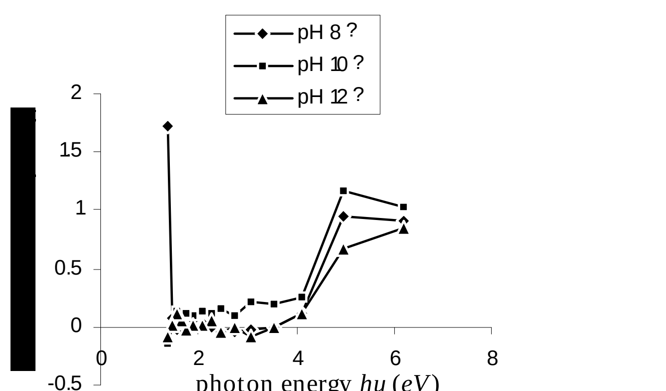

Figure 7: variation of coefficient ofabsorption (□) against photon energy for ZnS thin films produced at 300K

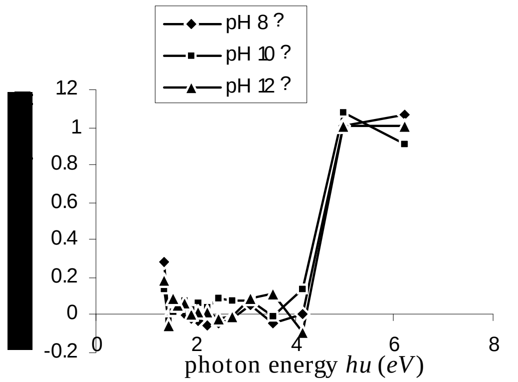

Figure 8: Variation of coefficient of absorption (□) against photon energy for CuS thin films produced at 300K

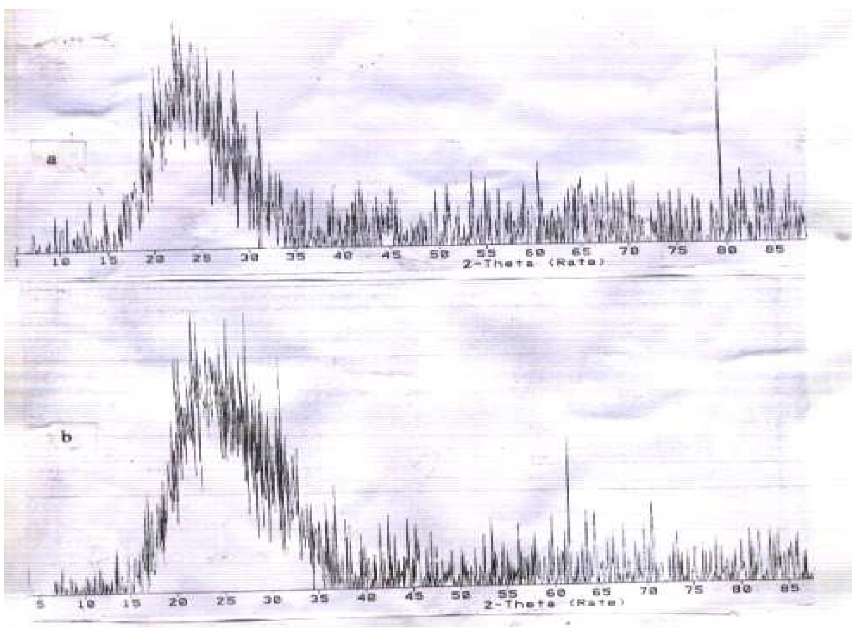

Figure 9: X-ray diffraction results for (a) uncoated glass slide (b) ZnS thin film produced at 300K

Figure 10: Electron micrograph of ZnS thin film deposited at 300K

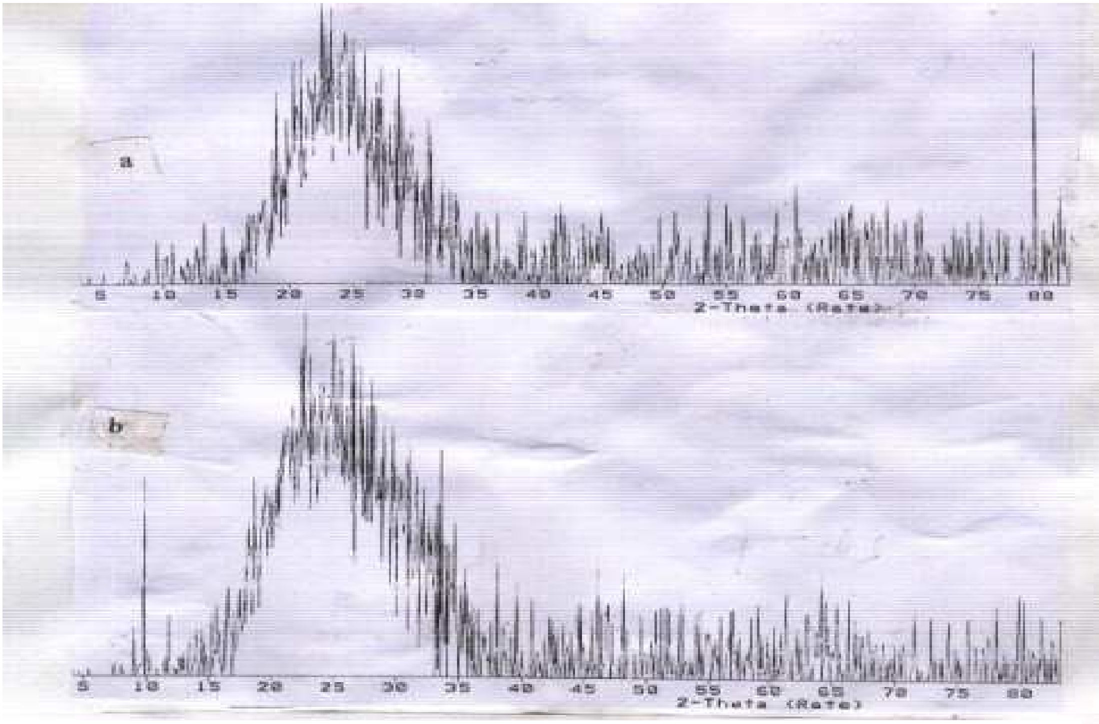

Figure 11: X-ray diffraction results for (a) uncoated glass slide (b) CdS thin film produced at 300K

Figure 12: Electron micrograph of CdS thin film deposited at 300K

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