Influence of Substrate Material on Structure Formation and Optical Properties of CdS Thin Films by Chemical Spray Pyrolysis

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Abstract

Cadmium sulfide (CdS) thin films have been deposited on a variety of substrates by using homemade chemical spray technique (CSP). A study of the effect of substrate material on the structure and optical properties of the fabricated films is reported. X-ray diffraction studies confirm the polycrystalline nature of the films with hexagonal structure and a preferential orientation along the (002) plane. The surface morphological studies revealed the nanocrystalline grains, and all the deposited films have uniform well covered grain size on the surface of the substrates. The compositional analyses showed highly stoichiometric, and the S/Cd atomic ratio is close to one. The optical studies showed the average transmittance of the films deposited on different substrates in the visible and near infrared regions is about (65 to 80) %, and band gap varied from (2.432 to 2.444) eV. The obtained results indicated that the substrate nature has a strong factor influencing on the formation and optical properties of CdS films.

Keywords: Cadmium supplied: substrate material; spray pyrolysis, structural formation; optical properties.

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تأثير مادة القاعدة على الخواص التركيبية و البصرية لشبه الموصل كبريتيد الكادميوم المحضرة بطريقة الرش الكيمياوي الحراري

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الخلاصة

تم ترسيب أغشية رقيقة لشبه الموصل كبريتيد الكادميوم (CdS) على قواعد مختلفة باستخدام تقنية الرش الكيمياويالحراري المصنع محليا. تم دراسة تأثيرمادة القاعدة على الخواص التركيبية والبصرية للافلام المحضرة. دراسة حيود الاشعة السينية اكدت طبيعة المتعدد البلورات للافلام مع التركيب السداسي وبالاتجاه المفضل للمستوى (002). دراسات البنية االسطحية كشفت الى تكون حبيبات نانوية ، وان الحبيبات تغطي بشكل منتظم سطح الافلام المرسبة على القواعد. التحليلات التركيبيه بينت اتحاد العناصر بشكل عالي ، وان النسبة الذرية للعناصر(S/Cd) قريبة من واحد. الدراسات البصرية بينت معدل النفاذية للاغشية المرسبة على قواعد مختلفة في المناطق المرئية والقريبة من التحت الحمراء هى حوالي (65-80) % ، وان فجوة الطاقة تتغير من 2.432 eV الىeV 2.444. النتائج كشفت أن طبيعة مادة القاعدة عامل مؤثر على الخواص التركيبية والبصرية لأغشية (CdS).

الكلمات المفتاحيه: كبريتيد الكادميوم ، مادة القاعدة ، الرش الحراري ، التكوين التركيبي ، الخواص البصرية.

Introduction

Cadmium sulfide (CdS) has been investigated because of its great applications in solar cells **[1]**, optical detectors **[2]** and optoelectronic devices **[3]**. Among various techniques **[4-9]**, chemical spray pyrolysis (CSP) is attractive method used to make CdS films because of its technical advantages including low cost, low deposition temperature, and convenient for large area deposition **[10,11]**. Usually, CdS thin films were deposited on the glass, and for special applications, CdS thin films were deposited on some flexible substrates such as organic materials and so on **[12]**. One of the difficulties in the growth of single crystal thin films lies in getting a suitable substrate material, every substrate has problems. Many works have been done to study the effects of different parameters on the growth and properties of the thin films

such as deposition technique, substrate temperature, stoichiometry, film thickness, etc. to achieve good quality thin films. Most of the researches on the thin films are concentrated on improve of the conductivity and the transparency of the films deposited on glass substrates by a variety of techniques.

However, little works were reported on the substrate materials effects, where the substrate is in direct contact with the film materials and that produce unwanted secondary phases and micro cracks in the texture of the films **[13-17]**. This may be attributed to the interaction of sample material with the elements in these substrates, hence, preventing the form of single phase **[18]**. Impurity diffusion from the substrate material into the thin film has to be avoided by either choosing appropriate substrate materials or introducing a suitable barrier layer. The size of grains was also found to be depending on the substrate material **[19-23]**. For example, the use of soda lime glass (SLG) as substrate material is very critical as sodium can diffuse out of SLG, in microscopic slide glass silicon or any other element in the compose of the slide can diffuse out of the slide, a native SiO_2 layer may be present on the substrate surface due to substrate preparation **[24]**. The device substrate is an integral component in the overall optimization of flexible electronics. It affects the final form factor of the device and performance and is also critical in determining process parameters and manufacturing yield. The substrate is a significant element in all aspects of the device (design, manufacturing, and performance) **[22]**. The substrate material provides mechanical support to the thin film device structure. The coefficient of thermal expansion has to match the film layer to avoid delaminating and the material has to be temperature resistant enough to endure the film device processing. In case the film is grown the substrate needs to be transparent and thermally stable. It would be interest to see the substrate effect on the structure formation and properties of thin films since the focus has been on films grown on slide glass substrates. In this work, we deposited CdS thin films on various substrates to see the effect of substrate type on the formation and optical properties of the films.

Materials and methods

CdS thin films were prepared by the deposition process on different substrate types by using spray pyrolysis technique under previous optimized experimental set-up and deposition conditions such as solution molarity, substrate temperature, film thickness etc., where all held constant during deposition process for all substrate types **[11]**. Details of the experimental set-up are given there, where it was obtained that the CdS films grown at a substrate temperature of 350 ^{o}C with precursor solution molarity 0.1M. The thickness of the films was determined by weight method, and kept constant for all the substrates for about 216 nm. The films were prepared from aqueous solution containing cadmium chloride ($CdCl_2.H_2O$) and thiourea [$CS(NH_2)_2$]. The solution was sprayed vertically onto the preheated substrate kept at a distance of 25 cm from the spray nozzle. Prior to the deposition, the (slide glass, quartz, and corning glass) substrates were cleaned by immersed in acetone and put them in ultrasonic path for 25 min., then cleaned with double distillation water. ITO substrates were immersed in a solution containing 10 % HCl and 90 % double distillation water for 1 min., and then washed with acetone. After that, the substrates were cleaned with double distillation water. The silicon substrate surfaces were cleaned using a standard Radio Corporation of America (RCA) cleaning method and dried by blowing Ar. Nitrogen (N_2) is used as a carrier gas with a pressure of 2.5 bars and the spray rate of the solution was maintained at 4 ml/min. To avoid thermal shock and excessive cooling of the substrates, the spraying time was fixed as 9 sec with the time interval between successive spraying was maintained within 10 sec to control the substrate temperature and to ensure the complete evaporation of the residue of starting material. Substrate temperature was controlled by K-type thermocouple fed to a temperature controller with an accurate of ±2 ^{o}C. The system was kept in aluminum and glass chamber, the chamber box was fitted with a fan to remove the toxic gases produced during the decomposition of the spray solution.

Various types of substrate materials were used: (i) quartz, (ii) corning glass, (iii) ITO (Indium Tin Oxide) coated glass, (iv) n-type silicon wafer, and (v) p-type silicon wafer. CdS films also deposited on slide glass substrate under the same conditions and their properties have been studied for comparison.

Characterization techniques

The structural properties of the deposited films were determined by X-ray diffraction using X-ray diffractometer (HR-XRD) system X-Pert Pro MRD model with CuKα radiation (λ = 0.154 nm). Surface morphology was studied by field emission scanning electron microscope (FESEM) LeoSupra 50 VP equipment. Film stoichiometric composition was determined by energy dispersive X-ray (EDX). The optical absorption measurements are carried out in the range of 300–1100 nm using UV-VIS spectrophotometer (Shimadzu UV-VIS mini 1240, Japan).

Results and discussion

Structural studies

The structural property of the films was investigated by X-ray diffraction. The XRD patterns obtained for the synthesized thin films deposited onto different substrates under the same deposition conditions are as shown in **(Fig. 1)**.

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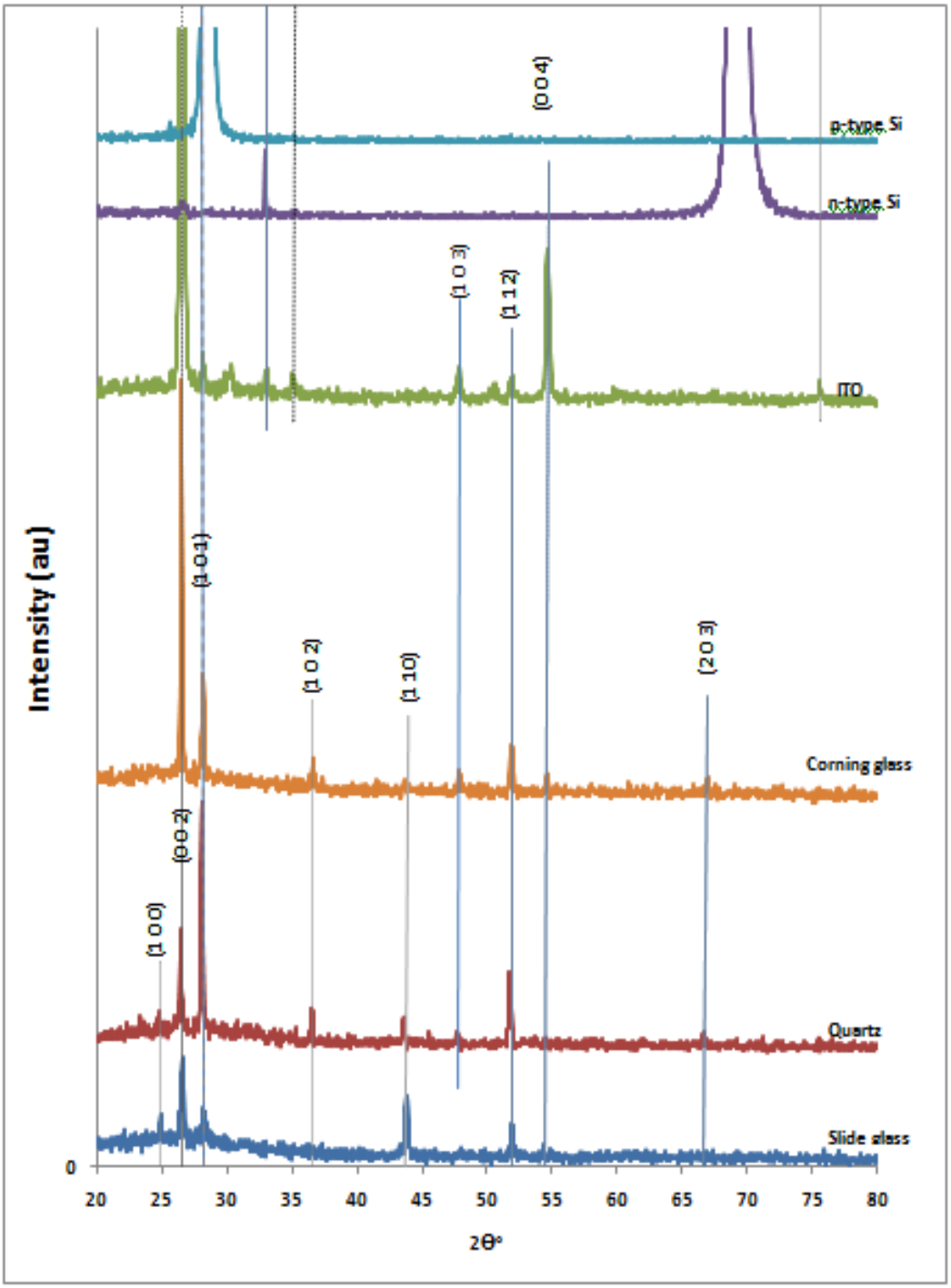


Fig. 1. XRD patterns of CdS thin films deposited on different substrates.

Regardless of the substrate material, the patterns show well-defined peaks suggesting the form of cadmium supplied, all films were polycrystalline in nature which crystallizes in hexagonal phase with a (0 0 2) preferred orientation. Another peaks corresponding to (101), (102), (110), (112) and (103) planes are observed with lower scattering intensity which also corresponds to hexagonal CdS films. However, in case of the XRD pattern of a film deposited onto ITO substrate, the additional reflection corresponding to the cubic CdS phase is also observed beside the hexagonal one. This shows the form of CdS films with mixed phase. Generally, the hexagonal (Wurtzite) phase and the cubic (Zinc blende) phase are two crystalline modifications of CdS **[13]**. Hexagonal CdS structure is recommended due to its higher stability regarding the cubic one **[26].** The hexagonal structure of our films due to prepare of the films at high temperatures 350 °C). Another small and broad secondary peaks corresponding to (203) and (004) plane orientations are also observed with lower scattering intensities. These predominant peaks are similar to those obtained by others using spray pyrolysis technique **[25, 26]**. X-ray data tells if a film is crystalline (if there are sharp peaks), and the most intense peak give information on the orientation. Higher (002) peak of CdS on ITO coated glass, corning glass, and quartz than that of CdS on the others substrates suggests a more oriented CdS film, and this is attributed to the strong influence of the oriented crystalline surface of these substrates **[27]**.

The crystallinity of CdS films on ITO coated glass and corning glass is sharper and better compared to that on the other ones, as given in (**Table 1)** for the strongest peaks, where higher X-ray intensity and lower full-width at half maximum (FWHM) peaks reveals high crystallinity of the film, this is may be attributed to the difference in grain size, where the films deposited on these two substrates have larger grain sizes.

Table 1.The determined structural parameters for the strongest peaks of CdS thin films deposited on different substrates compared with the standard data.

	Observed data									ICSD standard X-ray data base			
Substrate	Structure	hkl			I/I$_o$	FWHM 2θ°	2θ°	d (A°)	Lattice constant (A°)	Matched by Ref. Code	2θ°	d (A°)	Lattice constant (A°)
Slide glass	Hex.	0	0	2	100	0.5904	26.53	3.358	a=4.137 c=6.717	03-065-3414	26.45	3.367	a=4.132 c=6.734
		1	1	0	14.4	0.5904	43.76	2.068		03-065-3414	43.78	2.066	
		1	1	2	35.9	0.5904	51.91	1.761		03-065-3414	51.88	1.760	
Quartz	Hex.	0	0	2	82.4	0.264	26.43	3.371	a=4.163 b=6.742	01-075-1545	26.43	3.368	a=4.150 c=6.737
		1	0	1	100	0.5904	28.06	3.179		01-075-1545	28.11	3.171	
		1	1	2	36.9	0.5904	51.73	1.766		01-075-1545	51.69	1.766	
Corning	Hex.	0	0	2	100	0.5904	26.48	3.365	a=4.132 c=6.731	03-065-3414	26.45	3.367	a=4.132 c=6.734
		1	0	1	32.5	0.5904	28.13	3.171		03-065-3414	28.21	3.159	
		1	0	2	21.9	0.1508	36.64	2.452		03-065-3414	36.61	2.452	
ITO	Hex.	0	0	2	100	0.5904	26.53	3.359	a=5.818	01-077-2306	26.53	3.356	a=4.136 c=6.713
	Cubic	2	0	0	0.36	0.5904	30.15	2.964	a=4.135 c=6.719	01-089-0440	30.64	2.915	
	Hex.	0	0	4	3.99	0.5904	54.66	1.678		01-077-2306	54.64	1.678	
n-type	Hex.	1	0	0	10	0.7872	25.15	3.539	a=4.087 c=6.694	01-080-0006	24.92	3.568	a=4.121 c=6.682
	Hex.	0	0	2	20	0.5904	26.63	3.347		01-080-0006	26.66	3.341	
	Hex.	1	0	2	100	0.5904	36.49	2.462		01-080-0006	36.82	2.439	
p-type	Hex.	1	0	2	90	0.5904	35.94	2.567	a=4.093 c=6.715	03-065-3414	36.61	2.452	a=4.142 c=6.724
	Hex.	1	1	2	100	0.5904	51.43	1.776		03-065-3413	51.88	1.760	
	Hex.	2	0	3	90	0.96	66.71	1.400		00-002-0563	66.76	1.4	

The formation energy for the crystallization of the thin film on the amorphous substrates like glass is large in compared to its growth on crystalline substrates that promote nucleation and hence growth of high quality thin film on glass have been difficult. From XRD studies it is clear that the intensity of peaks is function of substrate material and that suggests the crystalline nature of the films.The interplanar spacing (d) for different planes of all the XRD patterns can be calculated by using the Bragg's diffraction condition **[28]**

$$2d\sin\theta = n\lambda \quad \text{.....(1)}$$

where, θ is the angle of diffraction, n is the order of diffraction and λ the X-ray wavelength of CuKα radiation (0.154 nm).The observed d values are found to be very close to the standard data given in ICSD for hexagonal CdS phase as shown in **(Table 1)**.

The lattice parameters (a) and (c) are calculated from the peaks positions using Bragg's formula of hexagonal system **[29]**:

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

Where d is the interplanar spacing. The obtained lattice parameter values are given in **(Table 1).** The ICSD values are also given for comparison. It was found that the observed values are in good agreement with the standard data. The slight deviation from the standard values might be due to the strain introduced in the samples due to excess Cd interstitials or S vacancies present in the samples **[29]**.

The average grain size of the particles can be calculated from the full width of half maximum (FWHM) values of the diffraction peaks using Debye-Scherrer formula **[28]** from the stronger peaks of (002) for each XRD patterns **[30]**:

$$D = \frac{k\lambda}{\beta\cos\theta} \quad \text{..... (3)}$$

Where D is the average crystallite size, λ the X-ray wavelength, k the shape factor equal 0.94, θ the diffraction angle, and β the FWHM. The average values of determined grain sizes are

found to be in the range from (14.49 to 93.36) nm, which revealed to the nanocrystalline grains of the films, as given in (**Table 2)** compared with the determined values by FESEM method.

Table 2. The average values of crystallite grain sizes determined by XRD and FESEM methods.

	XRD	FESEM
Substrate	**Grain size (nm)**	**Grain size (nm)**
Slide glass	15.6	82.23
Quartz glass	32.272	149
Corning glass	57.93	156.6
ITO coated glass	93.36	166
n-type Si	15.05	62.5
p-type Si	14.49	47.61

Smaller value for the grain size was found for the film deposited on p-type Si wafer and the crystallite grain size attains a maximum value for the films deposited on ITO coated glass and corning glass substrates, indicating to the better crystallinity of the films. This is due to the roughness and good adherence of the films which cause a higher density of nucleation centers in the case with ITO coated glass and corning glass generated denser CdS films **[29, 31]**.

Surface Morphological Studies

Field Emission Scanning Electron Microscopy (FESEM) is a promising technique for the study of morphology of thin films. It gives important information regarding growth, shape, and size of the particles. The surface morphology of the CdS thin films was investigated by scanning electron micrographs. **Fig. 2** shows the FESEM image of the fabricated thin films deposited on different substrates under the same magnification

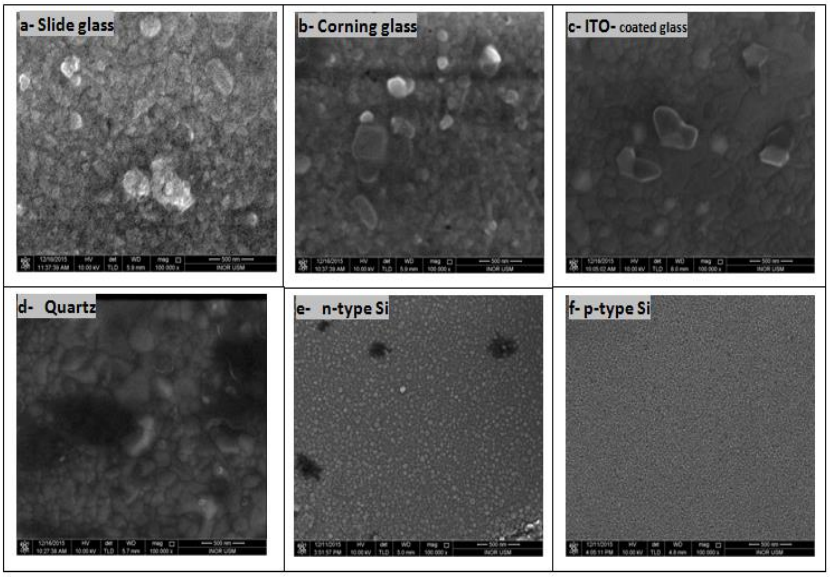


Fig. 2. FESEM images of CdS films deposited on different substrates.

It can be seen films morphologies show a significant change with changing the substrate type due to change in grain size regarding to substrate material. The FESEM micrograph of the film reveals the polycrystalline nature of the films which confirmed the XDR results. From FESEM one can see the well-covered crystalline grains on the films surface without no pinholes and cracks. The films deposited on Si substrates as shown in **(Fig. 2 e, and f)** are well covered by spherical grains, typically p-type Si whose size decreases and their density increases, the small particles accumulate and cover the entire surface of the substrate leading to a homogeneous layer. The FESEM micrograph of the film grown on silicon further reveals the polycrystalline nature of the films, and the well covered substrate with spherical grains confirming that the growth mechanism takes place of ion-by-ion mechanism, and this is supported by the pure hexagonal structure obtained as evident from XRD analysis **[25, 29]**.

Small particles are grouped to form overgrown larger clusters distributed in the films deposited on the other substrates as it is shown clearly in **(Fig. 2 a-d)**. This overgrowth can be explained on the basis of nucleation and coalescence process showing a remarkable increase in the size of the grains. Grown nanograins may have increased their size by further

deposition and come closer to each other **[31].** Thus, the larger grains appear to grow by coalescence of smaller ones. It is well known that large grain size films are important in the fabricate of high efficiency solar cells because such grains lead to reduced carrier scattering and improve carrier concentration by reducing carrier recombination **[23]**. It is observed from **Fig. 2** that the grains are compact, and homogeneous covered of the films grown on corning glass substrate compared to the surface of deposited films on the other substrates.

The average grain size of the particles was determined from FESEM image films deposited on different substrate by cross section analysis and using software scanning probe image processing (SPIP) software to measure grain size from cross section analysis tool as shown in the bottom of **(Fig.3)** for the film deposited on n-type Si substrate only.

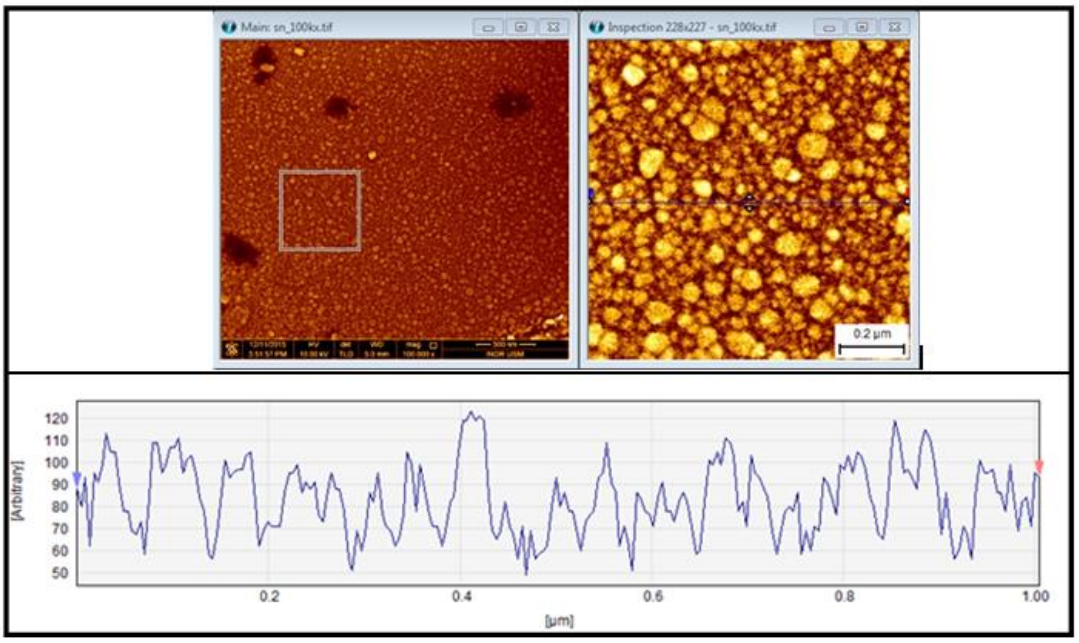


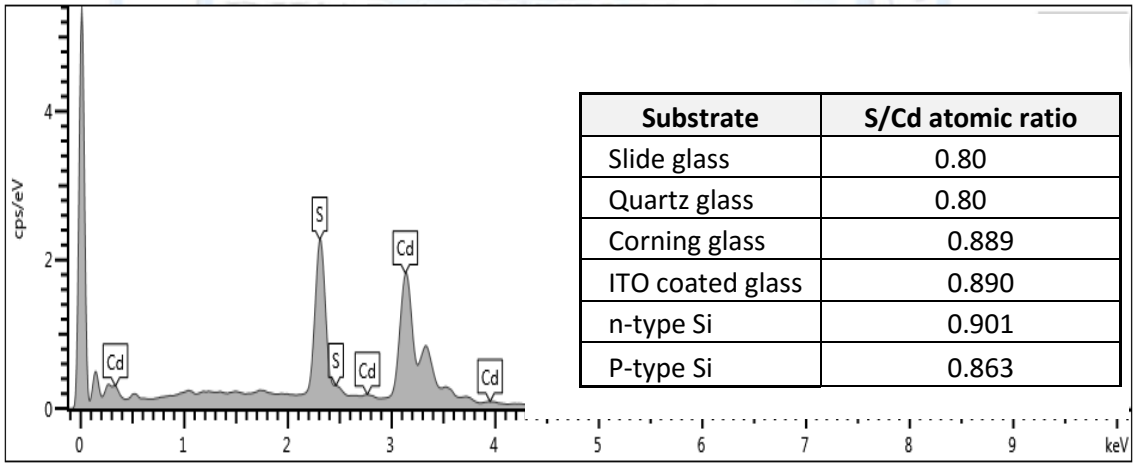
Fig. 3. FESEM image of the CdS thin film deposited on n-type substrate. The lower part of the image represents the surface cross section marked by dashed line.

The average crystallite grain size of the films determined by this method varies between 47.61 and 166 nm. Also smaller value for the grain size was found for the film deposited on p-type Si wafer and the crystallite grain size attains a maximum value for the films deposited on ITO

coated glass and corning glass substrates, the same as obtained in the XRD results. The values of determined grain size from Scherrer's equation are less than that from FESEM images. This is can be explained by agglomeration of small particles of the CdS to form large clusters **[29]**. Quite coincidence was found between the peak intensity and determined grain size from both XRD patterns and FESEM image for films deposited on different substrates as it can be seen from **(Fig. 1)** and **(Table 2)**.

Compositional Studies

Energy Dispersive X-ray (EDX) analysis was used to determine the ratio of atomic percentage of the films or to get the information about the elements in the CdS thin films. To confirm CdS structure we carried out EDX for the film deposited on ITO substrate only as shown in **(Fig. 4)**. The insert in the figure gives the determined elemental atomic ratio from EDX analysis for all the CdS films deposited on different substrates.



Substrate	S/Cd atomic ratio
Slide glass	0.80
Quartz glass	0.80
Corning glass	0.889
ITO coated glass	0.890
n-type Si	0.901
P-type Si	0.863

Fig. 4. EDX spectrum of CdS thin film deposited on ITO coated glass substrate. The inside table represents the elemental atomic ratio from EDX analysis for CdS thin films deposited on different substrates.

The figure shows peaks indicating the presence of S and Cd in the filims which confirms the high purity of the CdS thin films. EDX analysis revealed that the S/Cd atomic ratio is close to one for the films, which confirms the format of stoichiometric cadmium supplied films.The decreased value of S/Cd ratio obtained for the films deposited on some substrates like slide glass and quartz show that the films are deficient with sulfur or with surplus cadmium, which act as donors **[19]**. The S/Cd ratio of 0.9 for the films deposited on corning glass and ITO coated glass, showing the perfect stoichiometric nature of the films.

Optical Properties

To study the optical properties of the materials, the optical transmittance spectra of all the films were obtained. The optical measurements of the samples were carried out using a UV-VIS Spectrophotometer within the wavelength range (300-1100 nm). **(Fig. 5)** shows the optical transmission spectra as a function of wavelength of the all films grown on different substrates.

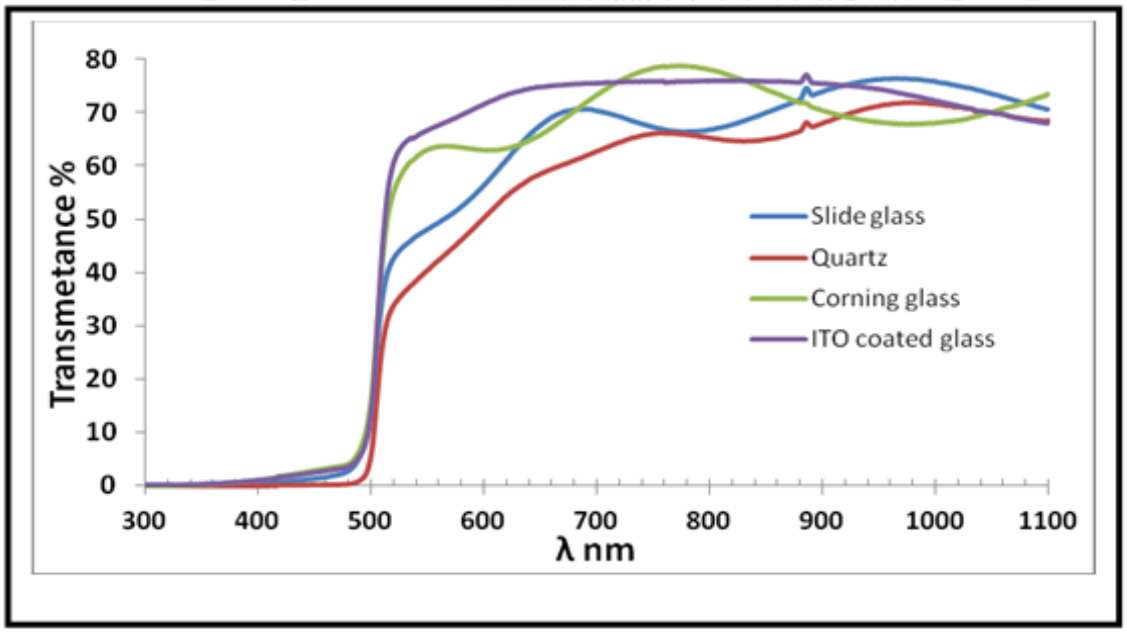


Fig. 5. Optical transmission spectra of CdS thin films deposited on different substrates.

The average transmittance of the films is about 65%, and increases to transparent up to 80% in the visible region for corning glass and ITO coated glass substrates. This improvement of the transmittance obtained from corning glass and ITO coated glass substrates can be explained by a less light scattering of these films due to the increase of grain size and high crystallization **[23].** This was well supported from the FESEM micrographs shown in **(Fig. 2)**.

The transmittance data can be used to calculate the optical absorption coefficients (α) of the films at different wavelength, which were used to determine the band gap (*Eg*) of the films deposited on different substrates. The value of α is obtained from the following relation **[32]**:

$$\alpha = 2.303 \frac{A}{t} \qquad \text{.....(4)}$$

where A is the absorbance and t is the thickness of the film.

The optical band gap (Eg) of the prepared films was calculated by using the classical relation for near edge optical absorption in semiconductors **[32]**:

$$(\alpha h\nu) = \mathrm{B}\,(h\nu - E_g)^{z} \qquad \text{.....(5)}$$

Where B is a constant, and z is a number depends on transition type, z is equal to 2 for direct gap compound. The $(\alpha h\nu)^2$ is plotted as a function of photon energy ($h\nu$) is shown in **(Fig.6).**

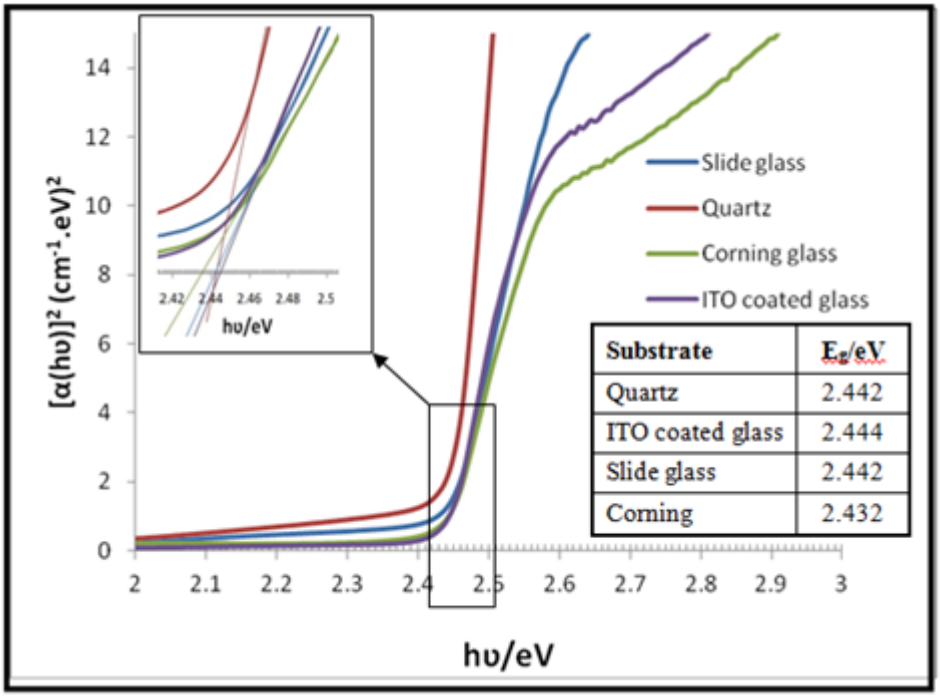


Fig. 6. Plot of $(\alpha h\nu)^2$ against incident photon energy (hv) of the CdS thin films deposited on different substrates.

It can be seen $(\alpha h\nu)^2$ varies almost linearly with photon energy above the band gap energy (E_g). The linear extrapolation of this curve to the energy axis at $\alpha = 0$ gives the value of band gap of the thin films. The estimated optical band gaps were found to be in the range 2.432 eV-2.444 eV for the films deposited on different substrates. The obtained values are in agreement with the earlier reports **[28, 30]**.

Conclusion

Cadmium sulfide thin films were prepared onto different substrates including quartz, corning glass, ITO coated glass, n-type silicon wafer, p-type silicon wafer, and slide glass by using chemical spray pyrolysis technique. The influence of substrate material on the structure and optical properties of the films was investigated. The structural analyses revealed that all films

were polycrystalline in nature which crystallizes in hexagonal phase with a preferential orientation along the (002) plane. The inter-planar spacing values are found close to the standard data for hexagonal CdS phase. The determined lattice constants show a slight deviation from the standard values. The crystallite grain size attains a maximum values for the film deposited on ITO coated glass and corning glass substrates to be(166 and 156.6) nm, and the smallest one corresponds to the films deposited on n-type and p-type Si substrate to be (15.05 and 14.49) nm, respectively. The surface morphological study showed that all the films have uniform well covered grain size on the surface of the substrates. The compositional analyses showed that the S/Cd atomic ratio is close to one, which confirms the form of stoichiometric cadmium sulfide films. The optical studies showed the films exhibited a good transmittance of about (65 to 80) % in the visible and near infrared regions of the electromagnetic spectrum. The optical band gap varied in the range (2.432 to 2.444) eV. The films obtained on ITO coated glass and corning glass were found to exhibit well oriented properties than films obtained on other substrates, and these two substrates seems to be the most favorable substrates in the form of CdS thin films using spray pyrolysis technique.

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