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"Variation in the structure and optical properties of CdO thin film with temperature"

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Abstract
CdO thin film has been deposited by using chemical bath deposition method on the glass substrate. The effect of temperature of annealing (573, 623 and 673 K) on the structural and optical properties of the films has been discussed. The structure of crystal is studied by using X-ray diffraction technique. The nature of annealed CdO films is polycrystalline with cubic structure. It is having a preferential orientation along (111) plane. The analysis of X-ray diffraction technique shows that the intensities of peaks of the crystalline phase have raised by increasing of annealing temperature. The parameters of structure for the CdO thin films as the grain size, strain, dislocation density, and texture coefficient were calculated and discussed. The particle size of the preferential orientation at annealing temperature 623 K is 16.95 nm while that for the annealing temperature 18.24 nm is 673 K. The study of optical properties of CdO thin films by using the technique of ultraviolet-visible spectrophotometer indicates blue shift in the energy band gap which is the clear indication of quantum confinement. It is found that the band gap energy decreases by increasing the annealing temperature. It means that the band gap energy is inversely proportional to the annealing temperature.

Keywords: CdO thin film, CBDM, annealing, structure and optical properties.

1. Introduction

It is observed that nanocrystalline semiconducting materials attracted a great deal of attention due to their size dependent properties and wide range of potential applications. A good number of technical advancements in the field of nanostructured materials have stimulated the wide range of research interest in recent years due to various new properties shown by them (Kathalingam *et. al.* 2010). Now-a-days, nanostructured semiconductors are used to design good varieties of device for microelectronics. Transparent conducting oxides (for example ZnO, SnO_2, BaO, Fe_2O_3, BiClO, Cu_2O and CdO) have long been a subject of different investigations because of its unique physical properties and their potential applications in commercial devices such as gas sensor, liquid crystal displays, phototransistors, solar cells, infrared detectors and anti-reflection coatings (Elttayef *et. al.* 2013 and Ghosh 2017). It is experimentally found that Cadmium oxide (CdO) is one of the promising transparent conducting oxides (TCOs) from II-VI group of semiconductor materials (Abdi *et. al.* 2015). Cadmium oxide (CdO) is an n-type semiconductor with a rock-salt crystal structure (face centered crystal) (Abdi *et. al.* 2015 and Ziabaria *et. al.* 2011). In this crystal, each cadmium or oxygen (Cd or O) ion is surrounded by 6 neighbors (Abdi *et. al.* 2015). Cadmium oxide (CdO) is an important semiconductor material for the development of various technologies of solid-state devices (panel display, optoelectronic components, thermally insulating glass (Elttayef *et. al.* 2013). Cadmium oxide (CdO) has its potential applications in the field optoelectronics devices such as solar cells, phototransistors, photodiodes, transparent electrodes, catalysts and gas sensor (Buba *et. al.* 2015). In recent years, Cadmium oxide (CdO) has become most attention material because of its transparent conducting oxide due to following:

a) easy in doping,
b) high carrier concentration,
c) chemical stability in hydrogen plasma,
d) abundance in nature and no toxicity (Elttayef *et. al.* 2013),

e) high electrical conductivity, (i.e. low electrical resistivity) (Elttayef *et. al.* 2013 and Habubi 2011),
f) high optical transmittance in the visible region of the spectrum, and moderate refractive index (Buba *et. al.* 2015) and
g) a direct band gap (~ 2.5 eV) (Elttayef *et. al.* 2013), 2.2eV to 2.5eV (Buba *et. al.* 2015), 2.2 and 2.7 eV (Habubi 2011), and an indirect band gap 1.98 eV (Lanje *et. al.* 2011), 1.36 eV (Radi *et. al.* 2006), 1.36-1.98 eV (Tadjarodi *et. al.* 2012).

In past years, different research groups around the world are working on the synthesis of different II-VI n-type transparent semiconducting oxide thin films in different process (Ghosh 2017). There are various methods for producing Cadmium oxide (CdO) as reported in the literature. By using these methods such as Physical, chemical and thermal hydrothermal methods, template assisted method, solvothermal method, thermal disruption method, photosynthetic method (Basgoz *et. al.* 2019), ultrasonic spray pyrolysis, electro deposition, chemical bath deposition, vacuum evaporation, Sol. Gel deposition method (Alkhayatt *et. al.* 2014 and Habubi 2015), Cadmium oxide (CdO) acquisition at Nano size is made possible practically. In this work the effect of annealing in air on structure and optical properties is studied and discussed in detail.

2- Experimental Details

Synthesis of CdO samples: The Cadmium oxide (CdO) thin films were deposited on a microscope glass slide of suitable dimension (25mm x 75mm x 1mm). The cleaning of substrates is done by using water and detergent solution (chromic acid) and then boiled for an hour and after that it is cleaned by using deionized water. In the last, it is immersed in acetone and rinsed with deionized water respectively to remove the contaminations at surface in order to make the surface more conductive to have uniform film deposition.

The chemical bath deposition method (CBD) permits the deposition of very thin films. The deposition of very thin films is of order of few nanometers. This method is very easy and inexpensive solution growth technique. The physical properties of the chemical deposition of Cadmium oxide (CdO) films depends upon the growth parameters like temperature of bath, the relative concentrations of the various reactants in the solution, pH value and type of substrate also. The precursors which are used for the preparation of solution of Cadmium oxide (CdO) are cadmium chloride monohydrate, Sodium hydroxide, liquid (Ammonium hydroxide solution). The deionized water having pH 11, is used as the medium of solution preparation. In the present case, $CdCl_2$ for 60 ml of solvent medium (distilled water) with the help of magnetic stirrer (80 rpm) were used. The pH was determined with a pH meter and controlled to obtain the pH value 11. For film deposition, the substrates were immersed in such a way that it is inclined vertically at 20^o angle to the wall beaker and we add 3 ml of Hydrazine Hydrate for good adhesion. The bath temperature was maintained at around 70°C and the deposition time was about 10 hrs. From these conditions uniform film deposition on all substrates was achieved.

Characterization techniques: After synthesis the coated films were proceeded for the characterization such as structural studies, X-ray diffraction through a computer software (MDI/JADE 5) as general X-ray diffractometer (λ = 1.54 Å for Cu-Kα, current value 20.4 mA and voltage of 36.3 x 10^3 V). The range of scan in 2Θ is of 3-75^o with scanning speed of 4^o/min. The optical studies as well as optical absorption of Cadmium oxide (CdO) thin films studies were taken out by analyzing a computer software Varian 50 conc. For spectral study, we have used the UV-vis spectrophotometer in the wavelength range 300-1100 nm at room temperature. The Carbolite electric oven having specification 50-60 Hertz, 6990 Watts, 220 Volt, maximum temperature is 1300 ^{o}C, is used for annealing film. The thickness of the Film is calculated by gravimetric weight difference method. It is found to be 330 nm.

3- Theory and calculation

3.1 Structural Parameters

Interplanar Distance (d): The interplanar distance is calculated by using (Yacobi 2004) following relation-

$$2d \sin \Theta = n\lambda \qquad (1)$$

Where n is order of the corresponding reflection which is an integer, λ is the wavelength of the radiation, d is the periodic spacing between the planes, and Θ is the Bragg's angle.

Lattice Constant (a): Lattice constant (a) refers to the physical dimension of unit cells in a crystal lattice, for cubic structure is calculated using the relation (Barman *et. al.* 2008)-

$$a = d(h^2 + k^2 + l^2)^{1/2}\pi r^2 \qquad (2)$$

Dislocation Density ($\boldsymbol{\delta}$): Dislocation density is defined as the dislocation line length in the unit volume and is a measure of the number of defects in the crystal. The dislocation density by using the simple approach of Williamson and Smallman (Malliga *et. al.* 2014)-

$$\delta = \frac{1}{D^2} \qquad (3)$$

Crystallite Size (D): The crystallite size (D) is calculated by using Scheerer's formula (Dantus *et. al.* 2008)-

$$D = \frac{0.9\lambda}{\beta \cos\theta} \qquad (4)$$

Where λ is the x-ray wavelength of Cu-Kα source (λ= 0.154056 nm), Θ is the Bragg's angle and β is the full width at half maximum of the diffraction peak in radians.

Average Strain ($\boldsymbol{\varepsilon}$): The average strain is calculated from the following relation (Shanmugavel *et. al.* 2014)

$$\varepsilon = \frac{\beta \cot\theta}{4} \qquad (5)$$

Texture Coefficient (TC): The textured coefficient (TC) of Cadmium oxide (CdO) thin films is calculated to quantify the preferential orientation of the film, from the following relation (Jassim *et. al.* 2013)-

$$TC(hkl) = \frac{I(hkl)/I_0(hkl)}{\sum_n I(hkl)/I_0(hkl)} N \qquad (6)$$

Where (I) is measured intensity, (I_o) is the (JCPDS) standard intensity, N reflection number of the peaks and n is the number of diffraction peaks.

Equation (6) shows that TC(hkl) approaches unity for a randomly distributed powder sample, while TC(hkl) is larger than unity if the (hkl) plane is preferentially oriented.

3.2 Optical Absorption

Optical Absorption Coefficient (α): The optical absorption coefficient (α) is calculated for thin films using the following equation (Nadeem *et. al.* 2004)-

$$I_t = I_o' \exp(-\alpha t) \qquad (7)$$

Where t is the film thickness, I_t and I_o' are the intensity of transmitted light and initial (incident) light, respectively. Also, Absorption coefficient is calculated by using Lambert law given as follows (Shind *et. al.* 2011 and Mohammad *et. al.* 2013)-

$$\alpha = 2.3026\, A/t \qquad (8)$$

Where A is absorbance, and t is the film thickness.

Extinction Coefficient (k): The extinction coefficient (k) depends mainly on absorption coefficient according to the following relation (Benramdane *et. al.* 1997, Jawad *et. al.* 2010 and Wolkenstein 1991)-

$$k = \frac{\alpha\lambda}{4\pi} \qquad (9)$$

Where λ is the wavelength of incident light.

Optical Band Gap: The fundamental transmission that corresponds to electron excitation from the valence band to conduction band, can be used to determine the nature and value of the optical band gap. The relation between the absorption coefficient (α) and the incident photon energy (hν) can be written by Tauc equation (Bhosale *et. al.* 2005 and Lalithambika *et. al.* 2014)-

$$\alpha h\nu = B(h\nu - E_g)^n \qquad (10)$$

Where B is a constant, hν is the photon energy, E_g is the band gap of the material, and the exponent n depends on the type of transition. N = 1/2, 2, 3/2, and 3 corresponding to allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions, respectively.

Particles Size of Nanoparticles: The particle sizes is calculated using Brus relation (Al-Hussam *et. al.* 2012)-

$$E_g^* = E_g + \frac{\pi^2 h^2}{8R^2}\left(\frac{1}{m_e^*} + \frac{1}{m_h^*}\right) - \frac{1.8e^2}{\varepsilon R} \qquad (11)$$

Where E_g^* is the size dependent band gap, E_g is the energy band gap of the bulk crystal, R is the radius of nanoparticles, m_e^* and m_h^* are the effective masses of electron and hole respectively ($0.21m_e^*$ for CdO), ε is the dielectric constant (6.07 for CdO) (Parvathi *et. al.* 2013).

4- Results and Discussion

Effect of Annealing on Structure of CdO Thin Films

Figure 1, shows X-ray diffraction (XRD) pattern of prepared Cadmium oxide (CdO) thin films deposited with concentration of $CdCl_2$ (0.1 Mol.) and deposition time is about 10 hrs, deposition temperature (70° C), were annealed at (573, 623 and 673 K) for 2 hrs, respectively. Bragg's peaks observed at (623, 673 K) at 2ϴ ≈ (33°, 38.318°, 55.282°, 65.958°, 69.201°), (33.118°, 38.399°, 55.361°, 66.038°, 69.359°), corresponded to the (111), (200), (220), (311), and (222) planes shows that the studied films are polycrystalline in nature with a cubic structure (Zaien *et. al.* 2013). The preferred orientation of the film is (111) plane as shown in Table 1. But at temperature 573 K, it is found that the film is amorphous, however when the annealing temperature increases the Bragg's peaks are slightly shifted in the direction of higher 2ϴ values and its intensity increases and get sharp peaks with slight improvement in the crystallinity (Gokul *et. al.* 2013). The observed d-spacing and lattice constant (a) are in good agreement with the standard data of Cadmium oxide (CdO) (Sahoo 2022) as given in Table 1. The FWHM (β) values decreases with the increase in annealing temperature as shown in Table 2. Texture coefficient (TC) was observed in the range of 0.83-1.13 nm, (see Table 2), with the increase in annealing temperature, the (TC) slightly also increases. Crystallite size (D) was found to be in the range (16.95- 21.26 nm) (See Table 2), as the annealing temperature increases the crystallite size increases as shown in Figure 2. Average strain (ε) was found to be in the range (0.00286-0.00721) (See Table 2). As the annealing temperature increases the average strain decreases. Dislocation density (G) was found to be in the range (0.00221-0.00348 nm^{-2}) (See Table 2). As annealing temperature increases the dislocation density decreases. All results of structural parameters are in good agreement with the literature references (Gokul *et. al.* 2013).

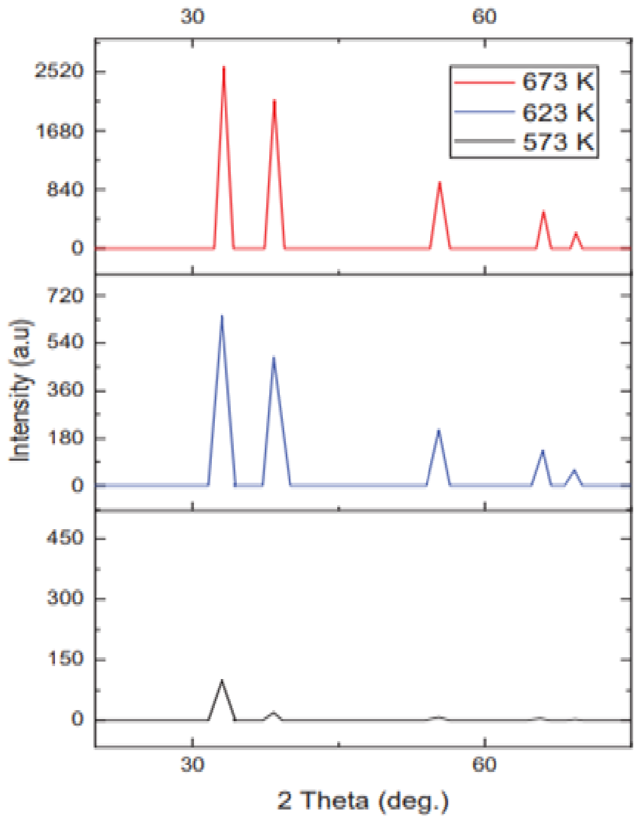

Fig. 1: XRD pattern for annealed CdO thin film at different annealing temperature.

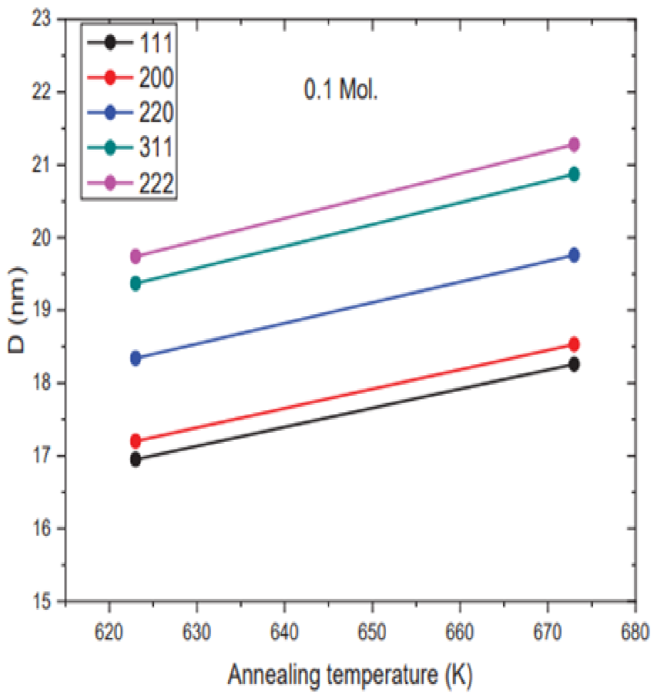

Fig. 2: Variation grain size with different annealing for CdO films.

Table 1: Comparison of observed and standard data of some XRD parameters for CdO thin film at different annealing temperature.

Annealing temperature (K)	2Θ Observe (deg.)	2Θ Standard (deg.)	d Observe (nm)	d Standard (nm)	a Observe (nm)	a Standard (nm)	h k l	1% Observe	1% Standard
623	33	33.019	0.2714	0.27106	0.4701	0.46948	1 1 1	100	100
	38.318	38.321	0.2349	0.23474	0.4698		2 0 0	74.9	84
	55.282	55.299	0.1662	0.16599	0.47		2 2 0	39.5	45.2
	65.958	65.937	0.1417	0.14157	0.4699		3 1 1	23.5	28.5
	69.201	69.272	0.1358	0.13554	0.4704		2 2 2	10.2	12.2
673	33.119	33.021	0.2706	0.27108	0.4685	0.46948	1 1 1	100	100
	33.400	38.324	0.2345	0.23476	0.4688		2 0 0	81.7	85
	55.262	55.301	0.1661	0.16601	0.4694		2 2 0	36.6	45.4
	66.039	65.937	0.1416	0.14157	0.4710		3 1 1	20.6	28.6
	69.361	69.272	0.1356	0.13555	0.4694		2 2 2	8.6	12.4

Effect of Annealing on Optical Properties of Cadmium oxide (CdO) Thin Film: The optical spectra were studied by using UV-vis spectrophotometer for Cadmium oxide (CdO) thin film as a function of wavelength in the range (300-1100 nm), annealing at different temperature (623, 673 K) and deposition time was about 10 hrs, as given in Table 3. Figure 3 shows that the absorbance (A) spectra of the prepared film increase with increasing of annealing temperature, due to increase in the grain size. Figure 4 shows that the transmittance (T) spectra decrease with increasing of annealing temperature. The increase in transmittance with increasing of wavelength in UV region is not sharp. This indicates that the absorption band gap transitions are due to direct and indirect transitions, which is characteristic of Cadmium oxide (CdO) (Dhawale *et. al.* 2008 and Dakhel *et. al.* 2003). The fundamental transmission, which corresponds to electron excitation from the valence band to conduction band, can be used to determine the nature and value of the optical band gap (Perumal *et. al.* 2012 and Sivaram 2018). Figure 5 shows the variation absorption coefficient (α) with wavelength for the coated films. The absorption coefficient (α) values are found to be of order 10^4 cm^{-1} for two samples, and increases with increasing of annealing temperature. Figure 6 shows extinction coefficient (k) as a function of wavelength (λ), and increases with increasing of annealing temperature. Figure 7, show typical $(\alpha h\nu)^2$ plot for direct energy gap at different annealing temperature. The energy gap was found to be (2.95, 2.87eV), Table 3.

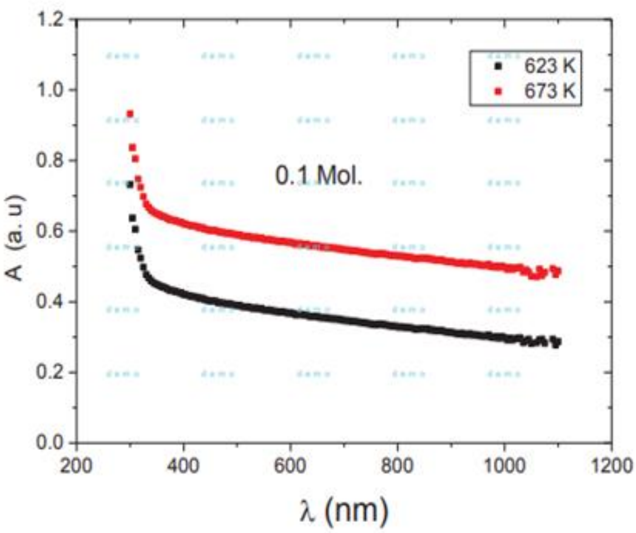

Fig. 3: UV -vis absorbance (A) spectra of CdO thin films deposited at different annealing temperature.

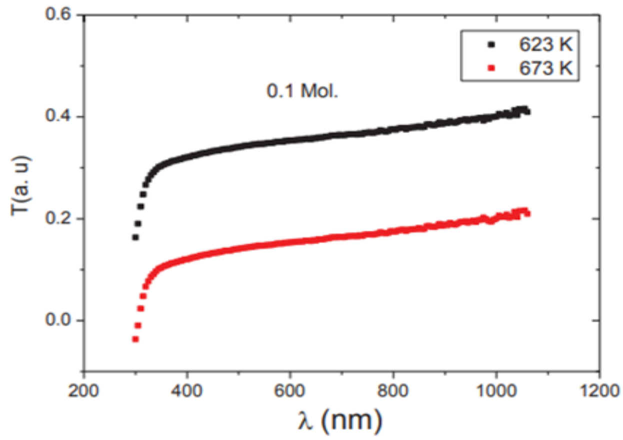

Fig. 4: UV -vis Transmittance (T) spectra of CdO thin films deposited at different annealing temperature.

Figure 8, show $(\alpha h\nu)^{1/2}$ vs. $(h\nu)$ plot for indirect energy gap at different annealing temperature. The indirect energy gap obtained was (1.945, 1.914 eV), Table 3. It is observed that the band gap energy decreases with increasing of annealing temperature as shown in Figure 9. This is may be due to the increase in the carrier concentration and also may be due to its quantum confinement effect (Gokul *et. al.* 2013). The values of calculated particle size for direct energy gap are (9.411-10.05 nm), Table 3. These values are slightly different with the sizes determined from XRD and increase with increasing of annealing temperature, Figure 10 shows, the particle size for indirect energy gap are (9.921-10.194 nm), Table 3.

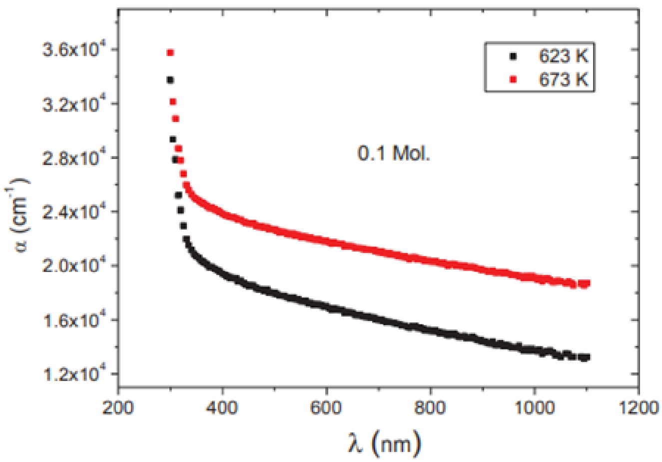

Fig. 5: Absorption coefficient (α) as a function of wavelength (λ) deposited at different annealing temperature.

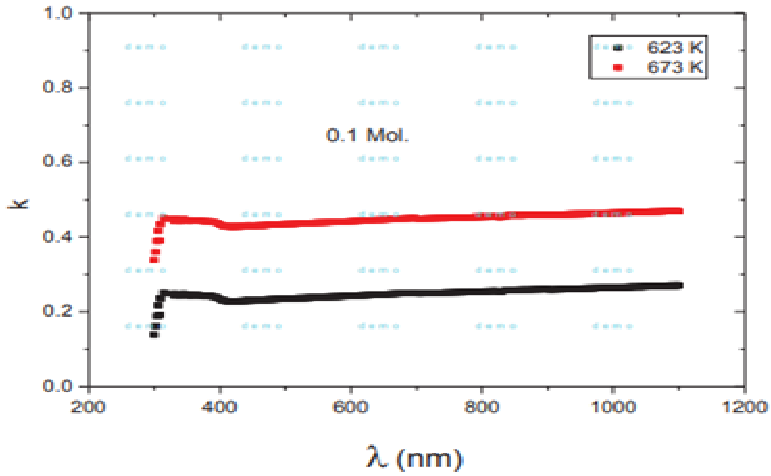

Fig. 6: Extinction coefficient (k) as a function of wavelength (λ) for CdO thin films deposited at different annealing temperature.

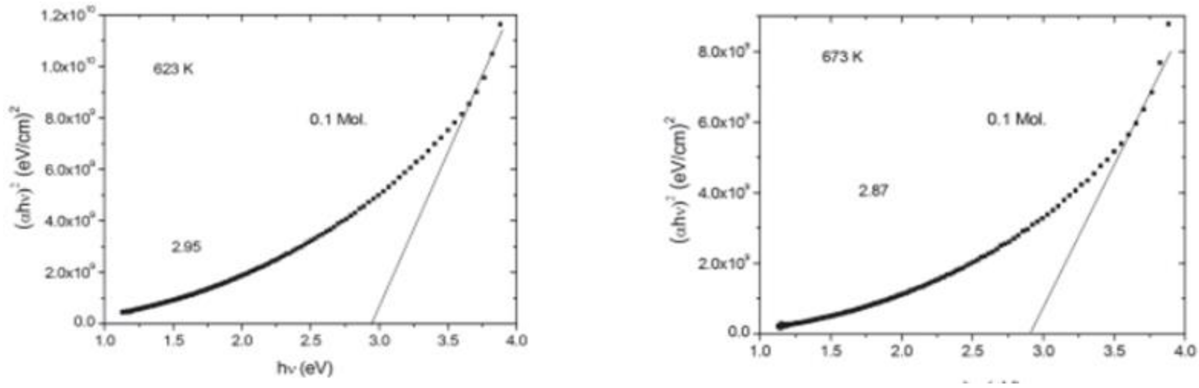

Fig. 7: A direct band gap of CdO thin film deposited at different annealing temperature.

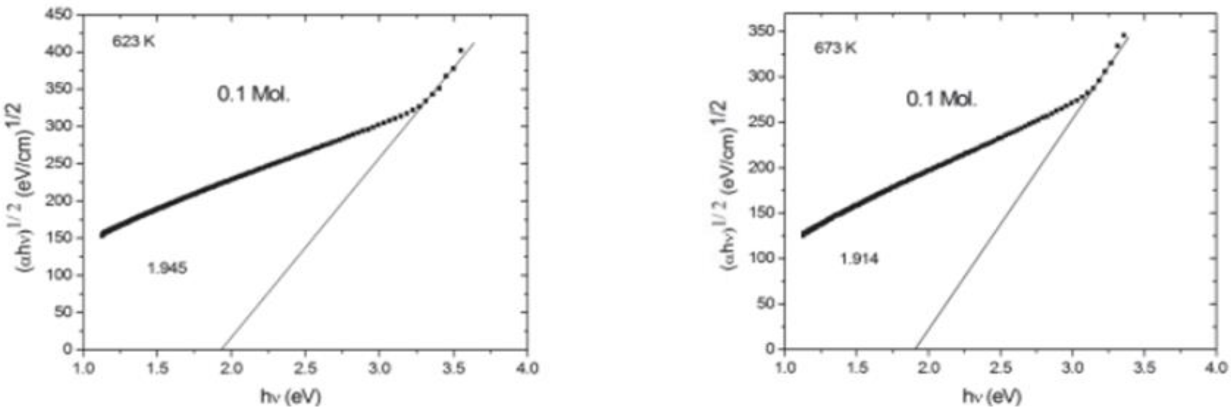

Fig. 8: An indirect band gap of CdO thin film deposited at different annealing temperature.

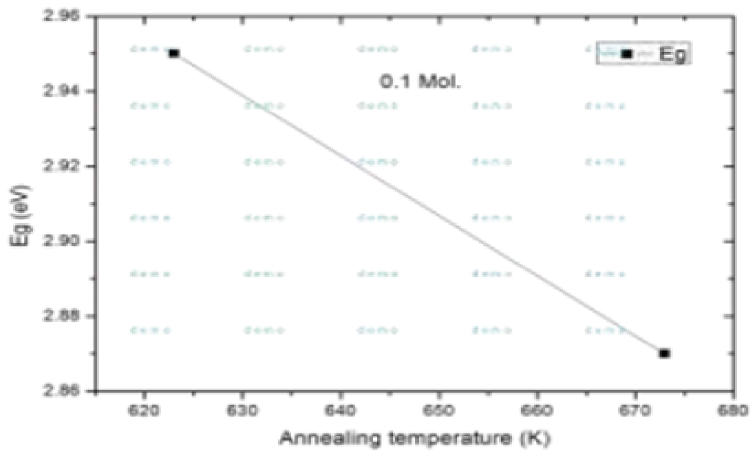

Fig. 9: Variation of energy gap with annealing temperature.

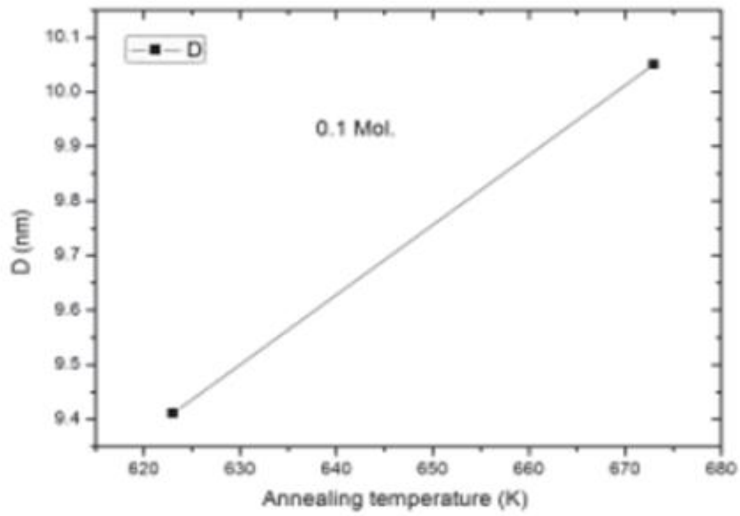

Fig. 10: Variation of particle size with annealing temperature.

Table 3: Experimental values of different energy gap, blue shift and particles size, for Cadmium oxide (CdO) thin films at different annealing temperature.

Annealing temperature (K)	Direct energy gap E_g^* (eV)	Blue shift E_{shift} (eV)	Particle size D (nm)	Indirect energy gap E_g^* (eV)	Blue shift E_{shift} (eV)	Particle size D (nm)
623	2.95	0.65	9.411	1.945	0.585	9.921
673	2.87	0.57	10.05	1.914	0.554	10.194

5 Conclusion

Cadmium oxide (CdO) thin film of high quality and grain size in the range of nanoparticles has been successfully prepared on glass substrate by CBD at different annealing temperatures. The X-ray diffraction (XRD) patterns confirmed the polycrystalline cubic Cadmium oxide (CdO) phase formation and the preferred orientation is (111) with crystallite size is (16.95- 18.24 nm). It was noticed that the annealing temperature increases the intensity of peaks and get sharper with slight improvement in crystallinity and crystallite size increases. UV-vis spectrophotometer show that the absorption spectra of CBD (CdO) thin film increases and the energy gap values decreases, with increasing of annealing temperature.

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