Optical, Structure and Electrical Properties of $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ Thin Films by Pulsed Laser Deposition (PLD) Method

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Abstract. In this work, CdS, PbS, and $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ nanoparticles with x ranged from 0.25 to 0.75 were synthesized using chemical co-precipitation mode. The pulsed laser deposition (PLD) method with laser system (Nd: YAG Laser system) was performed to make thin films of all samples which was normally performed under low gas pressure and under the following conditions; 300 mJ energy, 100 pulses, 6 Hz frequency and in the vacuum chamber on the substrates (10$^{-2}$ torr). Electrical properties (D.C conductivity, Hall Effect and current-voltage measurements) of the prepared semiconductive thin films were studied using different devices. The prepared samples were characterized by UV-Vis spectroscopy (in the range 300-1100 nm) to study the optical properties, AFM and SEM to check the surface morphology (Roughness average and shape) and the particle size. XRD technique was used to determine the crystalline structure, the purity of the phase and the crystal structure. The crystalline size averages of the nanoparticles have been found to be 19.8, 11.0, 8.8 and 11.7 and 27.3 nm for PbS, $\text{Pb}_{0.75}\text{Cd}_{0.25}\text{S}$, $\text{Pb}_{0.5}\text{Cd}_{0.5}\text{S}$, $\text{Pb}_{0.25}\text{Cd}_{0.75}\text{S}$, and CdS respectively. The results indicate that crystalline structure of all prepared samples is cubic except CdS which shows hexagonal and cubic structure.

Keywords: CdS; Chemical co- Precipitation Deposition; PLD; PbS; $\text{Pb}_{1-x}\text{Cd}_x\text{S}$

1. Introduction:

Cadmium sulfide (CdS) is a type of chalcogenide semiconductor material with high clarity, wide and clear band gap (2.4 eV), high sensitivity of the electron, and has hexagonal and cubic structure [1]. It is useful in photochemical hydrogen production applications [2], diode sensors [3], photovoltaic cells [4], photosensor [5]. PbS semiconductor has small band gap (0.41 eV), exhibits its importance in highly efficient photovoltaic conversion devices, optical switching, and related photoelectric devices [6-8]. These properties have been correlated with the growth conditions and the nature of substrates. For these reasons, many research groups have shown a great interest in the development and study of this material by various deposition processes such as electrodeposition, spray pyrolysis, photoaccelerated chemical deposition, microwave heating and chemical bath deposition (CBD), sol-gel spin coating and pulsed-laser deposition [9-12]. Among the new and up to date techniques used for thin film deposition, pulsed laser deposition (PLD) is one of the most versatile methods to obtain layers of several materials that can be processed into a pellet target. Therefore high quality films can be obtained at a fairly low substrate temperature. Furthermore, PLD is more flexible than other conventional techniques, and it is feasible to control the thickness of films [13, 14]. Mixed thin film structures of PbS and CdS ($\text{Pb}_{1-x}\text{Cd}_x\text{S}$) have generated significant interest because they offer the advantage of tunable optical and opto-electronic properties of PbS, viz., its band gap, electrical conductivity, and structural properties [15, 16]. In the current work, we report the structural and optical properties of $\text{Pb}_{1-x}\text{Cd}_x\text{S}$, where $x = 0, 0.25, 0.5, 0.75$, etc.
1, thin films on glass substrate grown by PLD method at various concentration. The characterization of thin films and the optical and electrical properties were also studied.

2. Experimental:

2.1. Chemicals

The cadmium acetate \([\text{Cd} (\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}]\), lead acetate \([\text{Pb} (\text{CH}_3\text{OO})_2 \cdot 2\text{H}_2\text{O}]\), thiourea as a precursor of \(\text{Cd}^{+2}\), \(\text{Pb}^{+2}\), \(\text{S}^{-2}\) ions respectively, as well as sodium hydroxide as a stream of medium alkaline growth shall be the base materials for the preparation. The quantities and molarities of chemical compounds used in nanoparticles preparation and their procedures are previously described [17].

2.2. The Procedure

The preparation of pellets is as follows: \(\text{PbS, CdS, Pb}_{0.75}\text{Cd}_{0.25}\text{S, Pb}_{0.5}\text{Cd}_{0.5}\text{S and Pb}_{0.25}\text{Cd}_{0.75}\text{S}\) were forced for 10 minutes under pressure of 5 tons into pellet with (0.2 cm) thickness and 1 cm diameter. Hydraulic piston style SPECAC was used.

The PLD methodology was performed to make thin films of all samples. In a vacuum chamber, the method of pulsed laser deposition was normally performed under 10\(^{-2}\) torr vacuum conditions, under low gas pressure. The substrate is positioned parallel to the target surface and in front of it. There is sufficient gap between the target and the substrate, so that the holder of substrate did not block the laser beam incident. The length between the target and the substrate is 2 cm and the length is 12 cm from the target to the laser supply. PLD was employed to deposit thin films under the following conditions; 300 mJ energy, 100 pulses, 6 Hz frequency, in the vacuum chamber on the substrates.

2.3. Characterization instruments:

XRD analysis was performed using X-ray diffractometer of copper-filtered monochromatic CuK\(\alpha\) radiation (\(\lambda=1.54\) 0Å), at scales 20 (20-80), Shimadzu, Japan. Ultraviolet-Vis spectrometer (Shimadzu, Japan) has been used to measure the optical absorption obtained from solution. Atomic force microscopy (AFM), SPMAA 3000, Advanced Angestrum Inc., USA, and field emission scanning electron microscopy (FE-SEM), (SEM; Oxford instruments model SEM: S-3200N), analyzed the morphology and roughness of the surface.

3. Results and Discussion:

The PbS, CdS and \(\text{Pb}_{1-x}\text{Cd}_x\text{S}\) films have been characterized by X-ray diffraction scanned from 20 = 20 to 80 and the results illustrated in figure 1.

The observed PbS diffraction peaks are at the values of 20; 25.94\(^{\circ}\), 30.07\(^{\circ}\), 43.03\(^{\circ}\) and 51.94\(^{\circ}\) correspond to the Miller indices (111), (200), (220) and (311) respectively. The PbS findings are compatible with findings contained in the table 1, which was indexed inside a cubic structure and checked using a Regular Card (JCPDS 96-901- 3403) [18, 19]. The peaks has not been found to suit metallic Pb or its oxide. CdS diffraction peaks found are 20; 25.16\(^{\circ}\), 25.59\(^{\circ}\), 28.30\(^{\circ}\), 44.30\(^{\circ}\) and 52.0\(^{\circ}\) be aligned with Miller indices as shown in table 1 of the standard JCPDS number [86-900-8863] [95-99-96-900-8840] and is indexed inside a cubic and hexagonal structure [20, 21]. The peaks observed of diffraction are 20 values of \(\text{Pb}_{0.75}\text{Cd}_{0.25}\text{S, Pb}_{0.5}\text{Cd}_{0.5}\text{S, and Pb}_{0.25}\text{Cd}_{0.75}\text{S}\), at Miller Indices (121), (200) and (220), which is compatible with Isaac Nkrumah et.al [22], and S. Rajathy et.al [23]. These peaks of
diffraction are 2θ values of 26.05°, 30.34°, 43.36°, 26.38° and 30.71°. Thus, with Cd\(^{2+}\) taking more and more of the Pb\(^{2+}\) space, the internal strain will increase and the PbCdS solid crystal structure will become unstable. The ionic range of Cd\(^{2+}\) is even smaller than that of Pb\(^{2+}\). This indicates very specifically that the Cd\(^{2+}\) ions are quite resolved in the PbS particle. When concentration increases, leading to the decrease in grain size, the diffraction peaks are somewhat broader. In the meantime, doping-inducing structural disorder decreases the intensity of diffraction peaks.

The calculation by scherrer equation shows that the crystalline size of PbS, Pb\(_{0.75}\)Cd\(_{0.25}\)S, Pb\(_{0.5}\)Cd\(_{0.5}\)S, Pb\(_{0.25}\)Cd\(_{0.75}\), Pb\(_{0.25}\)Cd\(_{0.75}\)S and CdS are averaged to 19.8, 11.0, 8.8 and 11.7 and 27.3 nm respectively.

In this work, the AFM is utilized to carry out surface morphology of the prepared thin films deposited on the glass substrates. Figure 2 shows smooth and consistent adhering films around the whole surface of the glass suspension without any voids, pinholes or crevices and with significant quantities of grains. The size of grain of thin films is several tens nanometers that demonstrate the crystalline quality of the films. These results of CdS and PbS size of grain agree with the results of Nabeel A. Bakr [24], and Abdul-Majeed E. Ibrahim et.al [19]. Table 2 indicates the typical roughness and grain size of films.

SEM is used in the surface morphology research. From the figures below, figure 3, which are display nanoparticles of various diameters with spherical shape and show uniformly homogeneous surface. This result confirms the results obtained in XRD and AFM measurements. Nucleation and coalescence is the product of the difference in grain size and shape. Generally, coalescence and nucleation affect the development of grains by shifting grain boundaries [23].

The optical transmittance spectra of the Pb\(_{1-x}\)Cd\(_x\)S thin films prepared at pulsed Laser Deposition are seen in figure 4. These films were calculated to transmit on average in observable wavelength areas to PbS, Pb\(_{0.75}\)Cd\(_{0.25}\)S, Pb\(_{0.5}\)Cd\(_{0.5}\)S.Pb\(_{0.25}\)Cd\(_{0.75}\) and CdS for 17.50, 23.52, 31.60, 49.51 and 80.06 respectively. This results are compatible with Abdul-Majeed E. Ibrahim et.al [19] S. Rajathi et.al [23], and Sachin H. Dhawankar et.al [25] results. With the increase in CdS concentration, transmission increases. The transmission increase can be clarified by the rise in reflection and absorption. This variation (increase and decrease) in transparency relates to the structural characteristics of film, and changes in transmission are understood to depend on the characteristics of films [26].

The Pb\(_{1-x}\)Cd\(_x\)S thin film absorption spectrum from 350 nm to 900 nm, figure 5, shows different concentrations. A red shift is found in the absorption edge to lower band gap, table 3. The energy level of dopants is under the edge of the conduction band (CBE) and over Pb\(_{1-x}\)Cd\(_x\)S valence band (VBE). This energy gap leads to a red transformation in the band gap and visible light absorption through charge transfers between a doping 3d electron and Pb\(_{1-x}\)Cd\(_x\)S (CB or VB) or apply a power-level crystal field transition [26]. The PbS and CdS film has also been changed to dope visible illumination with a red shift in the absorption layer. Increased absorption in the visible may be due to the change from a charge transfer, which can be defined as an electron excitation from the orbital d of metal ions to Pb\(_{1-x}\)Cd\(_x\)S [27]. The graph shows that the lead absorbance is greater than the CdS. The permeability is the opposite, which is known as cadmium permeability and lead absorbent.
The absorption coefficient $\alpha$ from the high absorption region is found for the films by equation 1 [28].

$$I = I_0 \exp(-\alpha t) \tag{1}$$

If $I_0$ is the light strength incident on the sample, $\alpha$ shall be the absorption coefficient, and $t$ shall have a sample thickness. The dependency of the absorption coefficient on the deposited $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ films analyzed in this study with wavelengths and at various concentrations. $\text{Pb}_{0.5}\text{Cd}_{0.5}\text{S}$ and $\text{Pb}_{0.75}\text{Cd}_{0.25}\text{S}$ is a large coefficient and CdS a low coefficient of absorption as opposed to the transmittance. The absorption coefficient values obtained of PbS and CdS are in agreement with values published by Yasmeen[29], Srinivasan [30] and Sachin H. Dhawankar [25].

The optical band gap ($E_g$) was known as a statistical transfer from the surface of the valence to the conductive band [27]. Figure (6) explains the plot of ($\alpha h\nu$) based on incident radiation energy. The thin films energy differences are rising with the rise in CdS concentration. The results of energy gap obtained are in agreement with the results found by Sachin H. Dhawankar et.al[25], Isaac Nkrumah et.al [22], F. Taghizadeh Chari [20], and Ashwini B. Rohom et.al [31].

The Hall Effect of the $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ film at various concentrations is utilized to calculate the electrical mobility, carrier concentration, electrical conductivity, Hall coefficient and type of carriers. Table (4) displays that Hall coefficient (P-type) of all samples is a positive and the CdS is show the largest carrier concentration, the increase of charging carrier is mainly due to the decrease of the potential barrier. In comparison, the electric mobility of hall $\mu_H$ is concluded from CdS is the smallest. The decrease in mobility is related to the opposite relationship of $\mu_H$ to $N_H$ which is characteristic of other polycrystalline thin films despite of the possible grain borders barriers. These results suggest an improvement in electric conductivity in PbS Scattering effects on the resistivity and structure depending upon; (1) decreases significantly the peak of XRD intensities and causes decreased crystallization and dislocation, imperfections, crystal defects and (2) decrease of crystallites, which improvement grain boundary and scattering that causes resistivity. The discontinuity of grain contacts may be due to low conductivity. This result almost corresponds to F. Taghizadeh Chari et.al[20], A.M. P`erez Gonz`alez et.al [32] and Mosiori et.al [33].

4. Conclusions:

Thin films of CdS, PbS, and $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ with 0, 0.25, 0.5, 0.75, and 1 at.wt % have been prepared by chemical co-precipitation method and PLD method. The XRD analysis in thin films is shows that PbS has a cubic structure while CdS has cubic and hexagonal structure and reveals that they are free from impurities. $\text{Pb}_{0.25}\text{Cd}_{0.75}\text{S}$, $\text{Pb}_{0.5}\text{Cd}_{0.5}\text{S}$ and $\text{Pb}_{0.75}\text{Cd}_{0.25}\text{S}$ have cubic structure. The 3D and 2D AFM pictures demonstrate in thin films the spherical shapes for all the samples studied and also show a non-compact surface which is not smooth and this increases the absorbance for the prepared samples. From measurement of the optical energy gap for $\text{Pb}_{1-x}\text{Cd}_x\text{S}$ nanoparticles, it has been found that: the optical transitions are direct transitions and the $\text{Pb}_{0.5}\text{Cd}_{0.5}\text{S}$ and $\text{Pb}_{0.75}\text{Cd}_{0.25}\text{S}$ are the best samples which may be used in the solar cells with high performance. SEM images of prepared samples display thin films of various diameters with spherical shape and show uniformly homogeneous surface which confirms the results of XRD and AFM. The Hall coefficient (P-type) of all sample, CdS is the largest carrier concentration, mobility of hall $N_H$ is concluded from CdS is the smallest.
5. References:

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Figure 1. XRD patterns of Pb$_{1-x}$Cd$_x$S thin films prepared with value of $x$ = (0, 0.25, 0.5, 0.75 and 1) wt%.
Table 1. The structural parameters of Pb$_{1-x}$Cd$_x$S thin films.

| Sample          | 2θ (Deg.) | FWHM (Deg.) | d$_{hkl}$ Exp. (Å) | C.S (nm) | d$_{hkl}$ Std. (Å) | hkl  | Phase         | card No.        |
|-----------------|----------|-------------|--------------------|---------|--------------------|------|---------------|-----------------|
| PbS             | 26.0216  | 0.3605      | 3.4215             | 22.6    | 3.4246             | (111)| Cub.PbS       | 96-901-3403     |
|                 | 30.2043  | 0.3966      | 2.9565             | 20.8    | 2.9657             | (200)| Cub.PbS       | 96-901-3403     |
|                 | 43.7620  | 0.3548      | 2.0669             | 24.1    | 2.0971             | (220)| Cub.PbS       | 96-901-3403     |
|                 | 51.9471  | 0.7571      | 1.7589             | 11.7    | 1.7884             | (311)| Cub.PbS       | 96-901-3403     |
| Pb$_{0.75}$Cd$_{0.25}$S | 26.0553  | 0.6130      | 3.4172             | 13.3    | 3.4246             | (111)| Cub.PbS       | 96-901-3403     |
|                 | 30.3462  | 0.8654      | 2.9430             | 9.5     | 2.9657             | (200)| Cub.PbS       | 96-901-3403     |
|                 | 43.3630  | 0.8293      | 2.0850             | 10.3    | 2.0971             | (220)| Cub.PbS       | 96-901-3403     |
| Pb$_{0.50}$Cd$_{0.50}$S | 26.0938  | 0.9096      | 3.4122             | 9.0     | 3.4246             | (111)| Cub.PbS       | 96-901-3403     |
|                 | 30.3486  | 0.9538      | 2.9428             | 8.6     | 2.9657             | (200)| Cub.PbS       | 96-901-3403     |
| Pb$_{0.25}$Cd$_{0.75}$S | 26.3904  | 0.7211      | 3.3745             | 11.3    | 3.4246             | (111)| Cub.PbS       | 96-901-3403     |
|                 | 30.7173  | 0.6851      | 2.9083             | 12.0    | 2.9657             | (200)| Cub.PbS       | 96-901-3403     |
| Cds             | 25.0120  | 0.2524      | 3.5573             | 32.2    | 3.5808             | (100)| Hex.CdS       | 96-900-8863     |
|                 | 26.6707  | 0.3246      | 3.3397             | 25.2    | 3.3590             | (111)| Cub.CdS       | 96-900-8840     |
|                 | 28.3654  | 0.3245      | 3.1439             | 25.3    | 3.3745             | (002)| Hex.CdS       | 96-900-8863     |
|                 | 43.8702  | 0.2884      | 2.0621             | 29.7    | 2.0570             | (220)| Cub.CdS       | 96-900-8840     |
|                 | 48.0529  | 0.3605      | 1.8919             | 24.1    | 2.0674             | (110)| Hex.CdS       | 96-900-8863     |
|                 | 52.0913  | 0.3245      | 1.7543             | 27.2    | 1.7542             | (311)| Cub.CdS       | 96-900-8840     |

Table 2. Grain size and average roughness of the Pb$_{1-x}$Cd$_x$S thin films.

| Sample          | Grain size(nm) | Roughness average(nm) |
|-----------------|----------------|-----------------------|
| PbS             | 83.80          | 11.2                  |
| Pb$_{0.75}$Cd$_{0.25}$S | 83.12          | 5.38                  |
| Pb$_{0.50}$Cd$_{0.50}$S | 77.42          | 5                     |
| Pb$_{0.25}$Cd$_{0.75}$S | 72.51          | 5.09                  |
| Cds             | 53.95          | 4.39                  |
PbS

\[ \text{PbS} \]

\[ \text{Pb}_{0.75}\text{Cd}_{0.25}\text{S} \]
Pb$_{0.5}$Cd$_{0.5}$S

Pb$_{0.25}$Cd$_{0.75}$S
CdS
Figure 2. AFM images in 2D, 3D and grain size distribution of the Pb$_{1-x}$Cd$_x$S thin films.

Figure 3. SEM images of the Pb$_{1-x}$Cd$_x$S thin films.
Figure 4. Optical transmission for the Pb$_{1-x}$Cd$_x$S thin films.

Figure 5. Absorption spectra of Pb$_{1-x}$Cd$_x$S thin films.
Figure 6. A plots of $(\alpha h\nu)^2$ verses photon energy ($h\nu$) of Pb$_{1-x}$Cd$_x$S thin films.

Table 3. List of the optical energy gaps of Pb$_{1-x}$Cd$_x$S thin films.

| Sample       | $E_g$(eV) |
|--------------|-----------|
| PbS          | 1.80      |
| Pb$_{0.75}$Cd$_{0.25}$S | 2.00      |
| Pb$_{0.5}$Cd$_{0.5}$S    | 2.20      |
| Pb$_{0.25}$Cd$_{0.75}$S  | 2.35      |
| CdS          | 2.90      |

Table 4. Hall effect parameters of Pb$_{1-x}$Cd$_x$S thin films with value of $x$=(0, 0.25, 0.5, 0.75and 1) at. wt. %.

| Samples       | $N_H$ $(cm^{-3})$ | $R_H$ $(cm^2.\text{C}^{-1})$ | $\sigma$ $(\Omega.\text{cm})^{-1}$ | $\mu_H$ $(\text{cm}^2.\text{V}^{-1}.\text{S}^{-1})$ | Type |
|---------------|-------------------|-----------------------------|---------------------------------|---------------------------------|-------|
| PbS           | 3.18E+14          | 1.97E+04                   | 1.90E+01                        | 3.74E+05                         | p     |
| Pb$_{0.75}$Cd$_{0.25}$S | 1.58E+14          | 3.95E+04                   | 4.77E-01                        | 1.88E+04                         | p     |
| Pb$_{0.5}$Cd$_{0.5}$S    | 1.39E+14          | 4.50E+04                   | 2.41E-01                        | 1.09E+04                         | p     |
| Pb$_{0.25}$Cd$_{0.75}$S  | 1.54E+14          | 4.05E+04                   | 1.62E-01                        | 6.54E+03                         | p     |
| CdS           | 5.06E+15          | 1.23E+03                   | 5.80E+00                        | 7.15E+03                         | p     |