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CdS thin films Fabricated by a simplified Spray technique from different Substrate Temperatures – Structural, Morphological, Optical and Electrical analysis

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Abstract

A comprehensive study of the effect of substrate temperature on the physical properties of CdS thin films fabricated by a simplified spray technique using perfume atomizer is reported. The films were deposited on glass substrates using cadmium chloride precursor as Cd source and thiourea as S source respectively at different substrate temperatures. X-ray diffraction, scanning electron microscopy, UV-Vis-NIR spectroscopy and Energy dispersive X-ray analysis were used to characterize the films. XRD studies confirm the polycrystalline nature of the deposited films with hexagonal structure. The structural parameters such as lattice constants, crystallite size, dislocation density and number of crystallites per unit area have been evaluated. The composition of the various constituents in CdS thin films has been determined by energy dispersive X-ray analysis (EDAX). Films coated at 425ºC were found to be highly stoichiometric. Electrical resistivity decreases with increase in substrate temperature and a minimum electrical resistivity of 0.1057 x 10^1 ohm-m was obtained for the film coated at 425ºC. The high transmittance and low resistivity values obtained for all the films makes them suitable as efficient window layer for solar cell applications.

Keywords: Semiconducting thin films, band gap energy, X-ray diffraction, optical studies, electrical resistivity.

Introduction

Cadmium sulfide (CdS) belongs to II - VI compound semiconductor whose extremely wide range of physical and chemical properties makes them important material both for technological and industrial applications, especially in optoelectronic devices such as solar cells[1], photo transistors[2], transparent electrodes[3] and solid state gas sensors[4]. CdS has been used as an efficient window layer for the fabrication of supersaturates type solar cell structure due to its high transmittivity and low resistivity[5]. CdS present a high transmittance in the visible and a high reflectance in the infrared region (E_g = 2.42 – 2.45 eV). For solar cell applications, CdS films need to have a suitable conductivity (> 10^{16} carriers/cm^3), and adequate thickness to allow high transmission and good uniformity to avoid electrical short- circuit effects. Being a direct transmission semiconductor, it is used as an antireflecting coating for heterojunction solar cells[6]. It is known to be the most convenient partner for heterojunction realization of p- type CdTe and CuInSe solar cells[7]. Several techniques have been used to prepare CdS thin films namely: sputtering[8], thermal evaporation[9], electrodeposition[10], spray pyrolysis[11] and chemical bath deposition[12]. For CdS films deposition, it has already attracted great attention to develop synthesis techniques with advantages of green, low cost and easy fabrication on a large scale. Spray pyrolysis is a low cost technique and can deposit high quality CdS films on large area substrates. Spray technique facilitates better orientation of crystallites with improved grain structure. In this work a simplified spray technique using perfume atomizer is adopted to fabricate CdS thin films. It is observed that the spray deposition parameters at which CdS thin films are grown play a vital role in deciding their uniformity and spectral response. Hence, an attempt has been made to prepare CdS thin films on glass substrates based on one of the spray parameter viz. substrate temperature. The effects of substrate temperature on structural, morphological, optical and electrical properties of the CdS films were investigated in order to study the film growth mechanism.

Methodology

CdS thin films used in this study were deposited by the simplified spray technique using perfume atomizer. The spraying solution is formed with 50 ml aqueous solution of 0.1M $CdCl_2$ used as source of Cd and 0.1M thiourea used as source of S. The substrate temperature was varied between 275º C to 425ºC in steps of 50ºC. Before coating, the substrates were ultrasonically cleaned with 30% nitric acid, deionized water and ethanol for 20 min, respectively. The structure, crystallinity and phase of the CdS thin films were determined with a diffractometer “XPERT-PRO” model, using CuKα radiation (λ = 1.5406 Å) with 2θ ranging from 20º to 80º. The surface morphology of the films was characterized by scanning electron microscope HITACHI model S-3000H. In order to determine the band gap energy of CdS in thin film form, the optical transmission studies were carried out in the wavelength range of

300 – 1200 nm, using PerkinElmer UV-vis-NIR double beam spectrophotometer (LAMBDA – 35). Electrical resistivity of the films was determined by two point probe method.

The crystallite size 'D' can be simply determined using the Scherrer formula:

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (1)$$

where λ is the wavelength of X-ray, β is the full width at half maximum (FWHM in radians) of the peak corrected for instrumental broadening, and θ is the Bragg angle, k is the Scherrer constant, equal to 0.9.

The absorption coefficient 'α' of the CdS films was calculated using the transmittance (T) value and thickness 't' using the relation: $\alpha = \frac{-\ln(T)}{t}$ (2)

The absorption coefficient can be expressed by Urbach relation:

$$\alpha = \frac{A\left(h\gamma - E_g\right)^n}{h\gamma} \quad (3)$$

where A is a constant, E_g is the energy gap, γ is the frequency of the incident radiation and h is Planck's constant. The exponent n is 1/2 for direct allowed transitions. The energy gaps of the films coated at various substrate temperatures have been determined by extrapolating the linear portion of the plots of $(\alpha h\gamma)^2$ against hγ to the energy axis.

Optical constants, refractive index 'n' and packing density 'p' were calculated using the expressions[13, 14]:

$$n = \frac{1+R}{1-R} + \sqrt{\frac{4R}{(1-R)^2} - k^2} \quad (4)$$

$$n^2 = \frac{\left(1-p\right)n_v^{\,4} + \left(1+p\right)n_v n_s^{\,2}}{\left(1+p\right)n_v^{\,2} + (1-p)n_s^{\,2}} \quad (5)$$

where k is the extinction coefficient given by the expression, $k = \frac{\alpha\lambda}{4\pi}$, R is the reflectance of the films, n_s is the substrate refractive index and n_v is the void refractive index (equal to 1 for air).

The electrical properties of the films were calculated according to the equation:

$$\rho = R \times W \times \frac{t}{L} \quad (6)$$

where R is the resistance of the film, W is the length of the film, L is the distance between the two aluminium conducting strips and t is the thickness of the film.

Results and Discussion

Film formation: When aqueous solution containing the precursor salts of 0.1M of cadmium chloride and thiourea was atomized over hot glass substrates using perfume atomizer, pyrolytic decomposition takes place resulting in the formation of yellow colored CdS thin films. Figure-1 shows the variation of the weight deposited in mg/cm^2 and thickness of the CdS films coated at different substrate temperatures. As seen the film deposition rate decreases i.e. the amount of mass being deposited decreases with increase in substrate temperature, which reflects on the film thickness. In CdS thin films, the deposition rate depends on the quantity of reactive species i.e. Cd and S in one hand and the reaction kinetics occurring at the substrate surface on the other hand. The S/Cd molar ratio used to fabricate CdS films in this work is maintained as 1 and there are plenty of earlier reports[15] of obtaining stoichiometric CdS films with this molar ratio. Thus it can be concluded that only the reaction kinetics occurring at the substrate surface (substrate temperature in this case) alone contributes to the growth rate of the CdS films fabricated by this simplified spray technique.

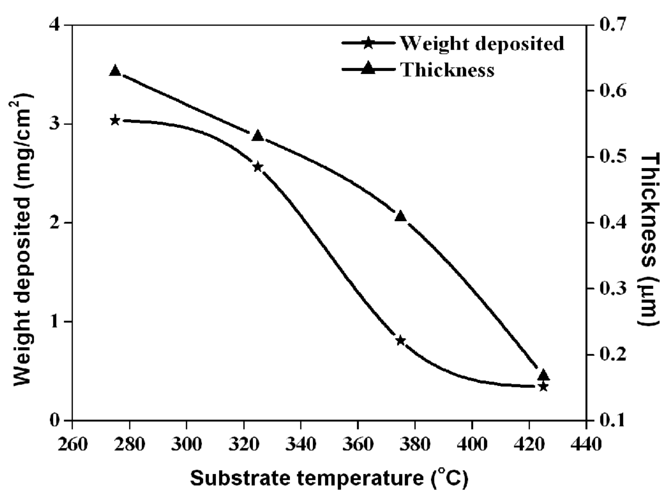

Figure-1
Variation of weight deposited and thickness of the CdS films

XRD results: The XRD spectra of CdS films are shown in figure-2. Regardless of the substrate temperature, all films were polycrystalline in nature with hexagonal structure. The diffraction peaks at 2θ positions of 24ºC, 26.5ºC and 28.2ºC can be assigned to the planes (1 0 0), (0 0 2) and (1 0 1) planes of the hexagonal wurtzite structure (JCPDS Card No. 77-2306). Besides these, peaks corresponding to (1 0 2), (1 1 0), (1 0 3) and (1 1 2) planes were observed in all the samples. Figure.3 shows the variation of the preferential orientation factor f(h k l) of the peaks (1 0 0) and (0 0 2) planes with substrate temperature.

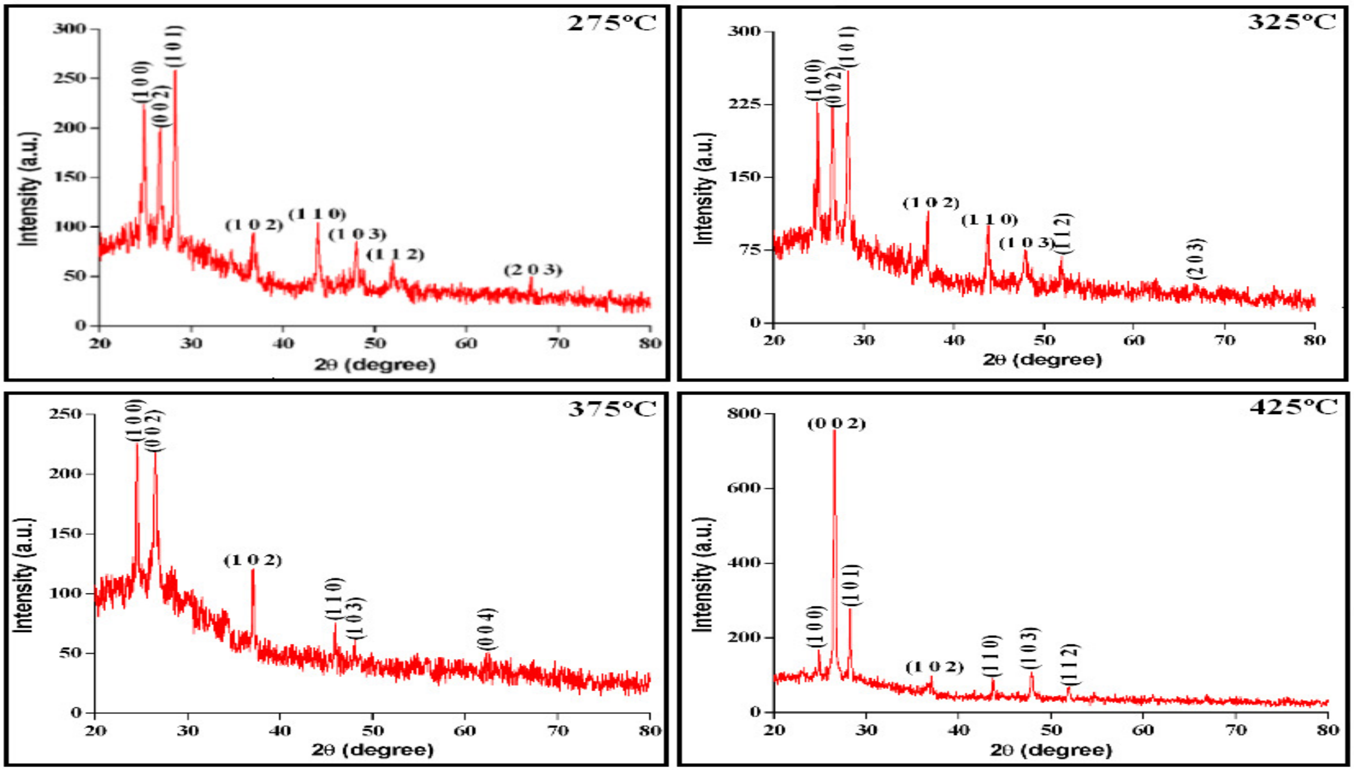

Figure-2
XRD spectra of the CdS films coated at different substrate temperatures

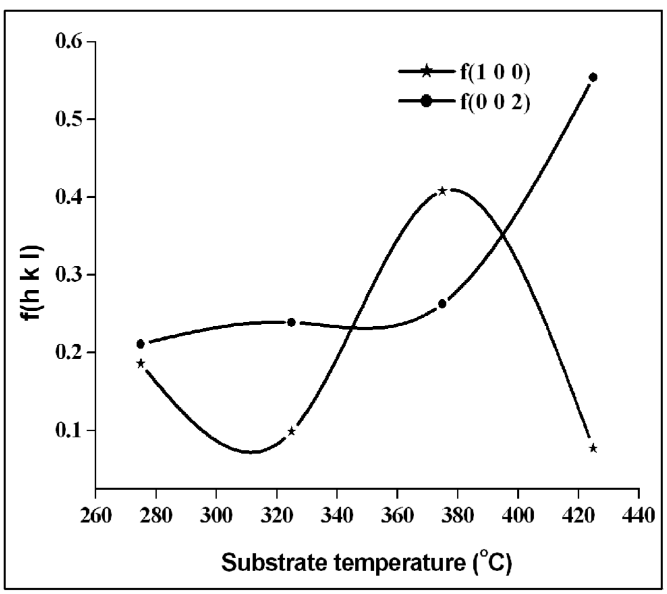

Figure-3
Variation of f(1 0 0) and f(0 0 2) planes with substrate temperature

The pure hexagonal structure obtained confirms that the CdS films formed in this work takes place of ion-by-ion growth mechanism which is strongly supported from the SEM images obtained (figure 4). It is worth noting that CdS thin film is the most important partner to the realization of the CdTe and CuInSe heterojunction based solar cells. For this application, hexagonal structured films are preferable[16]. Despite that hexagonal CdS have the higher lattice mismatch with $CuInSe_2$ (1.2 %) by comparison to that of cubic CdS (0.7 %). Hexagonal structure is mostly recommended due to its higher stability regarding the cubic one[17]. The lattice parameters 'a' and 'c' of the unit cell were evaluated according to the relation[18]:

$$\frac{1}{d^2} = \frac{4}{3}\frac{h^2 + hk + k^2}{a^2} + \frac{l^2}{c^2} \quad (8)$$

where d is the interplanar spacing. The calculated lattice parameter values of (0 0 2) plane are given in table-1.

The lattice parameter values of all the films show a slight deviation from the standard values which might be due to the strain introduced in the samples due to excess Cd interstitials or S vacancies present in the samples as evident from EDS analysis. The calculated values of microstructural parameters such as strain (ε), dislocation density (δ) and the number of crystallites (N) of the (0 0 2) plane are presented in Table-2.The crystallite size 'D' of the film increases above 325ºC and attains the highest value, showing that increased substrate temperature promoted crystallite growth and improvement in the crystallinity of the CdS films considerably. With increased substrate temperature, the density of the nucleation centers decreases and under these circumstances, a small number of centers start to grow, resulting in larger grains. Similar results have been reported by Ashour[19], for spray pyrolysed CdS thin films. Defects such as strain and dislocation density has the minimum value for the film coated at 425ºC which may be due to the improvement in the crystallinity and stoichiometry of the film coated at that temperature as evident from XRD pattern and elemental analysis.

Morphological studies: Scanning electron micrographs of the CdS films deposited by the simplified spray technique at different substrate temperatures are shown in figure-4. Films morphologies show a significant change with increase in substrate temperature. At 275ºC, the agglomeration of crystallites appears to be substantial, thereby showing a remarkable increase in the size of the grains. At 325ºC, the particles deposited on the surface were needle-like and the film beneath the particles appears to be relatively dense. Besides needle shaped grains, small spherical grains were found distributed throughout the surface. With further increase in substrate temperature to 375ºC, nanosized needle shaped grains were found to be spread throughout the film surface without any existence of small spherical grains. At 425ºC, the surface morphology of the film changes from less crystallized and porous films to more crystallized dense films. No needle shaped grains were observed and the substrate is well covered with spherical grains confirming the fact that the growth mechanism takes place of ion-by-ion mechanism, and this fact is strongly supported by the pure hexagonal structure obtained as evident from XRD analysis. This fact was well supported from the variation of preferential orientation factors f(1 0 0) and f(0 0 2) with substrate temperatures as shown in Figure-3.

Elemental analysis: The EDS spectra of the CdS films fabricated by the simplified spray technique at different substrates temperatures are given in figure-5, which show that all the films contain the elements Cd and S as expected.

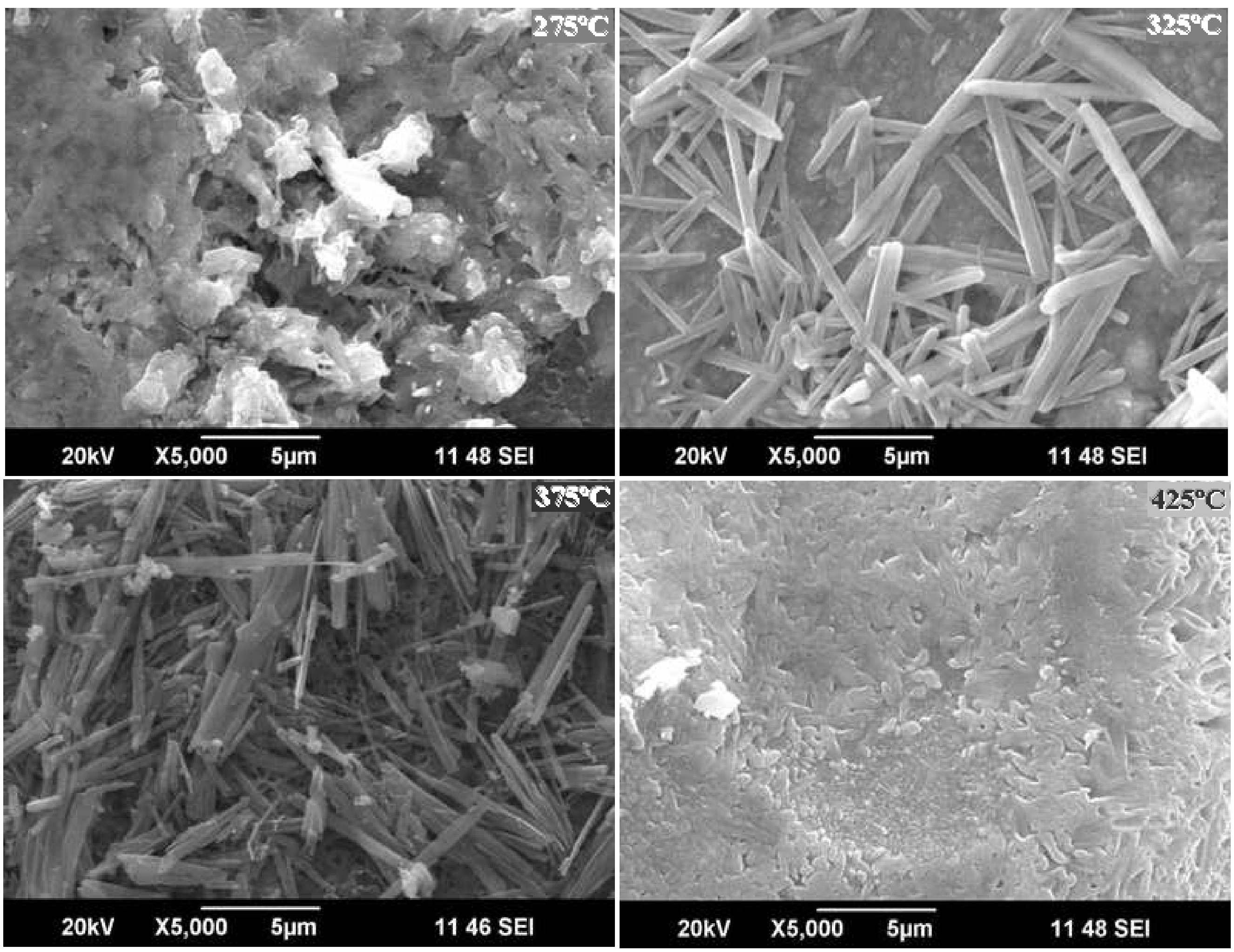

Figure-4
SEM images of CdS films

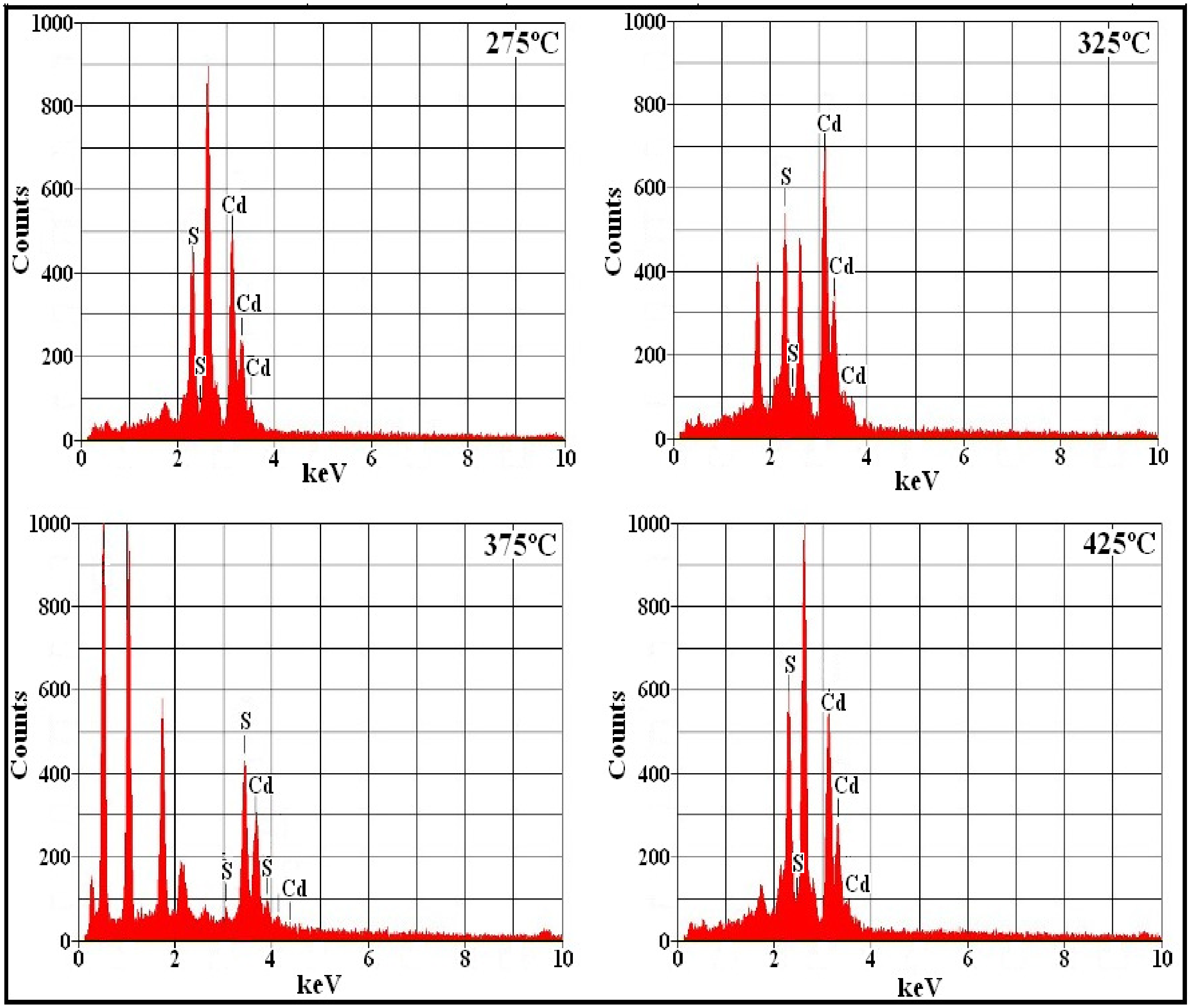

Figure-5
EDS spectra of CdS films fabricated at different substrate temperatures

The weight ratio and atomic ratio compositions of sulfur and cadmium in the films are presented in table-3.

The S/Cd ratio increases with substrate temperature and attains a maximum value of 1.01 for the film coated at 425ºC, indicating the perfect stoichiometric nature of the film coated at that temperature. The decreased value of S/Cd ratio obtained for the films coated at lower substrate temperatures indicate that the films are marginally deficient with sulfur or with surplus cadmium. Usually sulfur deficiency is a typical phenomenon for chemical bath deposited CdS films and it is a rare case for spray deposited films that too coated with equi molar concentrations of cadmium and sulfur source precursors (cadmium chloride and thiourea). Among the various cadmium source precursors, cadmium chloride has a high stability constant of equal to 2.93. At low substrate temperatures less than 400ºC, the release of Cd ions from the precursor salt with high stability constant takes place at a slow rate, which results in delayed growth mechanism of CdS, and the films are composed with excess unbounded Cd ions or deficient sulfur ions. On the other hand at higher substrate temperatures > 400ºC, the release of Cd ions from the precursor salt with high stability constant takes place at a rapid rate which improves the growth mechanism of CdS and the presence of excess unbounded Cd ions is minimized and films with perfect stoichiometry is obtained with increased carrier concentration which results in the decreased resistivity of the film coated at 425ºC. This fact was well supported by the low resistivity value obtained for the CdS film coated at 425ºC as evident from the electrical studies.

Table 1
Lattice parameters of the CdS films

Substrate temperature	a (Å)		c (Å)	
	Calculated	Standard*	Calculated	Standard*
275	4.1083	4.136	6.7089	6.713
325	4.1076		6.7078	
375	4.111		6.7134	
425	4.1083		6.7089	

* JCPDS Card No. 77-2306

Table 2
Microstructural parameters of the CdS films

Substrate temperature	Microstructural parameters			
	D (nm)	Strain $\varepsilon \times 10^{-3}$	Dislocation density $\delta \times 10^{15}$ lines/m^2	Number of crystallites $N \times 10^{16}$
275	27.19	5.548	1.35	3.129
325	18.97	7.95	2.78	7.776
375	27.20	5.65	1.352	20.3
425	40.79	3.698	0.601	24.7

Table 3
Results of quantitative elemental analysis

Substrate temperature	Element				S/Cd
	Cd wt%	S wt%	Cd at%	S at%	Compositional Ratio(atomic)
275	84.22	15.78	60.36	39.64	0.66
325	82.64	17.36	56.44	43.56	0.77
375	81.4	18.6	55.52	44.48	0.8
425	77.66	22.34	49.79	50.21	1.01

Optical properties: Figure-6 shows the optical transmission spectra of all the four films. All the films have high transmission, with the transmission of the film coated at 425ºC being the better than the other three films. This was actually expected, since the thickness of the film coated at 425ºC has the lowest value compared to the other three films. This fact was well supported from the SEM micrographs shown in figure-4, which showed that the film coated at 425ºC have much smoother surface than the other three films. The surface roughness due to coverage by crystallite overgrowth causes light scattering, which in turn lowers the transmission for the films coated at temperatures less than 425ºC. The higher transmittance obtained for the film coated at 425º indicates lower defect density and better electrical properties because absorption of light in the longer wavelength region (. 500 nm) is usually caused by crystalline defects such as grain boundaries and dislocations[20]. Another observation about these transmission spectra is that all the films share the same absorption edge. A red shift (towards longer wavelengths) for the films coated at higher temperatures (> 350ºC) and a blue shift in the case of the CdS films coated at lower temperatures (< 350ºC) were observed. This reflected on their corresponding optical band gap, as shown in figure-7.The optical band gap of the CdS film coated at 275ºC is the highest (2.55eV) and it decreases with increases in substrate temperature and attains a minimum value of 2.38eV for the film coated at 425ºC. An increase in the crystallite size for the film coated at 425ºC (table-3) is the reason for the decreased band gap obtained at that temperature. The obtained value of band gaps exactly matches with those obtained by Faraj et.al.[21]. Figure-8 shows the specular reflectance of all the films. The overall reflectance of all the samples is very low. The high transmittance and low reflectance values obtained makes the films suitable as antireflecting coating materials for thin film solar cells. The refractive index plays an important role in the search for optical materials, being a significant factor in optical communication and in designing devices for spectral dispersion. Therefore, it is important to determine refractive index of the thin film. The variation of the refractive index of the CdS films coated at different substrate temperatures is shown in Figure-9. It was seen that the refractive index decreased from 2.261 to 2.027 with increasing substrate temperature from 275ºC to 425ºC. The decrease in the refractive index is associated with the fundamental band gap absorption. From the refractive index data, the packing density (p) which is defined as the ratio of the solid volume to the total volume of the film is estimated. Figure-10 shows the variation of refractive index and packing density of the CdS films with substrate temperature. The high value of 'n' obtained for the CdS film coated at 275ºC might be due to the high value of packing density obtained. High refractive index corresponds to high surface roughness and lower grain size which tends to decrease the effective mean free path through increased surface scattering [22] and this fact strongly favors the reason for the reduction of its transparency.

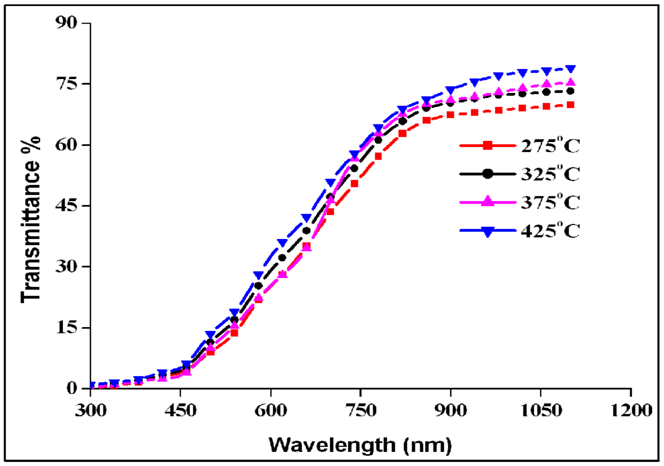

Figure-6
Optical transmission spectra of the CdS films

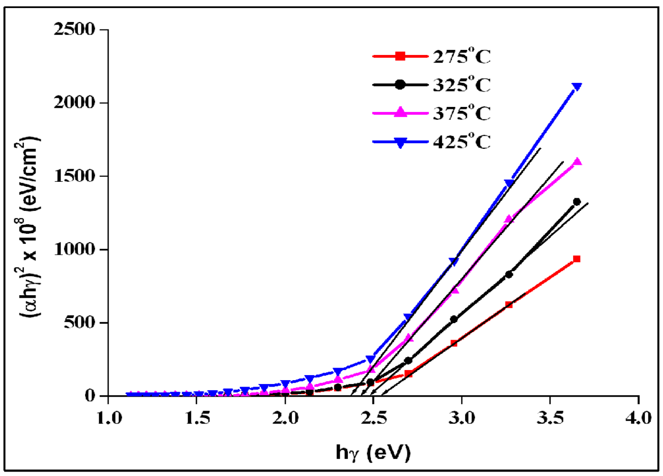

Figure-7
$(\alpha h\gamma)^2$ versus hγ graph of the CdS films

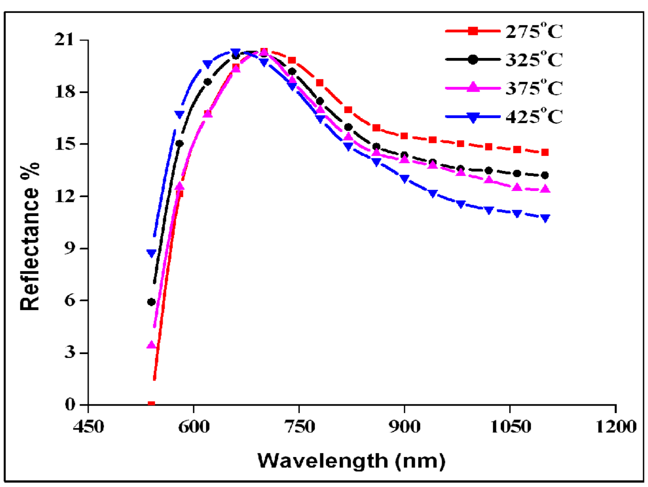

Figure-8
Reflectance spectra of the CdS films

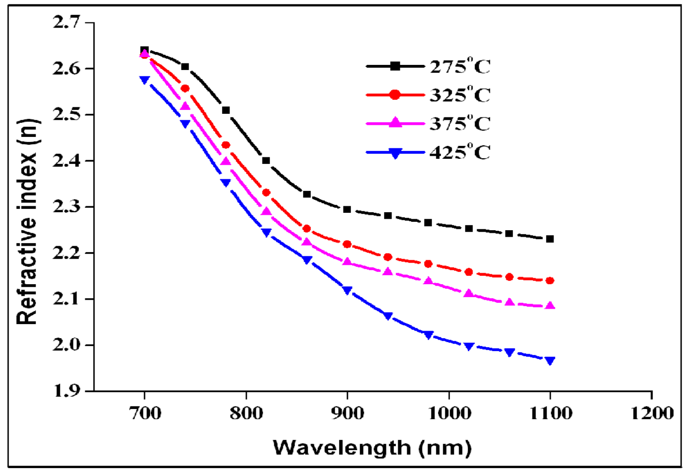

Figure-9
Variation of refractive index with wavelength of CdS films

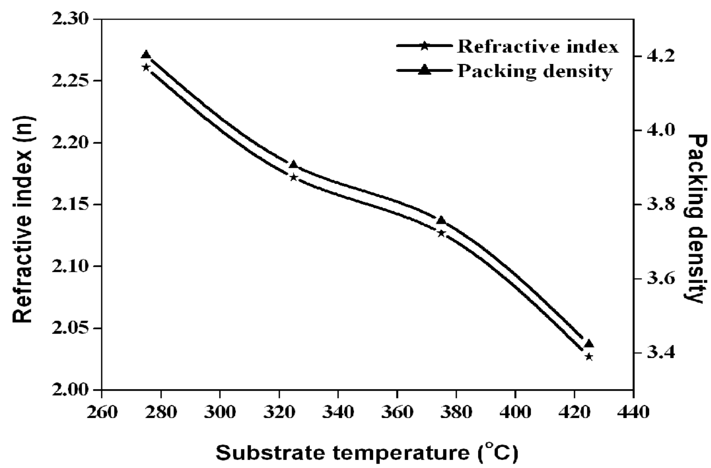

Figure-10
Variation of refractive index and packing density with substrate temperature

Electrical studies: The variation of electrical resistivity of CdS films prepared at different substrate temperatures by the simplified spray technique is shown in figure-11. For the film coated at 275ºC, the resistivity is found to be 16.33 x 10^1 Ω-m. With increase in substrate temperature, the resistivity decreases and attains a minimum value of 0.1057 x 10^1 Ω-m for the film coated at 425ºC. The resistivity values obtained in this work are in agreement with what has been reported in the literature for polycrystalline CdS thin films[22]. The decreased value of resistivity obtained for the film coated at 425ºC may be attributed to the growth in crystallite size and the improvement in film stoichiometry, as is indicated by the XRD patterns and from elemental analysis. The crystallite size of the CdS film coated at 425ºC (40.79 nm) is much larger than the films coated at other temperatures which means that the film coated at 425ºC has higher mobility which improves its conductivity.

PL studies: The analysis of photoluminescence (PL) spectroscopy of CdS films room temperature reveals various peaks as shown in figure-12. Photoluminescence spectroscopy can be used to determine the band gap of semiconductors since the most common radiative transition in the semiconductor occurs between states at the bottom of the conduction band and the top of the valence band[23]. Irrespective of the substrate temperature, all the films show a very high intense peak at 494 nm (2.51 eV) and small intense peaks at 520 nm (2.4 eV) and at 594 nm (2.09 eV) respectively. PL band appearing in the range of 2.18 – 2.54 eV are called green bands; band appearing between 2.07 and 2.18 eV are typically referred to as yellow bands; the orange band is located between 2 and 2.07 eV, and luminescence observed around 1.54 – 2 eV is called the infrared / red band. The radiative transition observed at 594 nm is discussed in the literature as a donor-acceptor pairs (DAP)[24]. A donor level of 0.21 eV below the conduction band is suggested to be related to a cadmium interstitial Cd_i or to a sulfur vacancy V_S [24]. The acceptor level located 0.29 – 0.30 eV above the valence band [25] is believed to originate from an impurity rather than from a native defect in CdS. The recombination of a free hole with an electron from native donor level, Cd_i or V_S, 0.11 eV below the conduction band leads to a green emission observed at 2.4 eV [26]. Several authors observe the green band for polycrystalline CdS films [27]. In our study, luminescence in the green band (2.51 eV) is observed in all the samples. The green band described for CdS single crystals is divided into a series of equally spaced lines. Lines related to excitons bound to neutral acceptors or donors are found to appear at 2.536 eV and 2.547 eV [28]. Thus the PL peak observed at 494 nm (2.51 eV) may be related to excitons bound to neutral acceptors or donors.

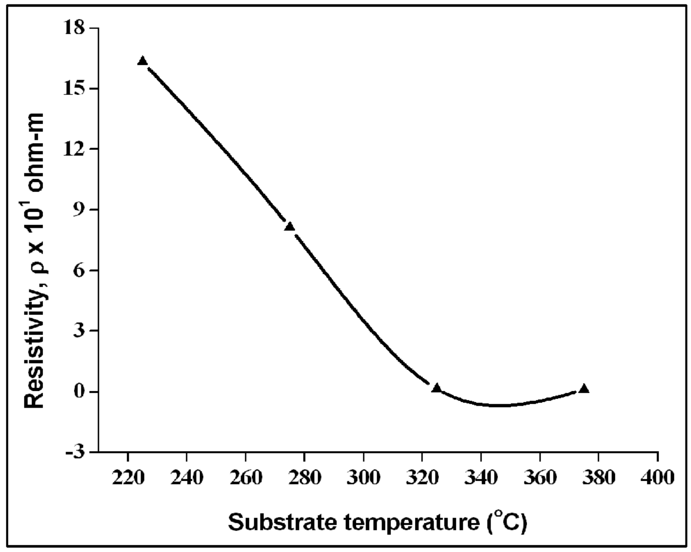

Figure-11
Electrical resistivity variation of CdS films with substrate temperature

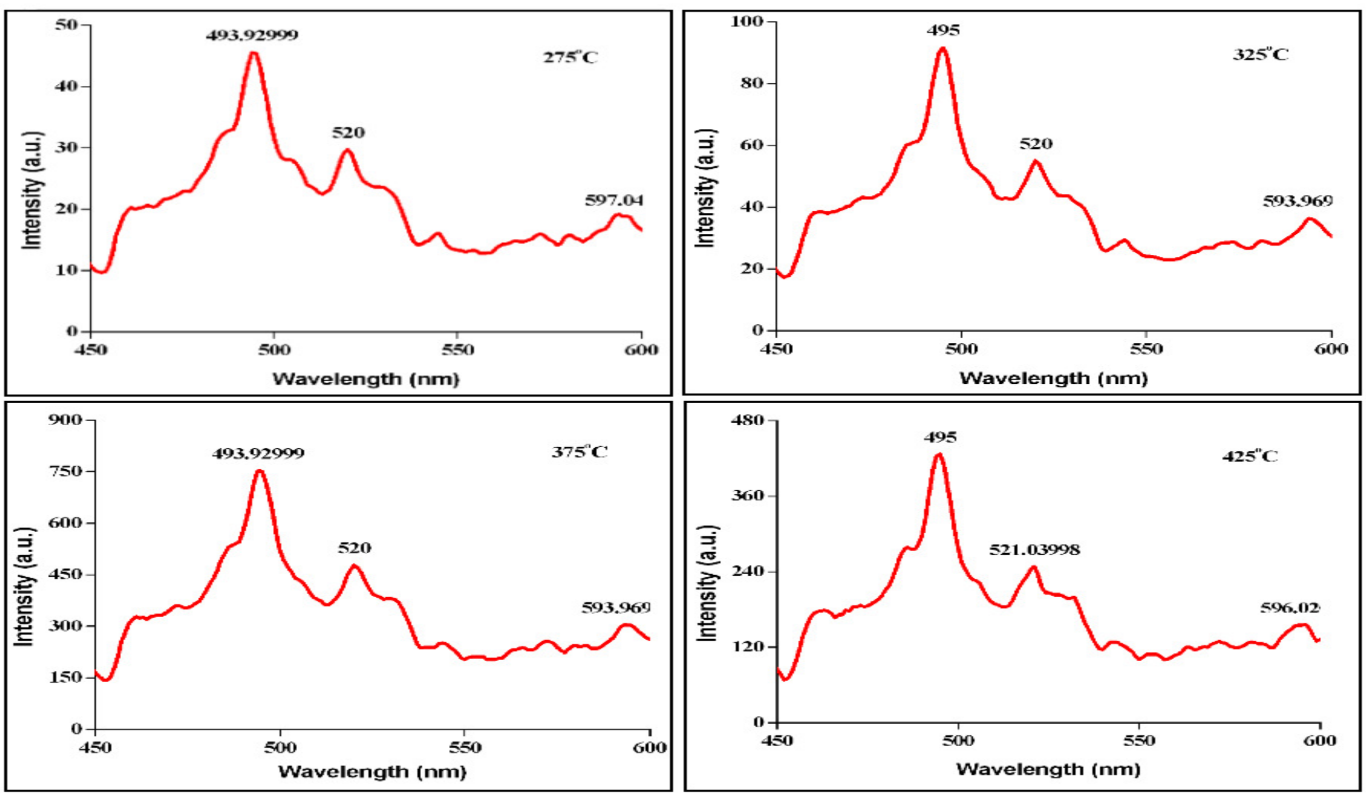

Figure-12
PL spectra of the CdS films coated at different substrate temperatures

Conclusion

A comprehensive study of the influence of substrate temperature on electrical/optical properties as well as thickness, structure, surface morphology and stoichiometry of spray deposited CdS thin films is presented. Film thickness decreases with increase in substrate temperature. All the films were found to be hexagonal, regardless of the substrate temperature. Crystallite size increases above 325ºC and attains a maximum value of 40.79 nm for the film coated at 425ºC. EDS analysis showed that the film coated at 425ºC is highly stoichiometric (S:Cd ratio = 1:1). A red shift in band gap for the films coated at higher temperatures (> 350ºC) and a blue shift for the films coated at lower temperatures (< 350ºC) were observed. A minimum resistivity of 0.1057 x 10^1 ohm-m was obtained for the film coated at 425ºC. The high transmittance and low resistivity value obtained for the film coated at 425ºC makes them convenient for optoelectronic applications.

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