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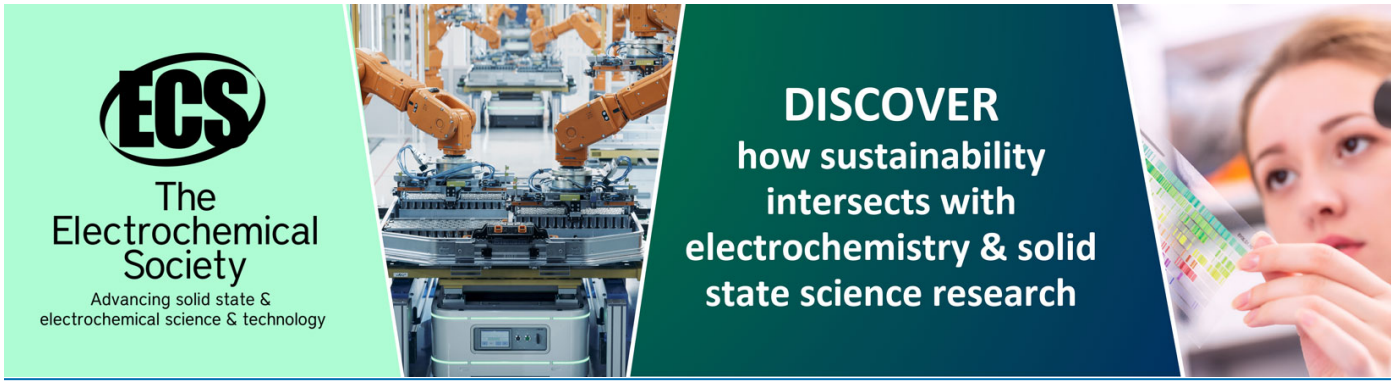

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Effect of annealing temperature on structure and optical properties of CdO nanocrystaline thin film prepare by chemical bath deposition method

Salah Abdul-Jabbar Jassim[a], Eman Mohammed Ali Nassar[b]
[a] Department of Optical Technology , College of Health and Medical Technology ,Al-Ayen University ,Thi-Qar , Iraq
[b] Department of physics, College of Education and Applied Science, Hajjah University, Hajjah ,Yemen
[a]Email; salahjassim200@yahoo.com

Abstract

CdO thin film has been deposited by chemical bath deposition method (CBD) on the glass substrate. Effect of annealing temperature(573,623 and 673 K) on the structural and optical properties of the films has been investigated. The crystal structure investigated by X-ray diffraction method. Annealed CdO films are polycrystalline in nature with cubic structure having a preferential orientation along (1 1 1) plane. Analysis of XRD indicates that the intensities of peaks of the crystalline phase have increased with the increasing of annealing temperature. The structural parameters for the CdO thin films as the grain size, strain, dislocation density, and texture coefficient were calculated. Particle size for the preferential orientation at different annealing temperature is (16.95, 18.24 nm) for annealing temperature 623 and 673 K respectively. Optical properties study of CdO thin films using UV-vis spectrophotometer shows blue shift in the energy band gap as an indication of quantum confinement. It is observed that the band gap energy decreases with increasing of annealing temperature. The direct and indirect band gap energy values were found to be (2.95 2.87) and (1.945,1.914) eV respectively.

Keywords ; CdO thin film, chemical bath deposition method ,annealing, structure properties and optical properties

1- Introduction

Nanocrystalline semiconducting materials have attracted a great deal of attention because of their size dependent properties and wide range of applications [1]. Numerous technical advancements in the field of nanostructured materials have stimulated the wide range of research interest in recent years because of various new properties exhibited by them. Recently, nanostructured semiconductors are widely used to design rich varieties of device for microelectronics.. Transparent conducting oxides (TCOs) such as

ZnO, SnO_2, BaO, Fe_2O_3, $BiClO$, Cu_2O, and CdO have long been a subject of various investigations due to its unique physical properties and applications in commercial devices, phototransistors, gas sensor, solar cells, liquid crystal displays, IR detectors and anti-reflection coatings [2,3]. Cadmium oxide (CdO) is one of the promising TCOs from II-VI group of semiconductors [4]. CdO is an n-type semiconductor with a rock-salt crystal structure (fcc) [4,5], in which each Cd or O ion is surrounded by 6 neighbors [4]. CdO is an important semiconductor material for the development of various technologies of solid-state devices (panel dis- play, optoelectronic components, thermally insulating glass [3]. CdO finds its potential applications in the field optoelectronics devices such as solar cells, phototransistors, photodiodes, transparent electrodes, catalysts and gas sensor [6]. Recently CdO has attracted attention as a transparent conducting oxide because of its (i) a direct band gap (~2.5 eV) [3], 2.2eV to 2.5eV [7], 2.2 and 2.7 eV [8], and an indirect band gap 1.98 eV [9], 1.36 eV [10], 1.36-1.98 eV [11] (ii) high electrical conductivity, (i.e. low electrical resistivity) [3,8] (iii) easy in doping, (iv) chemical stability in hydrogen plasma, (v) abundance in nature and no toxicity [3],(v1) high carrier concentration,(v11) high optical transmittance in the visible region of the spectrum, and moderate refractive index [7]. Recently, various research groups around the world are working on the synthesis of several II-VI n-type transparent semiconducting oxide thin films in different process [2].There are many different methods for producing CdO in the literature. As a result of these methods, CdO acquisition at Nano size is made possible. Physical, chemical and thermal hydrothermal methods, template assisted method, solvothermal method, , thermal disruption method, photosynthetic method [12], ultrasonic spray pyrolysis, electro deposition, chemical bath eposition, vacuum evaporation, Sol.Gel deposition method [13,14]. In this work the effect of annealing in air on structure and optical properties were studied.

2- Experimental

2.1 Synthesis of CdO samples

The CdO thin films were deposited on Microscope glass slides of ($25mm \times 75mm \times 1mm$).The cleaning of substrates using water and soap, detergent solution (chromic acid) and boiling for one hour and clean by De-ionized water. Finally, immerse in acetone and rinsed with De-ionized water respectively to remove the surface contaminations to make the surface more conductive for uniform film deposition.

CBD allows the deposition of very thin films, of the order of a few nanometers and it is an easy and inexpensive solution growth technique. The physical properties of the chemical deposition of CdO films are dependent upon the growth parameters such as the bath temperature, the relative concentrations of the various reactants in the solution the pH value and the type of substrate. Precursors used for CdO solution preparation are cadmium chloride monohydrate ($CdCl_2$,H_2O, Merck, 98%) {Cd^{+2} ion source}, Sodium hydroxide ($NaOH$, Scharlau, 99%). {O^{-2} ions source}, liquid (Ammonium hydroxide solution) (NH_4OH, Fluka-Chemicka, purum,~28% in water). (pH 11), De-ionized water used as the medium of solution preparation. In our case, $CdCl_2$ (0.1 $Mol.$) for 60 ml of solvent medium (distilled water) with the help of magnetic stirrer (80 rpm). The pH was determined with a pH meter and controlled to obtain pH =11. For the film deposition, the substrates were immersed inclined vertically at 20° 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 10 hrs. From these conditions uniform film deposition on all substrates was achieved.

2.2 Characterization techniques

Then the coated films were proceeding for characterization studies such as structural studies, X-ray diffraction using a computer software (MDI/JADE 5) Pgeneral XD-2 X-ray diffractometer (λ=1.54056 Å for Cu-Kα, current: 20.4 mA, voltage: 36.3 kV). The range of scan in 2θ of 3-75° with scan speed $4°/min$. Optical studies, the optical absorption of CdO thin films studies were carried out by analyzing a computer software (Cary Win UV scan software) Varian 50 conc. UV-vis spectrophotometer in the wavelength range 300-1100 nm at room temperature. The Carbolite CWF 13/23 electric oven (50-60 Hertz, 6990 Watts, 220 volt, maximum temperature is 1300 ℃) is used for annealing film. Film thickness is calculated by gravimetric weight difference method, it is 330 nm.

3- Theory and calculation

3.1 Structural Parameters

3.1.1 Interplanar Distance (d)

The interplanar distance is calculated by using [15]

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

where n is order of the corresponding reflection, and is an integer, λ is the wavelength of the radiation, d is the periodic spacing between the planes (interplanar distance), and θ is the Bragg's angle.

3.1.2 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 [16]

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

3.1.3 Crystallite Size (D)

The crystallite size (D) is calculated by using Scheerer's formula [17]

$$D = \frac{0.9\,\lambda}{\beta \cos\theta} \qquad (3)$$

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 (FWHM) of the diffraction peak in radians.

3.1.4 Dislocation Density (δ)

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 (δ) using the simple approach of Williamson and Smallman [18]

$$\delta = \frac{1}{D^2} \qquad (4)$$

3.1.5 Average Strain ($\boldsymbol{\varepsilon}$)

It is calculated from the following relation [19]

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

3.1.6 Texture Coefficient (TC)

Textured coefficient (TC) of CdO thin films is calculated to quantify the preferential orientation of the film, from the relation [20]

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

where (I) is measured intensity, $(I_{\circ})$ is the (JCPDS) standard intensity, N reflection number of the peaks and n is the number of diffraction peaks . Eq.(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

3.2.1 Optical Absorption Coefficient (α)

The optical absorption coefficient (α) is calculated for thin films using the equation [21]

$$I_t = \grave{I}_{\circ} exp(-\alpha t) \qquad (7)$$

Where *t* is the film thickness, I_t and $\grave{I}_{\circ}$ are the intensity of transmitted light and initial (incident) light, respectively.

Also, Absorption coefficient is calculated using Lambert law [22,23]

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

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

3.2.2 Extinction Coefficient (*k*)

The extinction coefficient (*k*) depends mainly on absorption coefficient according to the relation [24,25]

$$k = \alpha\lambda/4\pi \qquad (9)$$

Where λ is the incident light wavelength.

3.2.3 Optical Band Gap

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. The relation between the absorption coefficient (α) and the incident photon energy ($h\nu$) can be written by Tauc equation [26,27].

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

Where B is a constant, $h\nu$ 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.

3.2.4 Particles Size of Nanoparticles

The particle sizes is calculated using Brus relation [28]

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

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*) [29].

4- Results and Discussion

4.1 Effect *of Annealing on Structure of CdO Thin Films*

Fig.(1) shows XRD pattern of prepared CdO thin films deposited with concentration of $CdCl_2$ (0.1 $Mol.$) and deposition time (10 hrs), deposition temperature (70 ℃), were annealed at (573, 623 and 673 K) for (2 hrs), respectively,. Bragg's peaks observed at (623, 673 K) at $2\theta \cong$ (33°, 38.318°, 55.282°, 65.958°, 69.201°),(33.118°, 38.399°, 55.361°, 66.038°, 69.359°), corresponded to the (1 1 1), (2 0 0), (2 2 0), (3 1 1), and (2 2 2) planes indicates that the studied films are polycrystalline in nature with a cubic structure [30]. The preferred orientation of the film is (1 1 1) plane, Table (1). But at (573K) it is noticed the film is amorphous, but 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 sharper with slight improvement in crystallinity [31]. The observed d-spacing and lattice constant (a) in good agreement with the standard data of CdO (JCPDS file no.75-0592) [32], Table (1). The FWHM (β) values decreases as annealing temperature increases, Table (2). Texture coefficient (TC) was found to be in the range (0.83-1.13), Table (2), as the annealing temperature increases the (TC) slightly increases. Crystallite size (D) was found to be in the range (16.95-21.26 nm), Table(2). As the annealing temperature increases the crystallite size increases, Fig.(2). Average strain (ε) was found to be in the range (0.00286-0.00721), Table (2). As the annealing temperature increases the average strain decreases. Dislocation density (δ) was found to be in the range (0.00221-0.00348nm^{-2}), Table (2). As annealing temperature increases the dislocation density decreases. All results of structural parameters are in good agreement with the literature references [31].

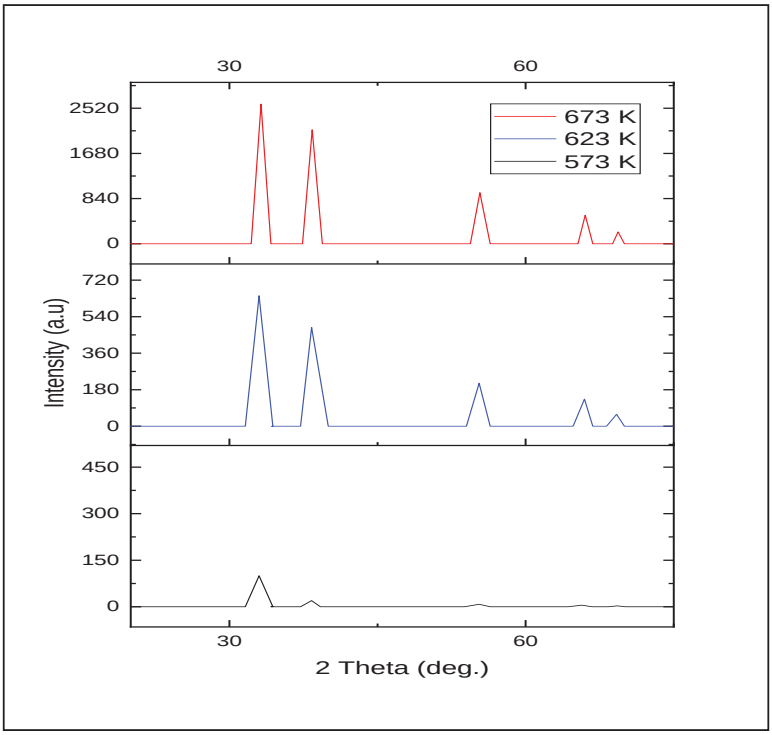


Fig.(1): XRD pattern for annealed CdO thin film at different annealing temperature.

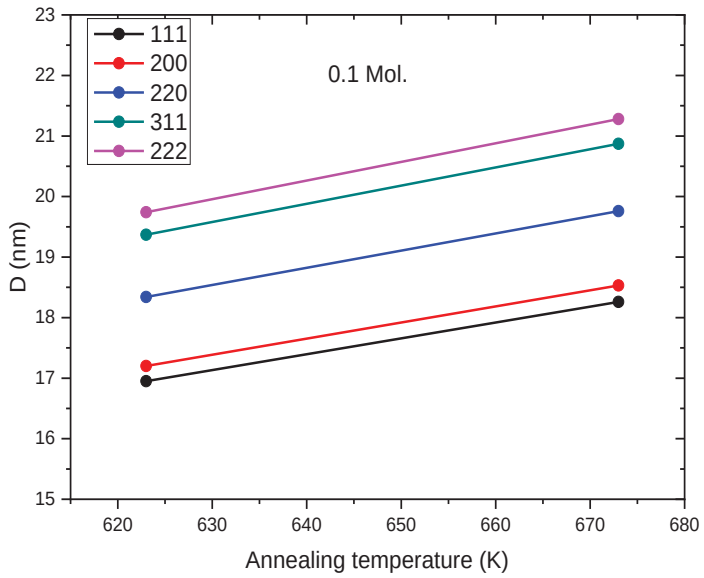


Fig.(2): Variation grain size with different different annealing for CdO films

*T*able (1): Comparison of observed and standard of some XRD parameters for CdO thin film at different annealing temperature

Annealing temperature (K)	2θ Observe $(deg.)$	2θ Stander $(deg.)$	d Observe (nm)	d Stander (nm)	a Observe (nm)	a Stander (nm)	$h\ k\ l$	I% Observe	I% Stander
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	65.935	0.1416	0.14155	0.4697		3 1 1	23.5	28.5
	69.201	69.271	0.1357	0.13553	0.4702		2 2 2	10.2	12.2
673	33.118	33.019	0.2704	0.27106	0.4683	0.46948	1 1 1	100	100
	38.399	38.321	0.2343	0.23474	0.4686		2 0 0	81.6	84
	55.361	55.299	0.1659	0.16599	0.4691		2 2 0	36.6	45.2
	66.038	65.935	0.1414	0.14155	0.469		3 1 1	20.5	28.5
	69.359	69.271	0.1354	0.13553	0.4691		2 2 2	8.5	12.2

Table (2): Experimental values of XRD parameters of CdO thin film at different .annealing tem prelature

Annealing temperature (K)	2θ 2 Theta $(deg.)$	β FWHM $(rad.)$	TC Texture coefficient	D Grain size (nm)	ε Average strain	δ Dislocation density (nm^{-2})
623	33	0.00853	1.13	16.95	0.00721	0.00348
	38.318	0.00853	1.007	17.2	0.00614	0.00338
	55.282	0.00853	0.987	18.34	0.00408	0.00297
	65.958	0.00853	0.931	19.37	0.00329	0.00267
	69.201	0.00853	0.944	19.75	0.00309	0.00256
673	33.118	0.00793	1.191	18.24	0.00666	0.00301
	38.399	0.00793	1.157	18.52	0.00569	0.00291
	55.361	0.00793	0.965	19.75	0.00378	0.00256
	66.038	0.00793	0.857	20.86	0.00305	0.0023
	69.359	0.00793	0.83	21.26	0.00286	0.00221

4.2 *Effect of Annealing on Optical Properties of CdO Thin Film*

The optical spectra were studied by UV-vis spectrophotometer for CdO thin film as a function of wavelength in the range (300-1100 nm), annealing at different temperature (623, 673 K) and deposition time (10 hrs), Table(3). Fig.(3) reveals that the absorbance (A) spectra of the prepared film increases with increasing of annealing temperature, due to increase in the grain size. Fig.(4) reveals that the transmittance (T) spectra decreases 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 CdO [33,34]. 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 [35]. Fig.(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. Fig.(6) shows extinction coefficient (k) as a function of wavelength (λ), and increases with increasing of annealing temperature. Fig.(7), show typical $(\alpha hv)^2$ plot for direct energy gap at different annealing temperature. The energy gap was found to be (2.95, 2.87 eV), Table(3). It is observed that the band gap energy decreases with increasing of annealing temperature, Fig.(9). This is may be due to the increase in the carrier concentration and also may be due to its quantum confinement effect [31]. Fig.(8), show $(\alpha hv)^{\frac{1}{2}}$ vs. (hv) plot for indirect energy gap at different annealing temperature. The indirect energy gap obtained was (1.945, 1.914 eV), Table(3). 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 [13], Fig.(10) The particle size for indirect energy gap are (9.921-10.194 nm), Table(3).

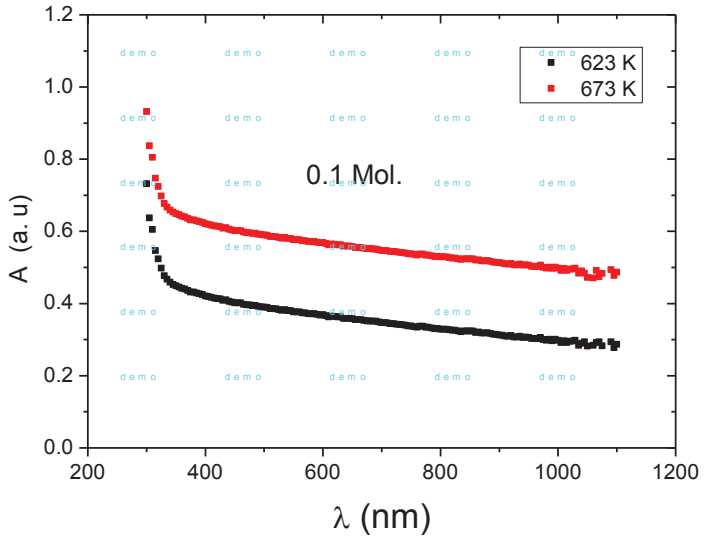


Fig.(3): UV-vis Absorbance (A) spectra of CdO thin film deposited at .different annealing temperature

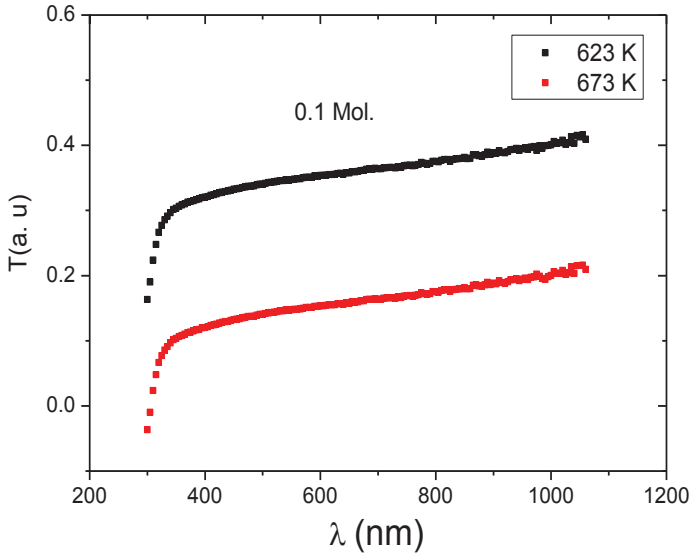


Fig.(4): UV-vis Transmittance (T) spectra of CdO thin film deposited at different .annealing temperature

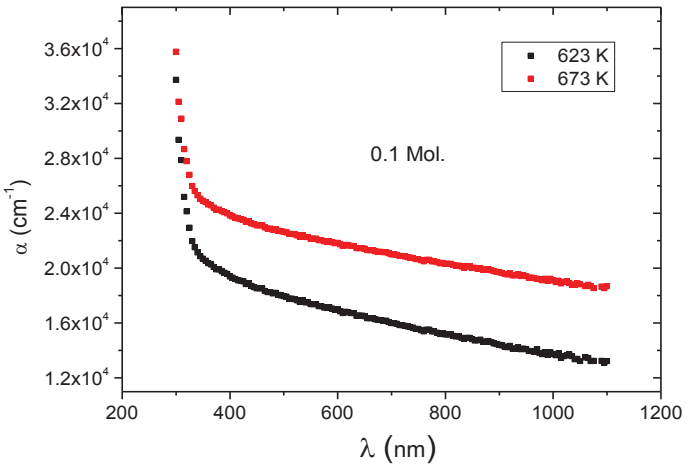


Fig.(5): Absorption coefficient (α) as a function of wavelength (λ) deposited at different .annealing temperature

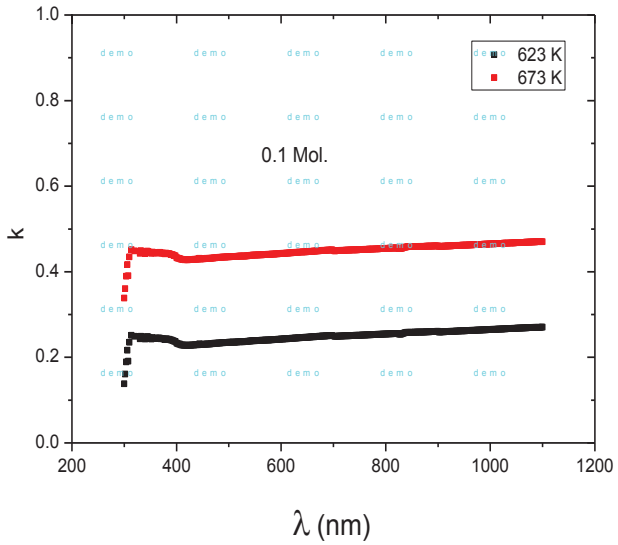
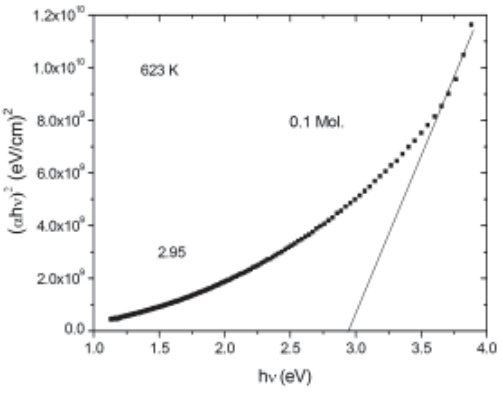


Fig.(6): Extinction coefficient (k) as a function of wavelength (λ) for CdO thin film .deposited at different annealing temperature



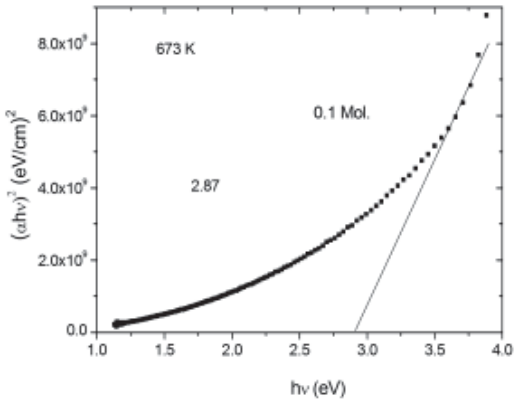


Fig.(7): A direct band gap of CdO thin film deposited at different annealing temperature.

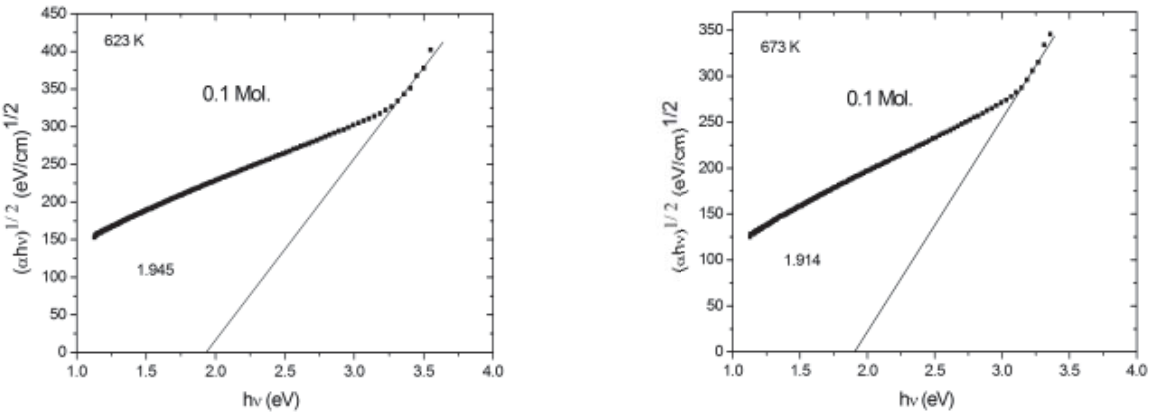


Fig.(8): An indirect band gap of CdO thin film deposited at different annealing temperature.

.temperature

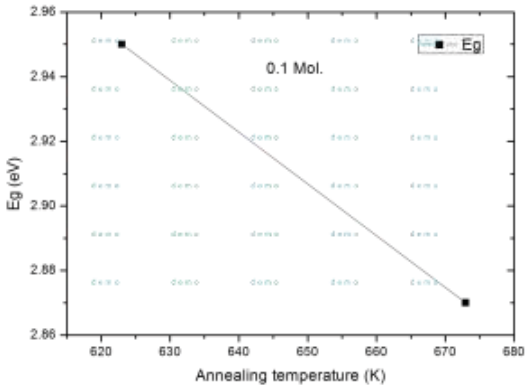


Fig.(9): Variation energy gap with annealing temperature.

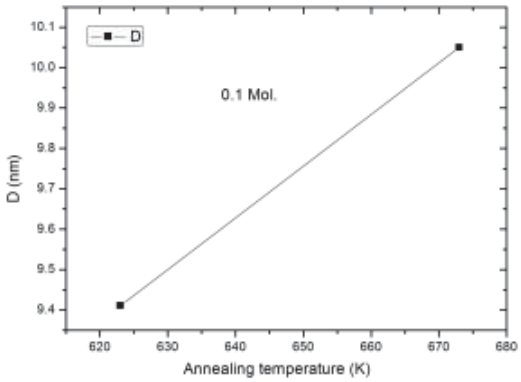


Fig.(10): Variation particle size with annealing temperature.

Table 3: Experimental values of energy gap, blue shift and particles size, for 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

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. XRD patterns confirmed the polycrystalline cubic CdO phase formation and the preferred orientation is (1 1 1) with crystallite sizeis (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.

6- References

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