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Kimyasal banyo depolama yöntemi ile elde edilen cds filmlerinin yapısal, optiksel ve morfolojik özellikleri üzerine tavlama işleminin etkisi

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ÖZ

Kadmiyum sülfür filmleri kimyasal banyo depolama yöntemi ile cam tabanlar üzerine biriktirildi. Elde edilen filmler azot gazında 1 saat boyunca 200-400 ºC sıcaklık aralığında tavlandı. Filmlerin kristal yapısı x-ışını kırınım (XRD) desenlerinden belirlendi. XRD verileri, tavlama etkisiyle CdS (FCC) yapısından α-CdS (Hawleyite) formuna geçiş olduğunu gösterdi. Elde edilen CdS filmlerinin optiksel bant aralığı değerleri optiksel absorbans spetrumlarından yararlanılarak 2,33 ve 2,39 eV olarak belirlendi. Bant sarkmaları değerleri 120-350 meV arasında olduğu hesaplandı. Filmlerin görünür bölgede %45-92 arasında geçirgenlik değerlerine sahip olduğu belirlendi.

Anahtar Kelimeler: CdS, Kimyasal banyo depolama, Tavlama, Optik özellikler

Influence of the annealing treatment on structural, optical and morphological properties of CdS films obtained by chemical bath deposition

ABSTRACT

Cadmium sulphide films have been deposited onto glass substrate by chemical bath deposition method. The CdS films have been annealed in the temperature range of 200-400 ºC at one hour in nitrogen gas. The crystal structures of the films were determined by x-ray diffraction (XRD) patterns. XRD data showed that the transition from CdS (FCC) to α-CdS (Hawleyite) form by annealing effect. The optical band gaps of the films have been obtained from the optical absorbance spectrum found to be 2.33 and 2.39 eV. The bands bending in the films were calculated to be between 120-350 meV. The optical transmissions of the films have been determined to be between 45-92 % in the visible region.

Keywords: CdS, Chemical bath deposition, Thermal annealing, Optical properties

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1. INTRODUCTION

Group II–VI compound semiconductor films have shown great advantage in various electronic devices and as optical windows for solar cells [1]. Among them, CdS films have an important role in applications such as light emitting diodes for flat panel displays, transistors for electronic switches [2]. It is known that CdS films can be deposited with either of two different structural phases like the hexagonal close packed (wurtzite) and the face-centred cubic (zinc blende or hawleyite) [3, 4]. CdS films have high absorption coefficient and direct band gap material, and its optical band gap values are from 2.30 to 2.44 eV [5-7]. CdS films have been prepared through different chemical and physical techniques like vacuum evaporation (VE) [8], sputtering [5, 9], electro-deposition [10], molecular beam epitaxy (MBE) [11], ultrasonic spray pyrolysis (USP) [6] and chemical bath deposition (CBD) [1, 9]. Among these, CBD has become the favoured route because of its simpleness, low temperature, low cost, reproducibility and large-area deposition technique for CdS films. CdS has been used as a window layer in CdTe solar cells and the buffer layer in Cu (In, Ga) Se_2 (CIGS) solar cells [12]. However, the quality and stoichiometry of the films differ as their structural and optical properties depended on the deposition conditions such as pH of the solution, deposition temperature and deposition time [1]

In this work, CdS films have been deposited using the CBD method at 80 ºC to obtain good and adherent films. The films were annealed in nitrogen atmosphere in order to prevent to reaction of the films with the oxygen in air. The influence of different annealing temperature on the growth formation of the film, crystal structural, optical and morphological properties of CdS films was investigated. If a material thought to be used in solar cells, optical characterization must be performed in detail. The observed changes in the crystal structure, optical and morphological properties were present. The effect of annealing temperature on crystal structure, optical and morphological properties of this work and discussed in detail.

2. EXPERIMENTAL DETAILS

The CdS films were obtained on the commercial microscope glass slide as the substrate 13 x 75 x 1 mm^3 using the CBD method. The glass slide was boiled in distilled water with detergent about ten minutes, rinsed and dried by compressed air. The substrate was degreased by ultrasonic treatment in distilled water about 15 min. and dried by compressed air. Finally, they were etched in acetone and propane and then dried by compressed air and so the cleaning procedures were completed.

CdS film was deposited on the glass substrates by the CBD method from the distilled water solutions containing 0.02 M $CdCl_2 \cdot H_2O$, 0.5 M KOH, 1.5 M NH_4NO_3 and 0.2 M thiourea in a 1:2.5:1:1 (by the volume). The chemical solution was continuously stirred to obtain a homogeneous mixture. The colour of the solution changed during the deposition from light yellow to orange. The pH of the solution in 100 ml beaker was adjusted to 10.50 drop by drop adding ammonia. The substrate was placed vertically into the chemical bath in the beaker. The temperature of aqueous solution bath was kept at 80 ºC for the entire duration of the deposition. The optimum deposition time was determined 40 minutes. The glass slide coated with CdS film was rinsed in distilled water in the beaker after deposition to remove some unwanted and loose particles and dried in air at 70 ºC. The film growth was seen better front side of the glass. The film of back side of the substrate was removed by using nitric acid. Four pieces were cut from the obtained film. The selected from the obtained films were annealed at 200, 300 and 400 ºC at 1 hour in nitrogen gas. Thus, the CdS films are obtained as-grown and annealed at 200, 300 and 400 ºC, respectively.

The average thickness of CdS films were calculated by weight difference method assuming the sample is uniform and dense as that of the bulk having density of 4.84 $g \cdot cm^{-3}$. The thicknesses of produced CdS films have been determined to be 225 nm.

The crystal structures of the films were analysed using Bruker D8-X-Ray Spectrophotometer with CuK_α radiation (1.5406 Å) scan rate 0.1 sec/step. The diffraction angle 2θ was varied between 20º and 60º. The optical absorption and transmittance spectra of the CdS films were measured in the wavelength range of between 200-3300 nm by Solid Spec-3700 UV-VIS NIR Spectrophometer. Surface morphology of the films was characterized

using Zeiss Ultra Plus Field Emission Scanning Electron Microscope (FESEM).

3. RESULT AND DISCUSSION

3.1. Crystal structural properties of the CdS films

The X-ray diffraction patterns ($20^o \leq 2\theta \leq 60^o$) of the CdS films as-grown and at three different annealing temperatures are given in Fig. 1. The characteristic peak in the diffraction pattern of the as-grown CdS film is assigned to the (111) plane at 2θ (27.23^o) value from ICDD powder diffraction file (PDF) 03-065-8873 and the crystal structure of the film is face-centred cubic (α-CdS, Hawleyite). The crystallized of the film show a preferential growth along to [111] direction. As annealing temperature increases, the peaks in the diffraction pattern of the CdS films as-grown and annealed at 200, 300 and 400 ^{o}C temperatures is shifted towards smaller 2θ degrees. The intense peaks at 26.79^o, 26.70^o and 26.72^o correspond to (111) plane of cubic phase form of the crystal structure on the basis of PDF 01-089-0440 data (Table 1.). According to the Fig. 1, the films have (111) as the preferred orientation. It can be concluded that the annealing affects the formation of CdS films. We can say that, we compare the annealing films each other, the intensity of the CdS films (111) peak increases, this finding suggest that the annealing in the nitrogen gas is effective for improving the crystalline quality. The intensity of the peak observed around 26^o increases as the annealing temperature increases. This verifies our expectation from annealing and more ordered films. We can say that the intensity of the annealed CdS peak increases when films are annealed at higher temperatures up to 400 ^{o}C. Similar situation of CdS films deposited by CBD have been reported in literature [13].

XRD patterns indicated that with changing annealing temperature of the film and the CdS film structure changes from α-CdS (Hawleyite) to CdS (FCC). The CdS films deposited at low temperatures (< 100 ^{o}C), normally show a cubic structure, and films deposited at high temperatures (> 300 ^{o}C), show hexagonal structures [8]. Also, the CdS film structure transforms from cubic structure to mixed cubic and hexagonal structure by increasing the pH values of the bath solution [14]. When the concentration of film changing, raising the bath temperature, increasing the reaction time or the annealing temperature can be improve the diffraction peaks become higher and narrower.

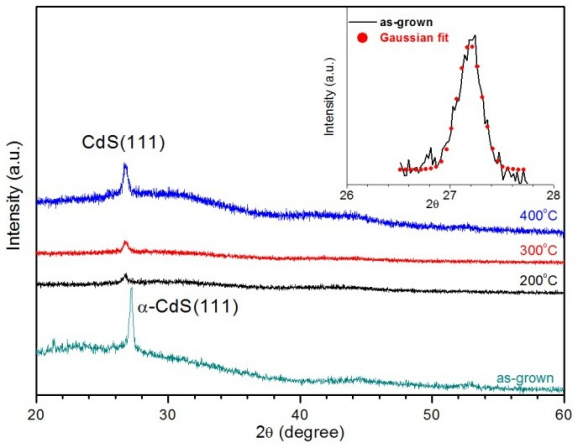


Figure 1. XRD image of the CdS films were obtained as-grown and annealed at 200, 300 and 400 ^{o}C temperatures. The inset shows Gaussian fit for as grown.

The crystallite size (D) of constituting the crystal structure is known to affect the optical properties of the film. The peaks on the diffraction pattern of the films can be calculated using crystallite size. The crystallite sizes of the CdS films can be estimated using by Debye-Scherrer's formula [15]

$$D = \frac{0.9\lambda}{B\cos\theta} \quad (1)$$

where λ is the wavelength of the of CuKα radiation, B is the full-width at half maximum (FWHM) and θ is the Bragg's diffraction angle. The intensity of the observed peaks in the XRD patterns is very small due to calculate the crystallite sizes of the films, for this reason, we subtract the background and fit the XRD peak into Gaussian in the inset Fig.1. The XRD spectrum of the CdS film is found to be best fitted to Gaussian peak by using computer program. The program uses a Marquardt–Levenberg algorithm to minimize the difference between the experimental data and the fitting equation. B values for cubic CdS peak have been calculated using the fit and the crystallite sizes have been estimated by Debye-Scherrer's formula. And also, the crystal lattice parameters of the film are estimated by using Eq. (2) for cubic crystals [15].

$$\frac{1}{d^2} = \frac{(h^2+k^2+\ell^2)}{a^2} \quad (2)$$

where d is the interplanar spacing, $(h\ k\ \ell)$ are Miller indices of the plane and a lattice parameters of the cubic crystal structures. The crystallite size values and lattices parameters of the CdS films were given in Table 1. The grain size of the films

Table 1. The crystallite size, lattice parameter, interplanar spacing and Urbach energy values of the CdS films obtained as-grown and annealed at 200, 300 and 400 °C temperatures.

		2 θ (degree)	Crystallite size D (nm)	Lattice parameter, a (Å)		Interplanar spacing, d (Å)		Urbach energy E_U (meV)
				calculated	standard	calculated	standard	
α-CdS	as-grown	27.23	30	5.665	5.720	3.271	3.303	270
CdS	200°C	26.79	23	5.757	5.830	3.323	3.330	350
	300°C	26.70	20	5.776		3.335		260
	400°C	26.72	21	5.772		3.332		120

decreases from 30 to 20 nm with increasing annealing temperature, after that it increases from 20 to 21 nm when annealing temperature reaches to at 400 °C. The grain size of the films changed with changing annealing temperature. Similar grain size values of CdS films by deposited CBD have been reported by literature [16]. This may indicate that ion by ion deposition dominated the deposition process and as a result a much smaller grain size and thinner films were obtained [17].

It can be said that the crystallite sizes were depend upon annealing temperature. For α-CdS film, the lattice constant standard value a was 5.720 Å, interplanar spacing d was 3.303 Å and annealed CdS film the lattice constant standard value was 5.830 Å, interplanar spacing 3.330 Å in PDF card, respectively. An annealed temperature of 300 °C and 400 °C increased the lattice constant (5.776 Å and 5.772 Å), approaching this of the standard constant (5.830 Å). This indicates the relaxation of internal stress induced during film growth and the improvement of crystallinity in the CdS films [18]. Moreover, lattice parameters of the films were shifted smaller angels indicating an increase in the interplanar spacing from 3.271 to 3.335 Å with the increase in annealing temperatures. It can be said that this variation related to the microstrain, structural disorder, impurities, lattice defects, vacancies or deformation faults and rearrangement of the lattice due to annealing [19, 20].

3.2. Optical properties of the CdS films

The optical absorbance spectra of the CdS films by the CBD method as-grown and at three different annealing temperatures are given in Fig. 2.

It is shown that a sharp rise in absorbance occurs between 470 and 550 nm. These regions are called fundamental absorption edges. The fundamental absorption refers to band to band transition, i.e., to the excitation of an electron from the valance band to the conduction band.

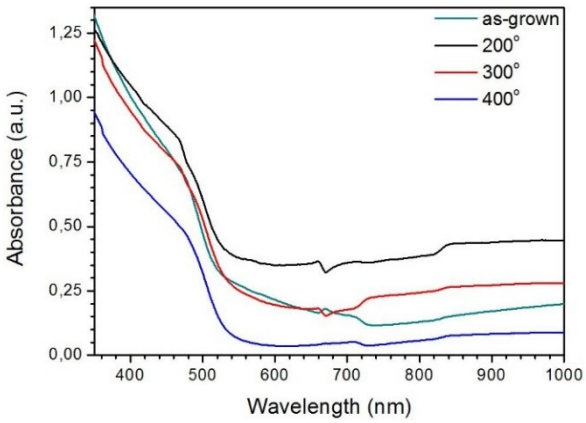


Figure 2. Optical absorbance spectra of the CdS films obtained as-grown and annealed at 200, 300 and 400 °C temperatures.

The fundamental absorption can be used to determine the optical energy gap (E_g) of the semiconductor films. The E_g of the CdS films are calculated on the basis of optical absorption spectra. The E_g values of the films were obtained from the absorbance measurements of the films by using the formula [21],

$$\alpha E \approx (E-E_g)^m \quad (3)$$

where α is the absorption coefficient, E is the incident photon energy on the films. The parameter m depends on the type of transition band equals to (1/2, 2, 3/2, 3), for allowed direct, allowed indirect, forbidden direct and indirect transitions, respectively. And also, the optical absorption coefficient of the sample with a direct transition band structure is larger than 10^4 cm^{-1}. $(\alpha E)^2$ versus E for the direct band gap on the energy axis. The E_g values were obtained by extrapolating the strait line to the E axis whose intercept to the E axis gives the E_g. The plot of $(\alpha E)^2$ versus E of CdS films are shown in Fig. 3. From the linear nature of the plot, the direct optical transition in CdS films is confirmed. The E_g is obtained by extrapolating the linear portion of the plot $(\alpha E)^2$ versus E of the energy axis at α=0. The E_g values are determined at 2.33 and 2.39 eV, which is similar to the other reports for CdS film [13, 17]. E_g of the as-grown film has 2.39 eV and

it decreases down to 2.33 eV with increasing annealing temperature up to at 300 ºC and higher temperature it starts to increase (2.39 eV) again suggesting annealing effect. The temperature dependent parameters that affect the E_g are reorganization of the film, volatilization of sulphur or self-oxidation of the CdS films [13]. Similar situation of CdS films deposited by CBD have been reported in literature [13]. This increment could be attributed to the increase in crystallite size, which induced a decrease in dislocation density [22].

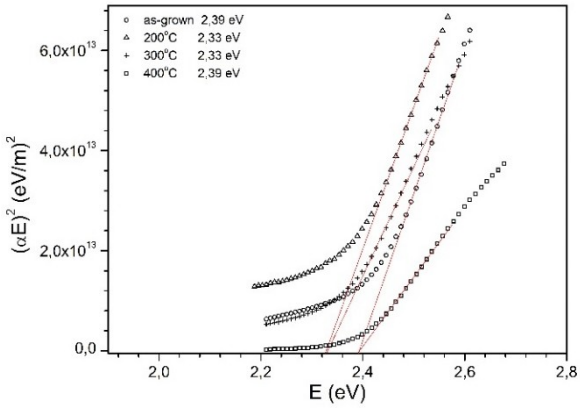


Figure 3. $(\alpha E)^2$ vs E and E_g values of the CdS films were obtained as-grown and annealed at 200, 300 and 400 ºC temperatures.

As a result of crystal structure defects into the semiconductor, the localized states available in the E_g of annealing films affects the band gap structure and optical transitions which reveals a tail for the density of states of either one of the valance and conduction band edge and the interactions with phonons. The Urbach energy values are calculated from the equations [23];

$$\propto (E) = \propto_o e^{\frac{E}{E_U}} \quad (4)$$

$$E_U = \left(\frac{dLn[\propto(E)]}{dE}\right)^{-1} \quad (5)$$

where α_o is a constant, E is the photon energy, and the Urbach energy E_U is determined from the reverse of the slope of the Ln(α) versus E for CdS films before and after annealing. E_U decrease from 350 to 120 meV with the increase in annealing temperatures in Fig. 4. The E_U values of the CdS films were given in Table 1. The similar results are come across in literature [16, 24]. The decrease in the amount of Urbach energies indicates the improvement of the crystallinity of the films. This means that there are a large number of defects in the films [24]. The E_U values change inversely with the E_g values of the CdS films. [22].

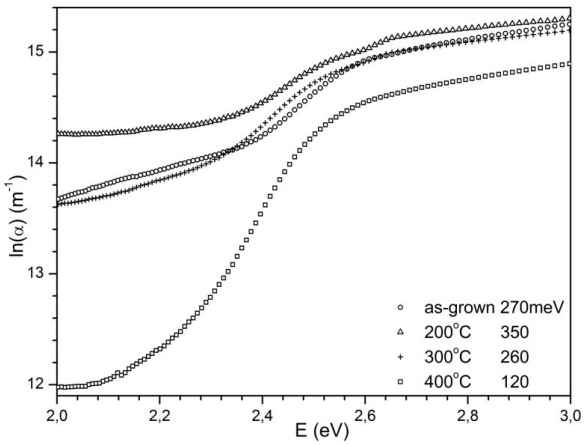


Figure 4. Ln(α) vs E and E_U values of the CdS films were obtained as-grown and annealed at 200, 300 and 400 ºC temperatures.

The optical transmission spectra of the CdS films by the CBD method as-grown and at three different annealing temperatures are given in Fig. 5.

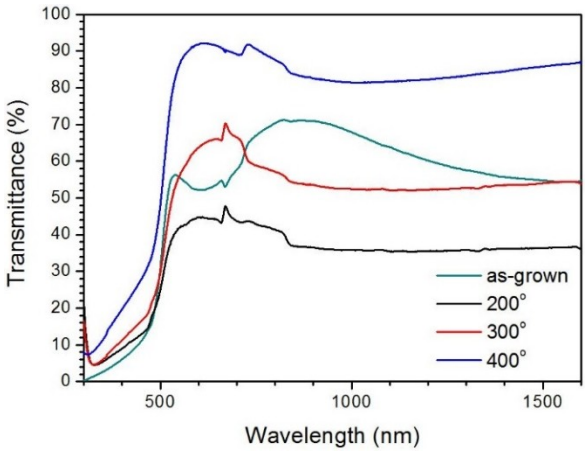


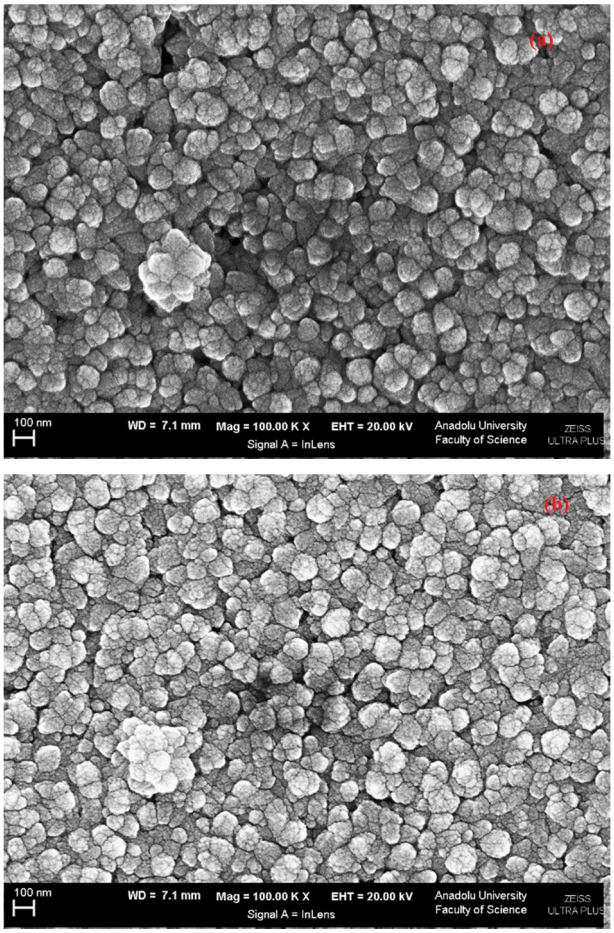
Figure 5. Optical transmittance spectra of the CdS films obtained as-grown and annealed at 200, 300 and 400 ºC temperatures.

All films have a sharp absorption edge near 500 nm wavelength, which corresponds to the energy band gap of films. The optical transmissions of the CdS films have been determined to be between 45% and 92% in the visible region. It can be seen that the transmission of the film annealed at 400 ºC being the better than the other three films. The optical transmission of as-grown film was approximately 70% and the annealed at 400 ºC showed above 90% of transmittance in the visible region. We compare the annealing films with different temperature, the transmittance of CdS films increases with the increasing temperature. In general, the optical transmittance of films is influenced mainly by the surface morphology as well as the density of the grains and defects at the grain boundaries. The number of defect states

would decrease with the increase in annealing temperature due to the improvement in the crystallite size as observed in the structural and surface morphological studies [25]. Also, due to the film thickness, structural properties, surface smoothness and defect density affect the transmission of the films [26].

3.3. Morphological properties of the CdS films

Fig. 6. (a-d) shows FESEM images of the CdS films at 100,000 magnifications with as-grown and three different annealing temperatures values. As illustrated in Fig. 6. (a-d), all the films have dense, homogeneous surface and round-grained structure. The films are well covered by many small cubic crystalline grains confirming the fact that the deposited mechanism takes place of ion-by-ion mechanism [27]. The FESEM images showed that all the films have good adherence on the substrates without pinholes or cracks. Films morphologies show not a significant change with increase annealing temperature.



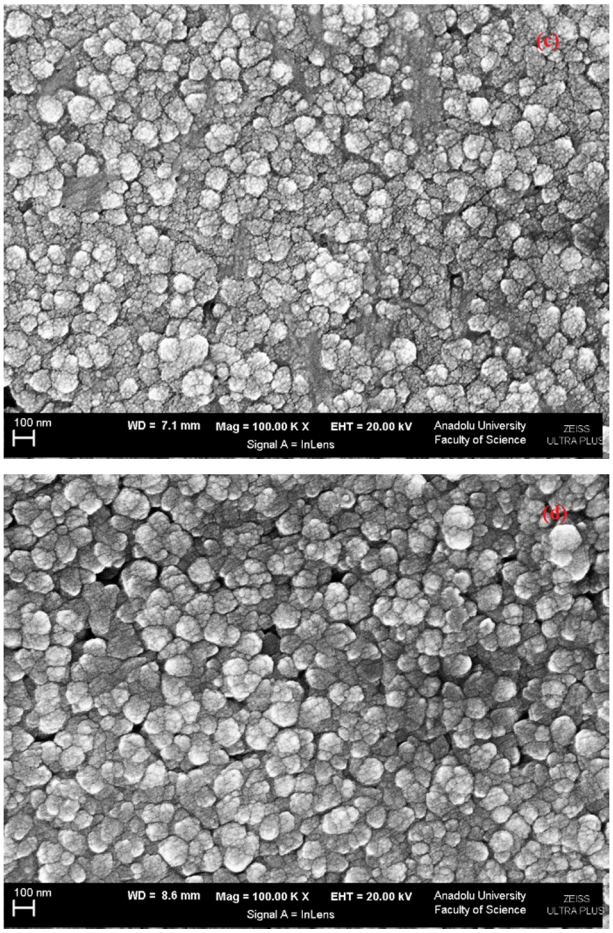


Figure 6. FESEM images of the CdS films obtained (a) as-grown, and annealed at (b) 200, (c) 300 and (d) 400 °C temperatures.

4. CONCLUSION

The CdS films were produced onto the glass slides as the substrates at 80 °C by the CBD method. The crystal structures of films were determined face centred cubic and the crystal structure of the films were observed to improve with increasing annealing temperature. X-ray diffraction studies revealed that with changing annealing temperature of the film and the CdS film structure changes from α-CdS (Hawleyite) to CdS (FCC). The grain size of CdS films were found to be between 21-30 nm. The band gap energies were estimated by assuming a direct transition and found to be slightly increasing with annealing temperature. The E_g values of the films varied from 2.33 to 2.39 eV. The Urbach energies of the films decrease with increasing annealing temperature in the range of between 350 and 120 meV. The reducing of the band bending values in the films is attributed to the decreasing defects in the crystal structure. This decrease is slightly more as the annealing temperature increases which indicates an important in the quality of CdS films on annealing. With increasing annealing temperature of the

films, the transmittance value was observed to reach above 90% at 400 ºC. The FESEM images showed that all the films have good adherence on the substrates without pinholes or cracks. Our results show that the best annealing temperature for CdS films by CBD in 400 ºC in nitrogen atmosphere. Low EU value, the transmittance (92%) and wide band gap (2.39 eV) obtained for the film produced in 400 ºC annealing temperature. It can be suggested for the application in window material for solar cells.

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