# Effect of Annealing Temperature and Thickness on the Structural and Optical Properties of CdSeThin Films

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**Received in :12 Qctober 2014 Accepted in :21 December 2014**

## Abstract

CdSe alloy has been prepared successfully from its high purity elements. Thin films of this alloy with different thicknesses (300,700)nm have been grown on glass substrates at room temperature under very low pressure ($10^{-5}$)Torr with rate of deposition (1.7)nm/sec by thermal evaporation technique, after that these thin films have been heat treated under low pressure ($10^{-2}$)Torr at (473,673)K for one hour. X-ray patterns showed that both CdSe alloy and thin films are polycrystalline and have the hexagonal structure with preferential orientation in the [100] and [002] direction respectively. The optical measurements indicated that CdSe thin films have allowed direct optical energy band gap, and it increases from (1.77-1.84) eV and from (1.6-1.65)eV with the increase in temperature from (R.T-673)K for thicknesses (300,700)nm, respectively. The optical constants (refractive index, extinction coefficient and the real, imaginary parts of dielectric constant) were also studied.

**Keywords:** CdSe alloy, CdSe thin films, X-ray patterns, optical measurement

# Theoretical

The II-VI semiconducting compounds, especially the cadmium chalcogenides,have been extensively studied due to their potential applications in semiconductor devices and solar cells fabrication[1-3].Among cadmium chalcogenides group semiconductors,CdSe is an important material which has been mainly utilized for solar cells[4,5].CdSe is also considered to be a potential candidate for various applications such as thin film transistors, gamma ray detectors, laser, photoconductors, sensors [6,7], acousto optic devices, photographic photoreceptors[5],and photo electrochemical cells[8]. It has high efficiency of radiative recombination, high absorption coefficient, high photosensitivity, direct band gap corresponding to a wide spectrum of wave lengths from ulttraviolet to infrared regions[9].The properties of materials prepared by different methods are critically dependent on the nature of preparation technique and subsequent heat treatments like annealing in air, vaccum or different gaseous environments etc[10].A variety of techniques have been used to prepare CdSe thin films including thermal evaporation[11-13],electron beam evaporation[14],chemical bath method[15] and molecular beam epitaxy[16]. Among the various deposition techniques available for the preparation of CdSe thin films, the vacuum evaporation technique is very important and promising deposition technique, because it offers many possibilities to modify the deposition parameters and to obtain films with controlled structures and properties [17]. The lattice structure of CdSe can be found in the forms of sphalerite(cubic) and wurtzite(hexagonal).The former is a metastable phase constituting the almost exclusive product of an electrochemical formation process, while the latter is the thermodynamically stable structure obtained either by annealing the cubic phase or directly by various, electro- less preparation techniques[18]. Deposited thin films may contain many defects such as voids, pinholes etc. Annealing of thin films reduces the defects and increases the crystallite size along with recrystallization process [14].

This paper analyzes the effect of thickness and annealing temperature on the structural and optical properties of CdSe thin films prepared by thermal evaporation method.

# Experimental

To prepare CdSe alloy, appropriate atomic percentage of high purity (99.999%) cadmium (Cd), and selenium (Se), were taken by using sensitive electrical balance type Precisa XB 220A with sensitivity of $10^{-4}$. These elements were put in a quartz ampoule. The quartz ampoule was cleaned carefully with water and alcohol respectively, to remove dust, grease, and other possible contaminants. Then the ampoule was linked by specific design to the vacuum unit. When the pressure reached ($2*10^{-2}$) Torr the ampoule was sealed. Then the ampoule was heated with a rate of (363)K per hour in steps up to (1173)K using a programmable furnace type Carbolite. The ampoule is kept at this temperature for three hours. After that the ampoule was cooled down in furnace, and then broken to bring out the alloy.

Edward coating unit model (306A) was used as a vacuum unit system, which is used to prepare thermally evaporated CdSe films at pressure ($10^{-5}$ )Torr on a freshly cleaned corning glass substrate at room temperature.

Molybdenum boat with pin holes cover was used for films preparation with distance about (9)cm from substrate. The thickness of prepared films has been determined using the optical interference fringes technique.

All the prepared films were treated under vacuum of ($10^{-2}$)Torr at different annealing temperatures (473,673)K for one hour.

Philips X-ray diffractometer with copper $K_\alpha$ radiation of the wavelength ($\lambda$=1.541)Å were used to determine the crystalline structure of CdSe alloy and thin films.

A spectrophotometer model (UV-Visible 2601) double-beam spectrophotometer is used to measure the transmittance and absorbance spectrum in the range (400-1100) nm region for all CdSe thin films, using a blank substrate as the reference position.

Absorption coefficient (α) represents the attenuation that occurs in incident photon energy on the material for unit thickness, and the main reason for this attenuation is attributed to the absorption processes [19].

The following relation below is refereed to the absorption coefficient [19]:

$$\alpha = 2.303\frac{A}{t} \qquad (1)$$

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

The refractive index (n) was obtained from the following relation [20]:

$$n = \frac{1+R}{1-R} + [\frac{4R}{(1-R)^2} - k^2]^{1/2} \qquad (2)$$

where k represents the extinction coefficient which is calculated by the relation [19]:

$$k = \frac{\alpha\lambda}{4\pi} \qquad (3)$$

and R is the reflection which is calculated by the relation:

$$R = 1 - A - T \qquad (4)$$

The real and imaginary part of dielectric constant ($\varepsilon_r, \varepsilon_i$) can be calculated by using equations[19]:

$$\varepsilon_r = n^2 - k^2 \qquad (5)$$

$$\varepsilon_i = 2nk \qquad (6)$$

# Results and Discussion

## Structural Properties

X-ray diffraction pattern of CdSe alloy is shown in Fig. (1). The spectrum is seen to exhibit nine sharp peaks(100),(002),(101),(102),(110),(103),(200),(112)and(201) corresponding to hexagonal phase of CdSe as compared with the standard values in ASTM cards. No diffraction peak corresponding to metallic Cd, Se, or other compounds were observed. The X-ray diffraction parameters (interplaner distance, Miller indices and Brag angle) for CdSe alloy are listed in table (1).

From the X-ray diffraction patterns of as-deposited CdSe thin films with different thickness (300, 700)nm, one can observe that the thin films have the hexagonal structure as shown in Fiq. (2a and c), the figure shows that the patterns include two sharp peaks referred to (002) and (103) direction. Also this figure confirms that the preferential orientation is in the (002) direction. These results were compared with the ASTM cards for CdSe, and match with [5, 14, 21]. The dominance of (002) hexagonal reflection indicates that the preferential growth of crystallites is along this particular direction which indicates that the preferred orientation of the crystallite grains with c-axis perpendicular to the plane of the substrate that makes (002) peak very strong as compared to the other peak [21]. The annealed CdSe thin films with different thickness (300, 700)nm at 673K exhibit the same behavior as in as-deposited thin films as illustrated in table (2), but with improving in the crystalline structure by noticing that the main peak (002) becomes sharper than that in as-deposited films with increase in their intensity as shown in Fig. (2b and d). This is attributed to the increase in crystalline grain size. The crystalline grain size (D), of the CdSe thin films has been calculated by using Scherrer͑s formula[10].

$$D=\frac{K\lambda}{\beta'\cos\theta} \qquad (7)$$

where $K$ is some constant 0.9 for CdSe, $\lambda$ the X-ray wave length used, $\beta'$ the angular line width of half maximum intensity and $\theta$ is the Bragg᾽s diffraction angle.

And also given in table (2). It is evident from the table the grain size of CdSe thin films increases with the increase in temperature and thickness, and this is also shown by [10,21] about thickness and annealing temperature respectively. This attributed to join the small crystallites each other in main peak due to annealing process, and this property increase with the increase of thickness. This result confirms what mentioned previously about annealing temperature in [14].

## Optical Properties

The experimental transmission spectra for the CdSe thin films with different thicknesses and annealing temperatures ($T_a$) were made at room temperature in the spectral range (400-1100) nm are shown in Fig. (3).

All spectra show good transparency in the visible and near infrared region. For heat treated samples the transmission is greater than before the heat treatment. This fact may be attributed to increase in the crystallite size observed for heat treated samples [20]. In general, from figure (3) we can observe that transmittance increase with the increase of $T_a$. Otherwise, the transmittance decreases but not uniforms with the increase of thickness and shifted to longer wavelengths. This may be attributed to the creation levels at the energy band by the increase of thickness. Similar notices are obtained by Baban et al. [22] and Hambrock et al. [23].

The behavior of the absorption spectra is opposite completely to that of the transmission spectra.

The absorption coefficient (α) was determined from the region of high absorption at the fundamental absorption edge of the films using the equation (1). Fig. (4) shows the variation of the absorption coefficient versus the wavelength for different $T_a$ and thicknesses of the CdSe films.

From this figure, we can observe that the α for all films were fairly high values reached above $(10^4)\text{cm}^{-1}$ , also we found that the α in general decreases with the increase of $T_a$ for different thicknesses which is attributed to an increase in transmittance of related films. This is in agreement with result of Makadsi et al. [24]. From table (3) we found that the value of α decreases from$(1.84-1.26)\times10^4\text{cm}^{-1}$ and from$(1.88-1.29)\times10^4\text{cm}^{-1}$ with the increase of $T_a$ for thicknesses (300,700)nm, respectively at λ equal 690nm, also from this table we notice that the variation of α with films thickness is non-systematic, and this may be attributed to the variation in crystal structure of the films, and the optical absorption coefficient increased with the increase of film thickness, because in the case of thicker film, more atoms are present in the film so more states will be available for the photons to be absorbed.

The optical band gap of material can be determined by using the following equation [25].

$$\alpha=\beta\,(h\upsilon-Eg)^r/h\upsilon \qquad (8)$$

where β is a constant , the exponent r depends on the type of transition have many values (1/2,2,3/2 and 3) corresponding to allowed direct , allowed in direct , forbidden direct and forbidden indirect transition , respectively and hυ is the incident photon energy, which is calculated from this relation:

$$h\upsilon(eV)=\frac{1240}{\lambda(nm)} \qquad (9)$$

Plot of $(\alpha h\upsilon)^2$ versus hυ for CdSe thin films with different annealing temperatures and thicknesses is shown in Fig. (5). The plot is linear at the absorption edge which confirms that CdSe is a semiconductor with a direct band gap [10]. Extrapolation of the line to the hυ axis gives the optical band gap.

The value of $E_g^{opt}$ increases from (1.77-1.84)eV and from (1.6-1.65)eV with the increase of $T_a$ for thicknesses(300,700)nm, respectively as shown in table (3). The increase in value of band gap may be due to the decrease in defect states near the bands and this turn increased the value of $E_g^{opt}$ [26]. This result is in agreement with Ugwu et al. [27].

Also from table (3) we can notice that the $E_g^{opt}$ decreases with the increase of thickness, this is due to an increase in partical size [5]. The same result is obtained by Padiyan et al. [28].

The variation of the refractive index (n) as function of the wavelength for CdSe films at different $T_a$ and thicknesses is shown in Fig. (6), which indicates that n decreases with the increase of $T_a$. This behavior is may be due to decrease in the reflection which the refractive index depend on it. From table (3) we observed that the variation of n with films thickness is non- systematic, this is may be due to the variation in crystal structure of the films.

Graph of extinction coefficient (k) as a function of wavelength for different $T_a$ and thicknesses of CdSe films is plotted in Fig. (7). This figure revealed that k in general decreases as film $T_a$ increases. The variation of k with film thickness is non- systematic, as shown table (3). This is attributed to the same reason mentioned previously in the absorption coefficient because the behavior of k is similar to α.

The dielectric constant consists of real part ($\varepsilon_r$) and imaginary part ($\varepsilon_i$) depends on the frequency of the electromagnetic wave. The variation of $\varepsilon_r$ and $\varepsilon_i$ versus wavelength at different $T_a$ and thicknesses are shown in Fig. (8a and b) respectively. The behavior of $\varepsilon_r$ is similar to that of the refractive index because of the smaller value of $k^2$ compared with $n^2$ , while $\varepsilon_i$ behavior is similar to that of extinction coefficient because it mainly depends on the k value, which are related to the variation of absorption coefficient. The variation of $\varepsilon_r$ and $\varepsilon_i$ with films thickness are non-systematic, this means that this material possesses a specialized property with thickness.

## Conclusions

CdSe alloy was prepared successfully and used for thin films preparation. XRD tests for alloy and thin films showed that both are polycrystalline and have the hexagonal structure with preferential orientation in the [100] and [002] direction respectively. Thus, it can be broadly concluded that CdSe films having hexagonal structures can be prepared by thermal evaporation method.

The influence of thickness and annealing temperatures on the values of optical parameters of CdSe thin films is investigated. All thin films exhibit allowed direct optical energy band gap and high transmittance in the visible and near infrared region, thus making the films suitable for optoelectronic devices, for instance as window layers in solar cells.

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**Table No. (1): Structural parameters of CdSe alloy**

| $2\theta$(exp.) (degree) | $2\theta$(stand.) (degree) | hkl (stand.) | d (exp.) (Å) | d(stand.) (Å) |
|---|---|---|---|---|
| 23.9 | 23.901 | 100 | 3.720 | 3.719 |
| 25.4 | 25.354 | 002 | 3.503 | 3.509 |
| 27 | 27.080 | 101 | 3.299 | 3.290 |
| 35.1 | 35.107 | 102 | 2.554 | 2.554 |
| 41.9 | 41.968 | 110 | 2.154 | 2.150 |
| 45.78 | 45.788 | 103 | 1.980 | 1.980 |
| 48.8 | 48.845 | 200 | 1.864 | 1.863 |
| 49.7 | 49.669 | 112 | 1.832 | 1.834 |
| 50.7 | 50.673 | 201 | 1.799 | 1.799 |

**Table No. (2): Structural parameters of as deposited and annealed at 673K CdSe thin films at different thicknesses**

| Thickness (nm) | $T_a$(K) | $2\theta$(exp.) (degree) | $2\theta$(stand.) (degree) | hkl (stand.) | d(exp.) (Å) | d(stand.) (Å) | D(nm) |
|---|---|---|---|---|---|---|---|
| 300 | R.T | 24.4 | 25.354 | 002 | 3.502 | 3.508 | 162.926 |
| | | 45.734 | 45.788 | 103 | 1.98 | 1.979 | |
| | 673 | 25.5 | 25.354 | 002 | 3.488 | 3.508 | 217.21 |
| | | 45.9 | 45.788 | 103 | 1.974 | 1.979 | |
| 700 | R.T | 25.5 | 25.354 | 002 | 3.488 | 3.508 | 325.87 |
| | | 45.9 | 45.788 | 103 | 1.974 | 1.979 | |
| | 673 | 25.5 | 25.345 | 002 | 3.488 | 3.508 | 362.048 |
| | | 45.9 | 45.788 | 103 | 1.974 | 1.979 | |

**Table No. (3): Optical parameters of CdSe thin films at different thicknesses and annealing temperatures**

| $\lambda$=690nm | | | | | | | |
|---|---|---|---|---|---|---|---|
| Thickness (nm) | $T_a$ (K) | $\alpha\times10^4$ $(cm)^{-1}$ | $E_g^{opt}$ (eV) | n | k | $\varepsilon_r$ | $\varepsilon_i$ |
| 300 | R.T | 1.84 | 1.77 | 2.499 | 0.1 | 6.238 | 0.503 |
| | 473 | 1.4 | 1.8 | 2.334 | 0.076 | 5.442 | 0.359 |
| | 673 | 1.26 | 1.84 | 2.267 | 0.069 | 5.138 | 0.314 |
| 700 | R.T | 1.88 | 1.6 | 2.511 | 0.101 | 6.296 | 0.509 |
| | 473 | 1.86 | 1.63 | 2.507 | 0.1 | 6.276 | 0.505 |
| | 673 | 1.29 | 1.65 | 2.280 | 0.07 | 5.196 | 0.322 |

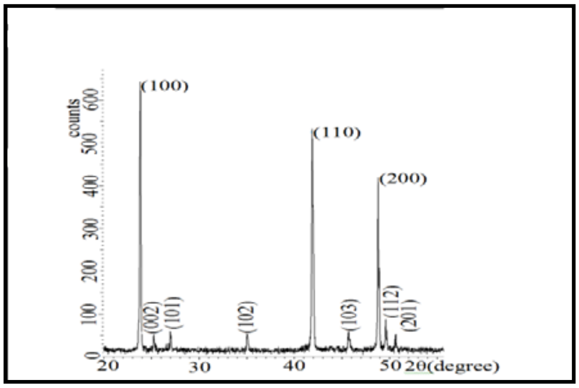

Figure No.(1): X-ray diffraction pattern of CdSe alloy

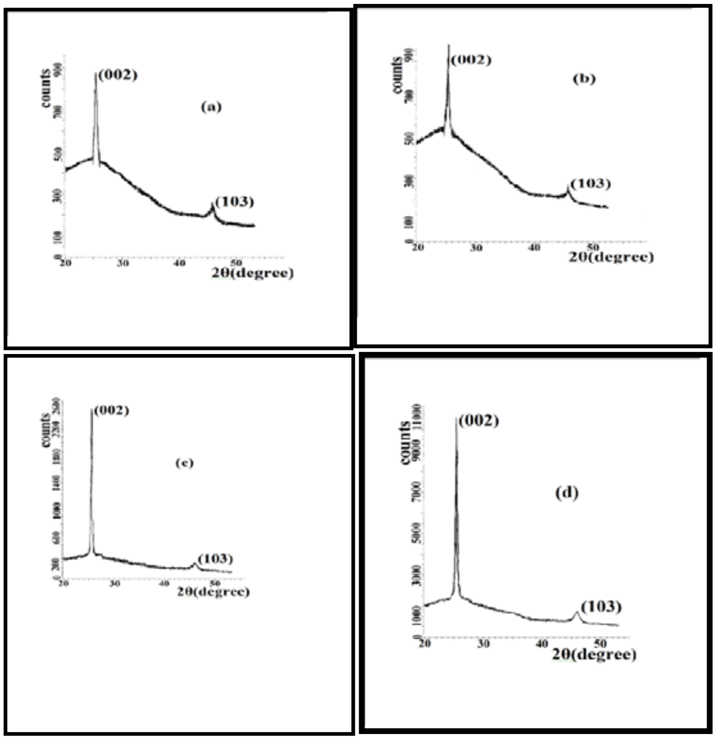

Figure No.(2): X-ray diffraction pattern of CdSe thin films:
a:300nm at R.T ,b:300nm at 673K ,c:700nm at R.T ,d:700nm at 673K.

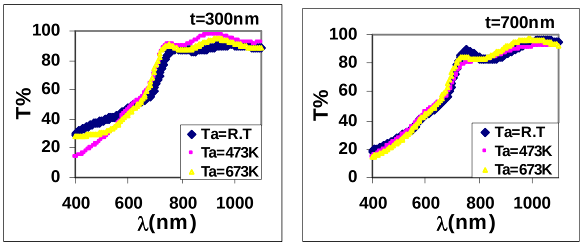

Figure No.(3): Transmittance spectra for CdSe thin films at different annealing temperatures and thicknesses

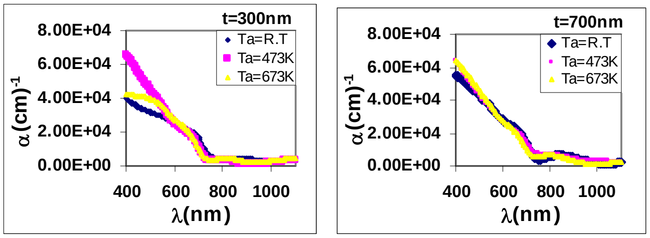

**Figure No.(4): Variation of absorption coefficient with wavelength for CdSe thin films at different annealing temperatures and thicknesses**

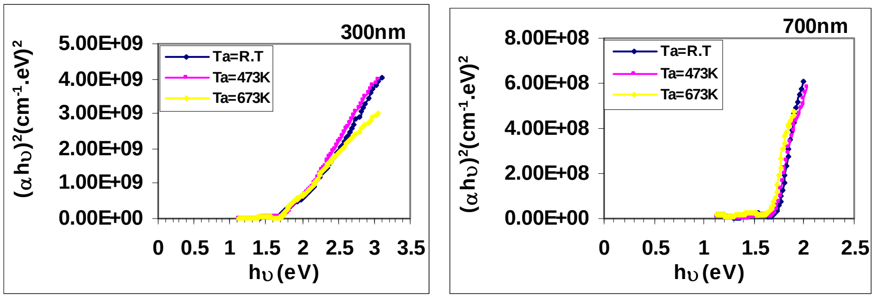

**Figure No.(5): $(\alpha h\upsilon)^2$ with photon energy for CdSe thin films at different annealing temperatures and thicknesses**

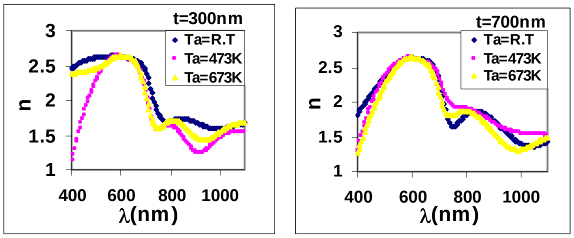

**Figure No.(6): Variation of refractive index with wavelength for CdSe thin films at different annealing temperatures and thicknesses**

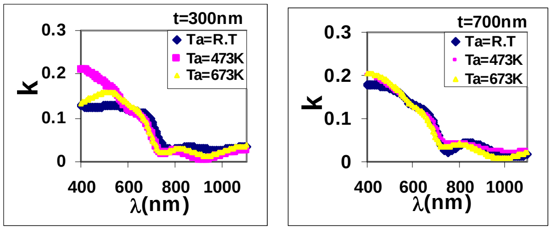

**Figure No.(7): Variation of extinction coefficient with wavelength for CdSe thin films at different annealing temperatures and thicknesses**

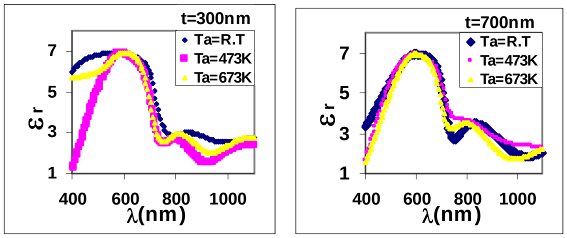

**Figure No.(8-a): Variation of $\varepsilon_r$ with wavelength for CdSe thin films at different annealing temperatures and thicknesses**

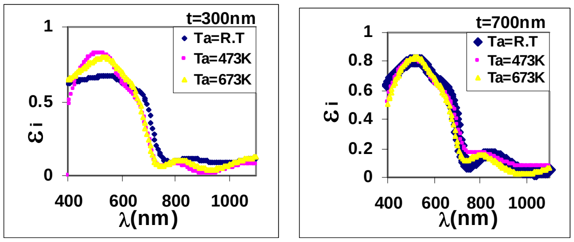

**Figure No.(8-b): Variation of $\varepsilon_i$ with wavelength for CdSe thin films at different annealing temperatures and thicknesses.**

# ثأثير درجة حرارة التلدين والسمك في الخصائص التركيبية والبصرية لأغشية CdSe الرقيقة

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**أستلم في : 12تشرين الاول 2014 قبل في :21كانون الاول 2014**

## الخلاصه

حضرت سبيكة CdSe بنجاح من عناصرها عالية النقاوه. ورسبت الأغشية الرقيقة لهذه السبيكة بأسماك مختلفة (300,700)nm على ارضيات زجاجية بدرجة حرارة الغرفة وتحت ضغط واطئ جدا $(10^{-5})$Torr بمعدل تبخير (1.7)nm/sec بواسطة تقانة التبخير الحراري ، وبعد ذلك تمت معاملة الأغشيه حراريا تحت ضغط واطئ $(10^{-2})$ Torr عند درجات حرارة (473,673)K لمدة ساعة. أظهرت فحوصات الاشعة السينية بأن كلاً من السبيكة والأغشية المحضرة هي متعددة البلورات, وانها تمتلك التركيب السداسي بتوجيه مفضل بالاتجاه [100],[002] على التوالي.وبينت القياسات البصرية لأغشية CdSe أنها تمتلك فجوة طاقة بصرية مباشره تزداد من (1.77- 1.84)eV ومن (1.6 -1.65)eV بزيادة درجة الحرارة من (R.T- 673)K للأسماك (300,700)nm على التوالي. كما دُرست الثوابت البصرية (معامل الانكسار ومعامل الخمود ,وثابت العزل بجزأيه الحقيقي والخيالي).

**الكلمات المفتاحية:** سبيكة CdSe, وأغشية CdSe الرقيقة, وفحوصات الاشعة السينية, والقياسات البصرية