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Effect of Annealing Temperature on Structural and Optical Properties of CdO Thin Films Prepared by pulsed laser deposited (PLD) technique

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ABSTRACT

In this paper Cadmium oxide (CdO) thin films has attracted interest due to its potential applications in optoelectronics devices. thin films were grown on glass substrates by the pulsed laser deposition (PLD) technique at room temperature and different annealing temperatures Ta=(573,673 and 773) K. The structural studies (XRD) measurements for CdO thin film is polycrystalline with cubic structure and it is evident that the film is highly oriented in the (111) direction. Atomic force microscopy (AFM) was used to examine CdO surfaces, After annealing the films become more homogeneity and notice to increment the root mean square, surface roughness and average grain size. The optical properties of CdO thin films are studied as a function to wavelength in region (375 - 1100) nm. The optical transmittance of the prepared CdO films notice that the transmittance pattern of all deposited thin films increases with increasing the annealing temperature (T_a). The direct energy gap for CdO films was decreases with increasing of annealing temperature for all samples due to the growth of the crystallites .The optical constants such as refractive index, extinction coefficient and dielectric constant were also calculated.

Keywords: Effect of Annealing, Cdo Thin Films, (PLD) technique.

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تاثير درجة حرارة التلدين على الخصائص التركيبية والبصرية لاغشية CdO الرقيقة المحضرة بتقنية الترسيب بالليزر النبضي

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الملخص

في هذا البحث الأغشية الرقيقة من أوكسيد الكادميوم (CdO) جذبت كثير من الاهتمامات نظراً لتطبيقاتها في الأجهزة الإلكترونية والضوئية، وقد رسبت الاغشية الرقيقة على الزجاج بتقنية الليزر النبضي (PLD) في درجة حرارة الغرفة وبدرجات حرارة تلدين مختلفة (573، 673 و773) كلفن. تم دراسة الخواص التركيبية باستخدام حيود الاشعة السينية (XRD) لمسحوق واغشية اوكسيد الكادميوم واظهرت بانها متعددة التبلور بتركيب مكعبي وباتجاهية (111). كذلك تم استخدام مجهر القوة الذرية لفحص التركيب السطحي لأغشية CdO بعد التلدين تبين بانها تصبح اكثر تجانساً ، ولوحظ ايضاً هنالك زيادة في متوسط الجذر التربيعي وخشونة السطح ومعدل الحجم الحبيبي. تم دراسة الخصائص البصرية لأغشية CdO النقية كدالة للطول الموجي الذي يتراوح من (375−1100) نانوميتر، حيث تم ملاحظة النفاذية لاغشية اوكسيد الكادميوم بانها تزداد مع زيادة حرارة التلدين، وتم ملاحظة فجوة الطاقة البصرية المباشرة لجميع العينات المحضرة تقل بزيادة درجة حرارة التلدين ويرجع سبب ذلك الى النمو البلوري، واضافة الى ذلك تم احتساب جميع الثوابت البصرية منها معامل الانكسار ومعامل التوهين وثابت العزل الكهربائي.

الكلمات الدالة: تاثير التلدين ،اغشية CdO الرقيقة ، تقنية الترسيب بالليزر النبضي.

1. Introduction

Transparent conducting oxides (TCOs) are potential candidates for energy efficient devices like solar cells, light emitting diodes, photo detectors because of their high transparency in the visible region and high electrical conductivity at room temperature [1]. A (II-VI) elements in the periodic table. CdO is an n–type semiconductor with band gap of approximately 2.5 eV, CdO is cubic structure with each ion surrounded by six ions of opposite electric charge, octahedrally arranged [2,3] with lattice constant equal to 4.69 A° and a unit cell of face center cubic (FCC) [4]. Cadmium oxide is a reddish powder and its color varies from greenish-yellow brown to nearly black depending on its thermal history and particle size [5]. CdO thin films are prepared by many physical and chemical techniques such as thermal evaporation, sputtering, solution growth and pulsed laser sputtering [6]. CdO films have been successfully used for many applications, including phototransistors [7], gas sensor [8], transparent electrodes, photovoltaic solar cells [9], liquid crystal displays, photodiodes, IR detectors and anti-reflection coatings and optoelectronic devices [1,9]. It is a promising candidate for a transparent conducting oxide material because it has a simple rock-salt crystal structure, high carrier mobility, and high conductivity [10]. Due to its low optical band gap, CdO is not widely used as transparent conducting electrodes, although CdO thin films show low resistivity due to defects of oxygen vacancies and cadmium interstitials [11].

2-Experimental Work

Cdo thin films are prepared by PLD technique. The pulsed laser deposition experiment is carried inside a vacuum chamber at vacuum (10-3 m bar) conditions, Pulsed laser deposition is a thin film growth method that consists of three steps

(1) The interaction of the pulsed laser beam with a ceramic target usually in the form of a sintered pellet (ablation).
(2) The formation and expansion of the laser-induced plasma.
(3) The deposition of the film on a substrate.

maintained at a certain temperature (growth temperature) at low pressure. The set-up of laser deposition chamber Photograph is given in Fig. (1) which shows the arrangement of the substrate holders and target inside the chamber with respect to the laser beam. The focused

Nd, YAG SHG Q-switching laser incident beam coming through a window is making an angle of 45° with the target surface. The substrate is placed parallel in front of with the surface of the target. Sufficient gap is kept between the target and the substrate and the substrate holder does not obstruct the incident laser beam. Many investigators were done this modification of the deposition technique from time to time with the aim of obtaining better quality films by this process. These include rotation of the target, a positioning of the substrate with respect to the target. In addition, PLD is a versatile epitaxial route that allows the rapid growth of almost any kind of material over a wide range of gas ambient, substrate temperatures, pressures, laser power densities (fluencies) and repetition rates.

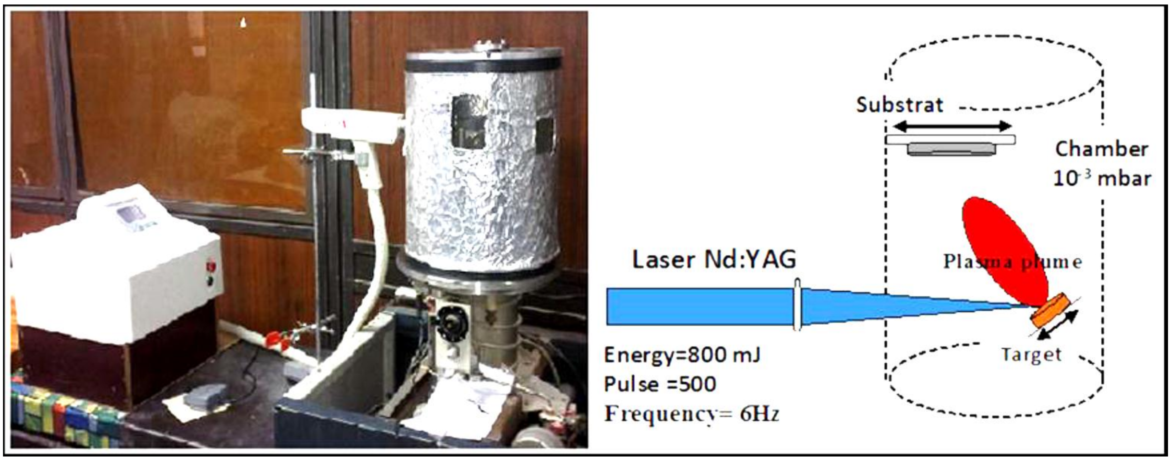

Fig. (1): Pulse laser deposition experimental set up

The specifications of used laser are:

1- Nd: YAG laser (Huafei Tongda Technology- DIAMOND-288 pattern EPLS).
2- Power= 500 m J, 3- F = 6 HZ 4- λ = 1064 nm
3- Number of shots = (100, 200, 300, 400 and 500) pulse.
4- Cooling method: inner circulation water cooling.

2.1 Target preparation

Cadmium oxide (CdO)High purity (99.999) powder by Redel-De Haen, German Company of these materials were mixed in gate mortar was used to form the target as a disk of 1.5cm diameter and 0.3cm thickness using hydraulic piston type (SPECAC), under pressure of 6 tons for 10 minute, as shown in Fig. (2). It should be as dense and homogenous as possible to ensure a good quality of the deposit.

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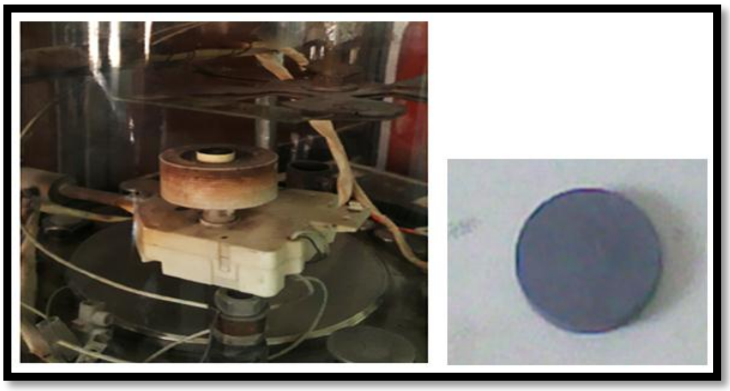

Fig. (2): Target before ablation.

To study the structural and optical films properties, CdO films are deposited on glass substrates with (2.5x 2.5) cm^2 dimensions to obtain the most durable and adherent coating substrate, the support surface must be free from contamination films such as grease, absorber water etc…., the cleaning of the substrates was very necessary. The Film deposition on the Substrat. Thin film was achieved immediately after the laser beam hits the target resulting in the evaporation of the target material, which itself mounted on rotating holder with 45° orientation from the substrate in order to ensure that the plasma plume is right-angled with respect to the substrate. The following Fig. (3) show the target after ablation Process. The rotation of the target was employed to avoid the ablation of the same spot on the target. The distance between the target and the substrate is about 1.5 cm and it was found to be the optimum distance.

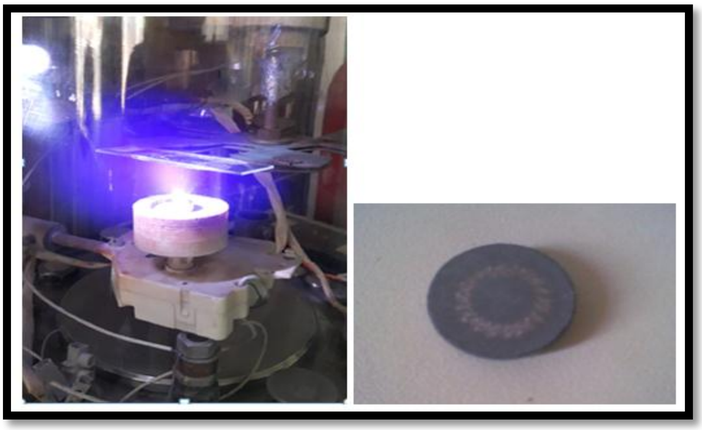

Fig. (3): Target after ablation

All the samples were annealed in a furnace type England, S30 2AU at temperature (573,673 and 773) K for 2 hours were studied by X-ray diffraction (XRD) techniques using a(Philips PW) X-ray diffractometer system. This system recorded the intensity as a function of Bragg's angle. The film surface topography(AFM) of the samples were studied by taking image for the films surface with tapping mode using atomic force microscope (AA3000 Scanning Probe Microscope SPM. Angstrom Ad-Vance Inc, tip NSC35/AIBS). the optical measurements of thin films depend on, thickness, homogeneity, structure, materials used and the preparation conditions. were measured using UV/ Visible SP-8001 spectrophotometerover the range (375-1100) nm.

3. Results and discussion

3.1 Structural properties

The X-ray diffraction patterns of CdO thin films grown on glass substrate were prepared by pulsed laser deposition (PLD) at different annealing temperatures Ta=(573, 673 and 773)K for 2 hour. All the films show polycrystalline nature containing cubic structure of pure CdO phase are shown in Fig. (4). The planes (111), (200), (202) and (311) indicate the CdO Cubicstructure phase with strongly preferred orientation at ($2\theta = 33.01^o$) along the (111) planes from the figure. It can be clearly seen that the preferential orientation peak with increase the annealing temperature became sharper and more intense. While the film grown at (773)K shows (111) and (200) orientation. But the films grown at higher temperature especially for (111) planes. This may be attributed to the crystallinity of the CdO films being improved with increasing the annealing temperatures. The observed diffraction patterns are in good agreement with the standard crystallographic data for the CdO metals. In addition, from the XRD peak width is found that the peak width decreases as the annealing temperature increases. The average size of the grains has been calculated from the XRD pattern using Debye-Scherer's formula.

$$G.S = \frac{0.94\,\lambda}{\beta \cos\theta}$$

where $G.S$ is the grain size, β is the full width at half maximum (FWHM) of the preferential orientation diffraction peak and¸ λ is the wavelength of the X-rays. When annealing increases from (573,673and 773) K, the crystallite size also increases. The intensity

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of the diffraction peak was also found to increase with increasing annealing temperature and get sharper with decreasing full width half maximum (FWHM). This can be attributed to the improvement in crystallinity of CdO thin films. The structural parameters thin films are given in Table (1). These results was found in a good agreement with the [12, 13, 14].

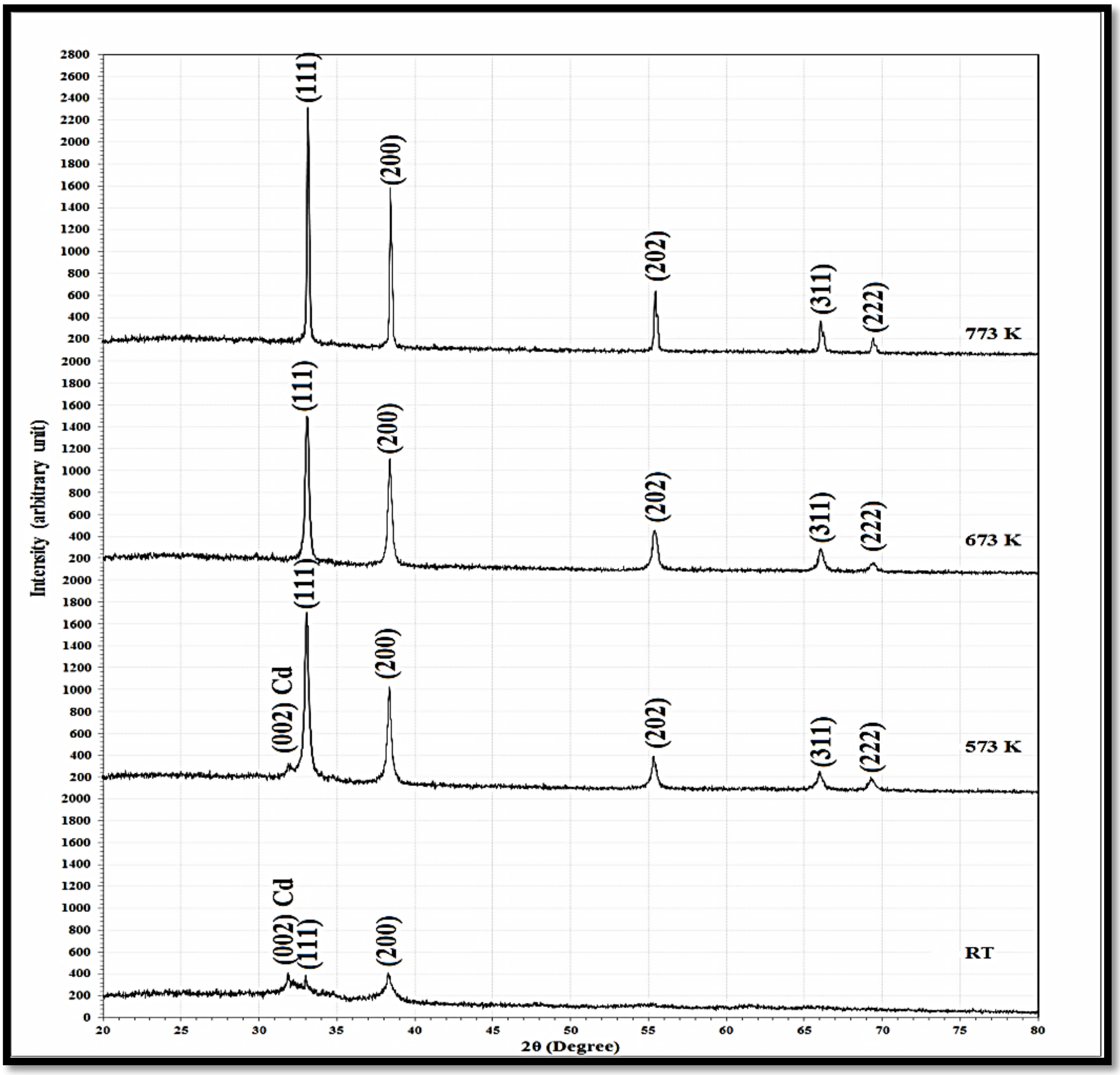

Fig. (1): The XRD pattern at different annealing temperatures (RT, 573, 673 and 773) K.

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Table (1): Effect of annealing temperature on the structural properties of CdO thin films.

Ta (K)	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	d_{hkl} Std.(Å)	hkl	Phase	card No.
RT	31.8351	0.5692	2.8087	14.5	2.8035	(002)	Cd	96-901-2437
	33.0128	0.3141	2.7112	26.4	2.7108	(111)	CdO	96-900-8610
	38.2728	0.5299	2.3498	15.9	2.3477	(200)	CdO	96-900-8610
573	31.8548	0.3925	2.8070	21.1	2.8035	(002)	Cd	96-901-2437
	33.0520	0.3337	2.7080	24.8	2.7108	(111)	CdO	96-900-8610
	38.3710	0.3141	2.3440	26.8	2.3477	(200)	CdO	96-900-8610
	55.3288	0.3337	1.6591	26.9	1.6600	(202)	CdO	96-900-8610
	66.0059	0.3533	1.4142	26.8	1.4157	(311)	CdO	96-900-8610
	69.2640	0.3533	1.3554	27.3	1.3554	(222)	CdO	96-900-8610
673	33.0913	0.2944	2.7049	28.2	2.7108	(111)	CdO	96-900-8610
	38.3906	0.3140	2.3428	26.8	2.3477	(200)	CdO	96-900-8610
	55.4073	0.3336	1.6569	26.9	1.6600	(202)	CdO	96-900-8610
	66.0648	0.3729	1.4131	25.4	1.4157	(311)	CdO	96-900-8610
	69.4210	0.4121	1.3528	23.5	1.3554	(222)	CdO	96-900-8610
773	33.1501	0.1767	2.7002	46.9	2.7108	(111)	CdO	96-900-8610
	38.4298	0.1766	2.3405	47.7	2.3477	(200)	CdO	96-900-8610
	55.4465	0.2355	1.6558	38.1	1.6600	(202)	CdO	96-900-8610
	66.0451	0.2356	1.4135	40.2	1.4157	(311)	CdO	96-900-8610
	69.4210	0.2552	1.3528	37.9	1.3554	(222)	CdO	96-900-8610

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3.2 Atomic Force Microscopy

Atomic force microscopy (AFM) was used to examine CdOfilms surfaces, average pore diameter, depth and roughness surface for each sample. The increase in surface roughness of the films leads to an increase in the efficiency for sensing properties, therefore, it is very important to investigate the surface morphology of the films. Fig . (5) show two-dimensional and three-dimensional of (AFM) images of pure CdOthin films deposited on glass substrateby pulsed laser deposition (PLD) prepared for room temperature and with different annealing temperatures (573,673 and 773) K. AFM observations, as evidenced by the scanning process for an area with films prepared at room temperature and then annealed clearly show that there is a remarkable change in surface morphology and roughness of the CdO films depending on the annealing temperatures. The most likely reason for the dramatic topographic change is the recrystallization in the films due to thermal annealing which is in agreement with the previous results[15,16]. The average diameter, average roughness, root mean square roughness (r.m.s) and peak –peak are deduced from AFM images of these samples were listed in Table (2). After annealing the film becomes more homogeneity and notice to increase the root mean square of surface roughness and average grain size. This result may be attributed to higher atom mobility with the increase in temperature which causes more effective recrystallization and grain growth of the films that result in larger grains. On the other hand, the larger grains or hillocks on the surfaces of the films reveal an improvement in the crystalline quality of the CdO films, which is in agreement with XRD results.

Table (2): Morphology Parameters for CdO thin films.

Sample	Ta (K)	Average diameter (nm)	Average roughness (nm)	Peak –Peak (nm)	r.m.s (nm)
CdO	**RT**	77.58	3.5	13.4	4.03
	573	78.45	3.39	16.4	4.05
	673	83.5	4.65	32.7	5.46
	773	121.09	9.16	37.5	10.5

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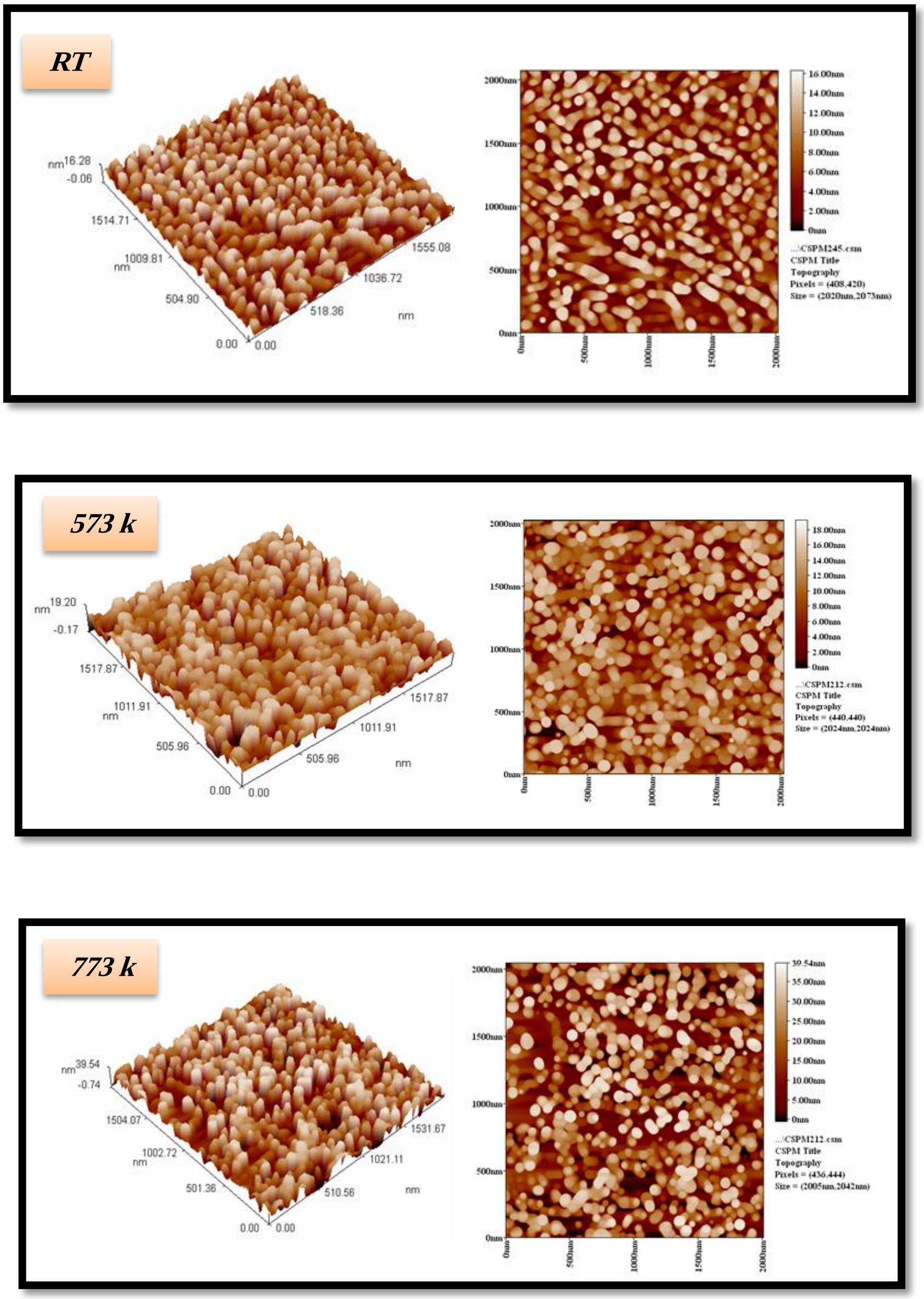

Fig. (5): 2D and 3D AFM images pure CdO thin films.

4. Optical Properties

The Optical properties of CdO thin films prepared, by pulse laser deposition (PLD) technique, on glass substrate at room temperature and different annealing temperatures (573,673 and 773) K for 2 hour have been investigatedusing UV-Visible transmission and

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absorbance spectrum in the wavelength range (375-1000) nm. The optical energy gap E_g and optical constant which includes the extinction coefficient k ,which involves refractive index (n)such as real dielectric constant (ε_r) and imaginary dielectric constant (ε_i) also were calculated.

4.1 The Transmission Spectrum (T)

CdOfilm deposited on glass substrate. The variation of optical transmittance of the film was studied in the wavelength range (375–1100) nm. Fig (6) shows plots of transmittance spectra versus wavelength for the CdOthin films annealed at different temperatures. It is observed that all films showed the light transmittance is low in UV region since thin film absorbs less energy at high wave length, while light transmittance is high in VIS region since thin film absorbs more energy at low wavelength (below 375nm) transparency depends on the growth temperature. We notice also that the transmittancepattern of all deposited thin films increases with increasing the annealing temperatures T_a=(RT,573,673 and 773) K and shifts to longer wavelengths, which means a decrease in absorption that occur due to the increase of annealing temperature. It is observed that the film which has higher temperature at (773 K) displayed lower transmittance because of the improved structure of the film and large grain size which both cause increase of absorption. This increase and decrease in transparency is related to the structural properties of the film characteristics.

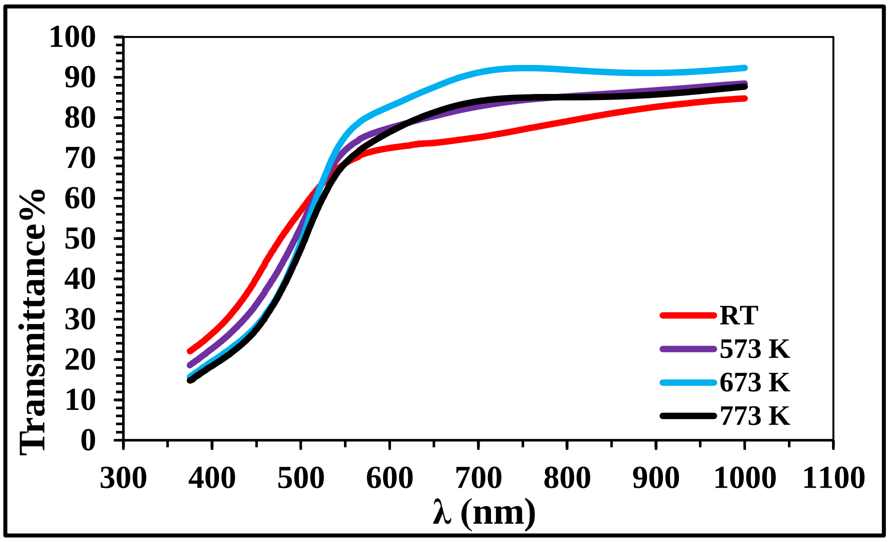

Fig. (6): The Variation of the Transmittance with Wavelength of the CdO Films with Different Annealing Temperatures.

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The optical transmittance of the prepared films was typically higher than 80% at wavelengths beyond the absorption edge. Clear that the decreases or the shift towards longer wavelengths is not sharp. The parallel transmission shift however indicates that it is related to changes in film structure.

It can be seen also that the increase in transmittance in UV region is not sharp. This indicates that the absorption band gap transitions in the studied films are due to direct and indirect transitions. The absorption is attributed to the fundamental band gap. These results are consistent with other published results such as [17, 18, 15]. Transmittance of all films was calculated high transparency in the visible region. This is due to the lower film absorption [12,14] which means that films suitable as a window gap for solar cells because the effective spectral region in solar cells are located in the visible region of the spectrum.

4.3 The Optical Energy Gap

The optical energy gap in general depends on the arrangement and distribution of atoms in the crystal lattice and the films crystal structure. In order to determine the optical energy gap values (E_g) for CdO thin films have been determined by using Tauc relation equation which is used to find the type of the optical transition, graph is plotted with $(\alpha h\nu)^2$ against hν (eV) as shown in Fig. (7) with different annealing temperatures is shown in the plot is linear indicating the direct band gap of the films. Extrapolation of the linear of the line to the hυ axis gives the band gap. The value of the optical energy gap at room temperature and different annealing temperatures at T_a =(573,673,and 773) K decreases from (2.65 to 2.500) eV sequentially with increasing of annealing temperature for all sample due to the growth of the crystallites shown in the Table (3) a good agreement with the literature references [15,19].

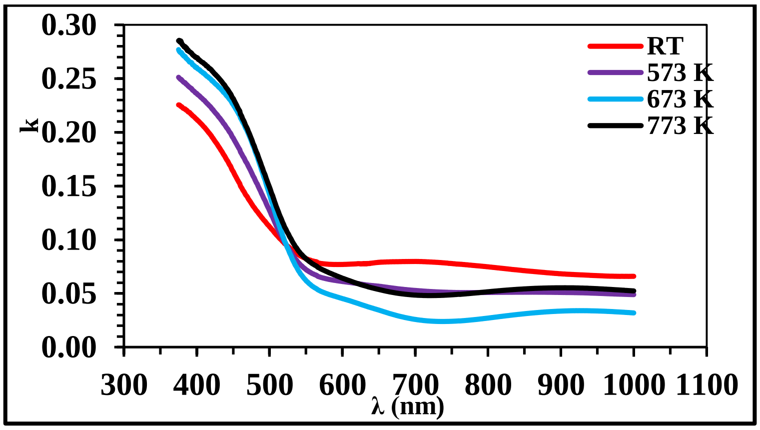

Fig. (7): The Variation of the Extinction Coefficient (K) With Wavelength of CdO Films with Different Annealing Temperatures

4.6 The Dielectric Constants for CdOfilms

The variation of the real (ε_r) and imaginary (ε_i) parts of the dielectric constant values respectively. The plots of real and imaginary (ε_r and ε_i) parts of the dielectric constant with wavelength for CdO deposited at different annealing temperature. Also Fig. (11 a and b) show the variation of real and imaginary parts of dielectric constants for CdO at different annealing temperatures (573,673 and 773) K. It is observed that real part and imaginary part of dielectric constants decrease with the increase of annealing temperature and this is due to the decrease of the refractive index and the extinction coefficient with wavelength. This behavior is in agreement with the results Minden [23, 24]. The variation in the optical constants is summarized in Table (4).

Table (4): illustrate values of optical constants at λ=500 nm for CdOfilms with different annealing temperatures .

Ta (K)	T%	α (cm^{-1})	K	n	ε_r	ε_i	E_g (eV)
RT	56.96	28137	0.112	2.517	6.321	0.564	2.650
563	52.82	31912	0.127	2.579	6.635	0.655	2.600
673	48.98	35686	0.142	2.620	6.844	0.744	2.520
773	47.39	37339	0.149	2.632	6.903	0.782	2.500

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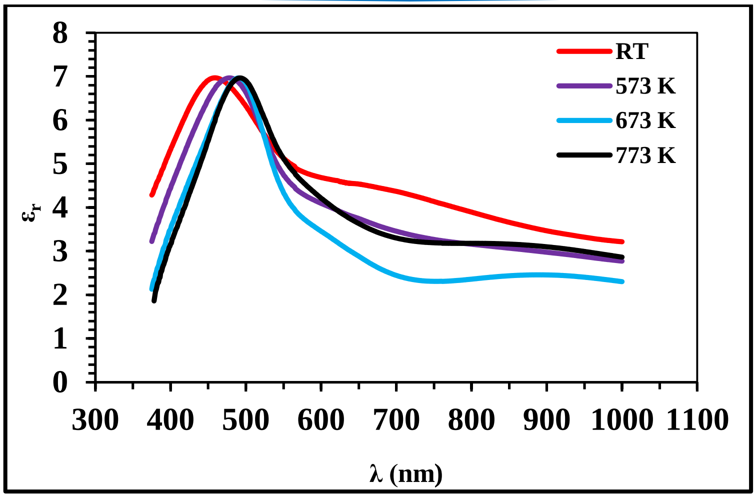
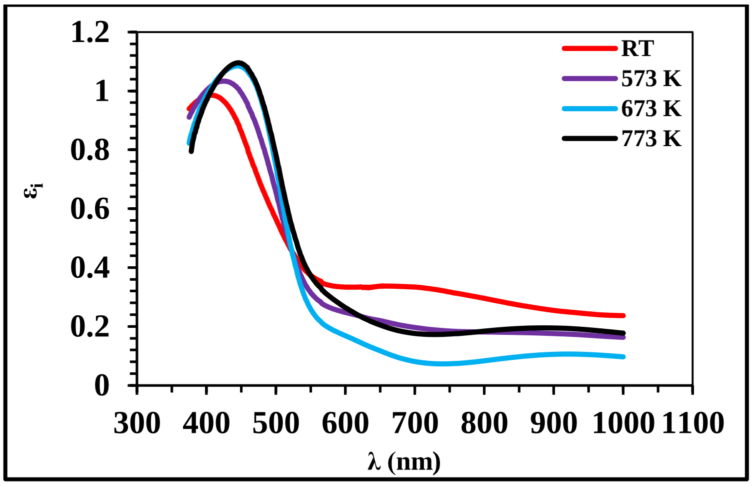


a



b

Fig. (11): The Variation of The Dielectric Constants a- real part (ε_r) and b- imaginary (ε_i) parts with Wavelength of CdO Films with Different Annealing Temperatures.

5. Conclusions

Thin films of CdO were deposited on glass substrates are prepared by means of simple and low cost by pulsed laser deposition (PLD) technique. Post-annealing process helps to improve the crystalline quality thin films. The effect of growth temperature on structural and optical properties was studied. All the films show polycrystalline nature containing cubic structure of pure CdO phase. The optical transmittance spectra show the NIR transmittance of 80% and direct band gap values decreases with increasing annealing temperatures about 2.5 eV for the film annealed at 773 K for 2 hour.

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