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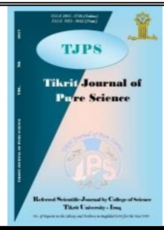

Synthesis and Characterization of CdO Nanoparticles Prepared by Pulse Laser Deposition

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ABSTRACT

Thin films of cadmium oxide CdO nanoparticles were prepared on glass substrates using pulse laser deposition technique (PLD). N:YAG laser is used with different laser energies (400,500,600,700)mJ, number of shots=100, when preparing all the thin films. The structure and optical characteristics were studied for these films. X-ray diffraction (XRD) results showed that all the prepared thin films were of a polycrystalline type and that the high and dominant crystalline levels were (111) at the angle (2θ =32.986). AFM results demonstrated a grain size of deposited films increase from (50.63-85)nm with laser energy from 400 to 700mJ. Also the absorbance spectrum increases from (0.53-1.45) with increasing laser energy, while the transmittance spectrum decreases with increasing laser energy of all prepared models so that transmittance is (26.53%) at the laser deposit energy (400mJ) until it becomes (1.70%) at the laser deposit energy (700mJ) and at the wavelength (550nm), as for the energy gap it decreases with increasing laser deposit energy and ranges from (2.3-2.5)eV.

1. Introduction

Cadmium oxide is transparent conducting oxides (TCO) materials that hold both high electrical conductivity and high optical transparency in the visible light region of the electromagnetic spectrum [1]. Cadmium oxide is one of the inorganic chemical compounds that can be obtained industrially from the intense heating of pure cadmium elements in the air without its melting point [2], it is also prepared in laboratory through the oxidation of cadmium vapor deposited in the presence of air and in special ovens [3,4].It has ability to dissolve in the salts of ammonia and acids , which its solubility is lacking in water and bases .cadmium oxide is present in nature in two structures forms, one is crystalline and the other is random. The crystalline structures formula is brown or red color, while its random structures formula is colorless [5]. The nature of the crystalline composition has the advantage face center cubic (FCC) similar to composition of NaCl crystal [6]. The classification compound in terms of its electrical conductivity is of the negative type (N), because of the presence of oxygen vacancies caused by molecular mismatch between non- stoichiometric compound. cadmium oxide to the (II-VI) group compounds of the periodic table. Cadmium oxide has distinctive properties that enable it to be used in many different scientific applications and technological industries, among these characteristics is that it possesses a with a direct band gap of (2.5)eV and indirect band gap of (1.98)eV. [7]. Therefore, it is a transparent conductive material and a thin films in the manufacture of electro- optical devices such as solar cells, optical transistors, liquid crystal display, light emitting diodes, anti reflecting coating, reflecting coating, cadmium oxide is also used in the manufacture infrared radiation detectors [8]. Pulse laser deposition is widely used technique to synthesize crystalline nanoparticle thin films of different materials of high quality in one step, with lower cost that any other technologies.[9]. In this technique, the largest number of films can be prepared in record time, by controlling the laser energy, the number of pulses, distance between the target material and the glass substrates, and its effect on the properties of thin films.

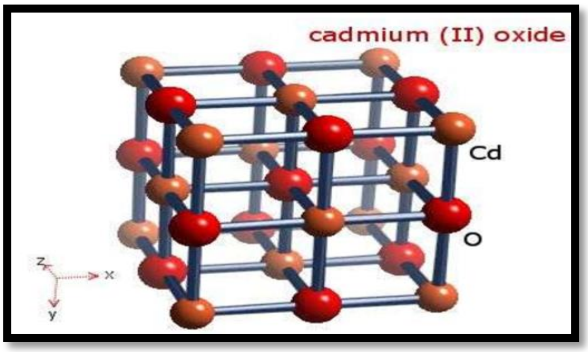

Fig. 1: lattice structure of the CdO[10].

2. Experimental part

2.1 Sample preparation

Thin films from nanoparticle powders were deposited of high purity (99.96) of CdO, It is manufactured by the American company (Sky spring nanomaterial's) to study its structural and optical properties by pulse laser deposition (PLD), on glass substrates of engineering dimensions (75×25×1.2) mm after being configured by cleaning it with distilled water and ethanol- pure alcohol (99.9) using an ultrasonic bath , we took the weight of 3gm of the material using a sensitive scale (metller.A.K-160) with a sensitive of (10^{-4}) gm, the pressing was done using a hydraulic press with a pressure force (10^{7}) N/m^2 for a period of 20 minutes. The model was prepared in the form of circular discs with a diameter (1.2) cm and a thickness (0.3 cm).

2.2.Thin film Deposition System

The Nd:YAY laser was used in this research conducted by (DIMON-288 Pattern Epls System) in this process of thin films deposition at energies (400,500,600,700) mJ, wavelength (λ=1064 nm), frequency (f=6Hz), number of pulses is constant (100) pulses, the best angle of incidence laser pulses on the target material surface (45^0) to form the largest possible deposition area on the glass base. The chamber used in the deposition process is a cylindrical chamber with a diameter (30cm) and a height (40cm) made of glass, and it was the process of depositing thin films at any height discharge under pressure (10^{-2}) Torr. The optical interferometer method was used, for measure the thickness of the films, at each deposition energy.

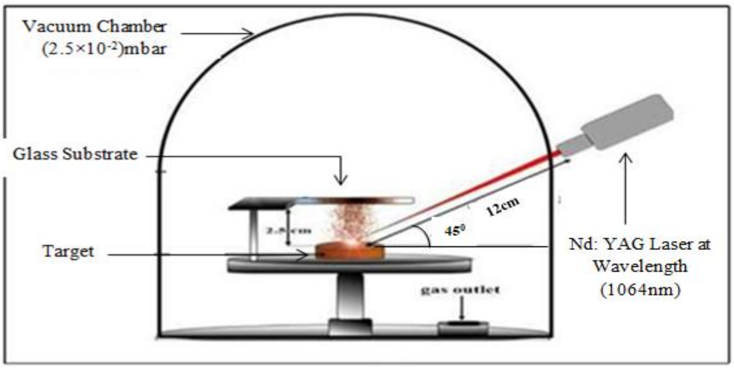

Fig. 2: pulse laser deposition process diagram .

3. Results and Discussion

3.1. Structural Characteristics

The main and important purpose of this part is to know the type of crystal structure of the semiconductor materials used in this research. Figures (3) and (4) show the results of X-ray diffraction (XRD) for CdO as a powder and for films grown on glass substrates by pulse laser deposition technique at laser energies (400,500,600,700) mJ . It shows a polycrystalline structure for all films, where five peaks located in CdO model at (32.98^0), (38.28^0), (55.24^0), (65.88^0), (69.25^0) corresponds to (111), (200), (220), (311), (222) respectively as a result of the Bragg reflection at the parallel crystal surfaces are due to CdO substance with a face center cubic crystal structure (FCC), which perfectly matches with the CdO reference of according to (card No 96-900-8610). These results are the same with the other researches[11,12,13]. The preferred orientation was along (111) for CdO structure. From fig (4) we notice the increase in the intensity of peaks with increased deposition energy. The (FWHM) (β) method that is often calculated by Debye – Scherrer relation[14].

$$D_{hkl} = \frac{K\lambda}{\beta\cos\theta} \quad \ldots (1)$$

Where is the D_{hkl} average grain size, λ is the X-ray wavelength (nm), β is the full width at half maximum , K is the constant (0.94) and θ is the Bragg angle. Table.1 shows the diffraction angles, full width at half maximum, as well as the global cards for each peak, grain size(D_{hkl}) and inter planer distance (d_{hkl}) for films at each deposition energy. The average grain size evaluated from (111) peaks increased from 32.4 to 38.8 nm with the increase of laser deposition energy from 400 to 700 mJ .

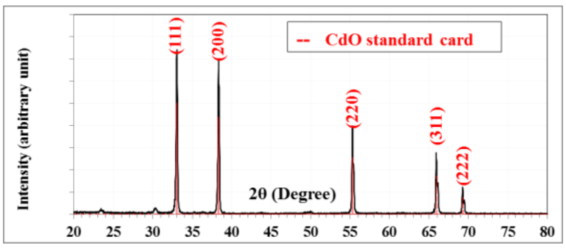

Fig.: 3 X-ray diffraction results for powder (CdO).

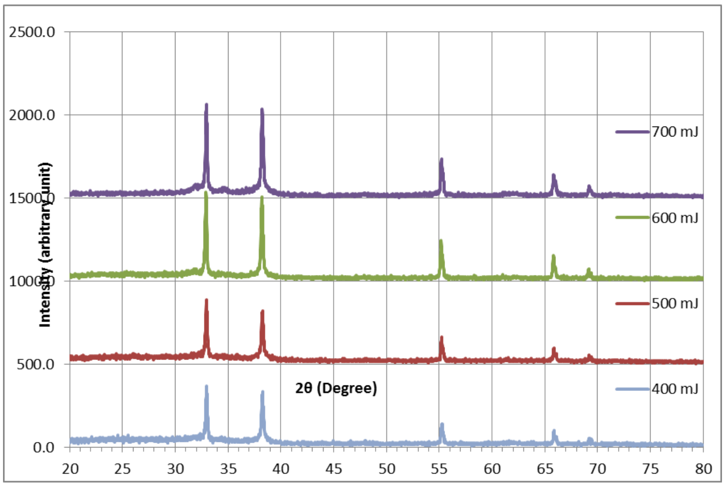

Fig. 4: X-ray diffraction results for CdO films at laser energies (400,500,600,700)mJ.

Table 1: parameters of structural characteristics of prepared CdO films at laser energies (400,500,600,700) mJ.

Energy (mJ)	2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	d_{hkl} Std.(Å)	Phase	hkl	card No.
400	32.9638	0.2560	2.7151	32.4	2.7108	Cub. CdO	(111)	96-900-8610
	38.2353	0.2450	2.3520	34.3	2.3477	Cub. CdO	(200)	96-900-8610
	55.2262	0.2942	1.6619	30.5	1.6600	Cub. CdO	(202)	96-900-8610
	65.8597	0.2714	1.4170	34.9	1.4157	Cub. CdO	(311)	96-900-8610
	69.2308	0.3391	1.3560	28.5	1.3554	Cub. CdO	(222)	96-900-8610
500	32.9186	0.2334	2.7187	35.5	2.7108	Cub. CdO	(111)	96-900-8610
	38.1900	0.2262	2.3547	37.2	2.3477	Cub. CdO	(200)	96-900-8610
	55.1810	0.2485	1.6632	36.1	1.6600	Cub. CdO	(202)	96-900-8610
	65.8371	0.2714	1.4174	34.9	1.4157	Cub. CdO	(311)	96-900-8610
	69.1629	0.2715	1.3572	35.5	1.3554	Cub. CdO	(222)	96-900-8610
600	32.9864	0.2260	2.7133	36.7	2.7108	Cub. CdO	(111)	96-900-8610
	38.2805	0.2487	2.3493	33.8	2.3477	Cub. CdO	(200)	96-900-8610
	55.2489	0.2715	1.6613	33.0	1.6600	Cub. CdO	(202)	96-900-8610
	65.8824	0.3167	1.4166	29.9	1.4157	Cub. CdO	(311)	96-900-8610
	69.2534	0.3162	1.3556	30.5	1.3554	Cub. CdO	(222)	96-900-8610
700	32.9864	0.2136	2.7133	38.8	2.7108	Cub. CdO	(111)	96-900-8610
	38.2805	0.2261	2.3493	37.2	2.3477	Cub. CdO	(200)	96-900-8610
	55.2262	0.2700	1.6619	33.2	1.6600	Cub. CdO	(202)	96-900-8610
	65.8597	0.2485	1.4170	38.1	1.4157	Cub. CdO	(311)	96-900-8610
	69.1629	0.2710	1.3572	35.6	1.3554	Cub. CdO	(222)	96-900-8610

Atomic force microscope (AFM) as one of the important and preferred methods of analyzing the layers of outer surface of prepared films with the technology of pulse laser deposition. Figure (5) show analytical (AFM) images in two (2D) and three (3D) dimensions of pure cadmium oxide films prepared at laser energies (400,500,600,700)mJ. It is seems to be that the external appearance of these prepared models have large number of size of grain and they are spread homogenously and that mean the crystallization nature of the model. The surface roughness (S_a) and root mean square (S_p) values were increased with the increasing of laser deposition energy as shown in table.2. The average grain size evaluated increased from 50.63 to 85 nm with the increase of laser deposition energy from 400 to 700 mJ. As increasing surface roughness has an important benefit in solar cells by increasing the angles of radiation fall on the surface of the membrane and decreasing the reflectivity values and thus the absorption of electromagnetic spectrum will increased efficiency of the solar cell [15]. These results are the same with the other researches [16].

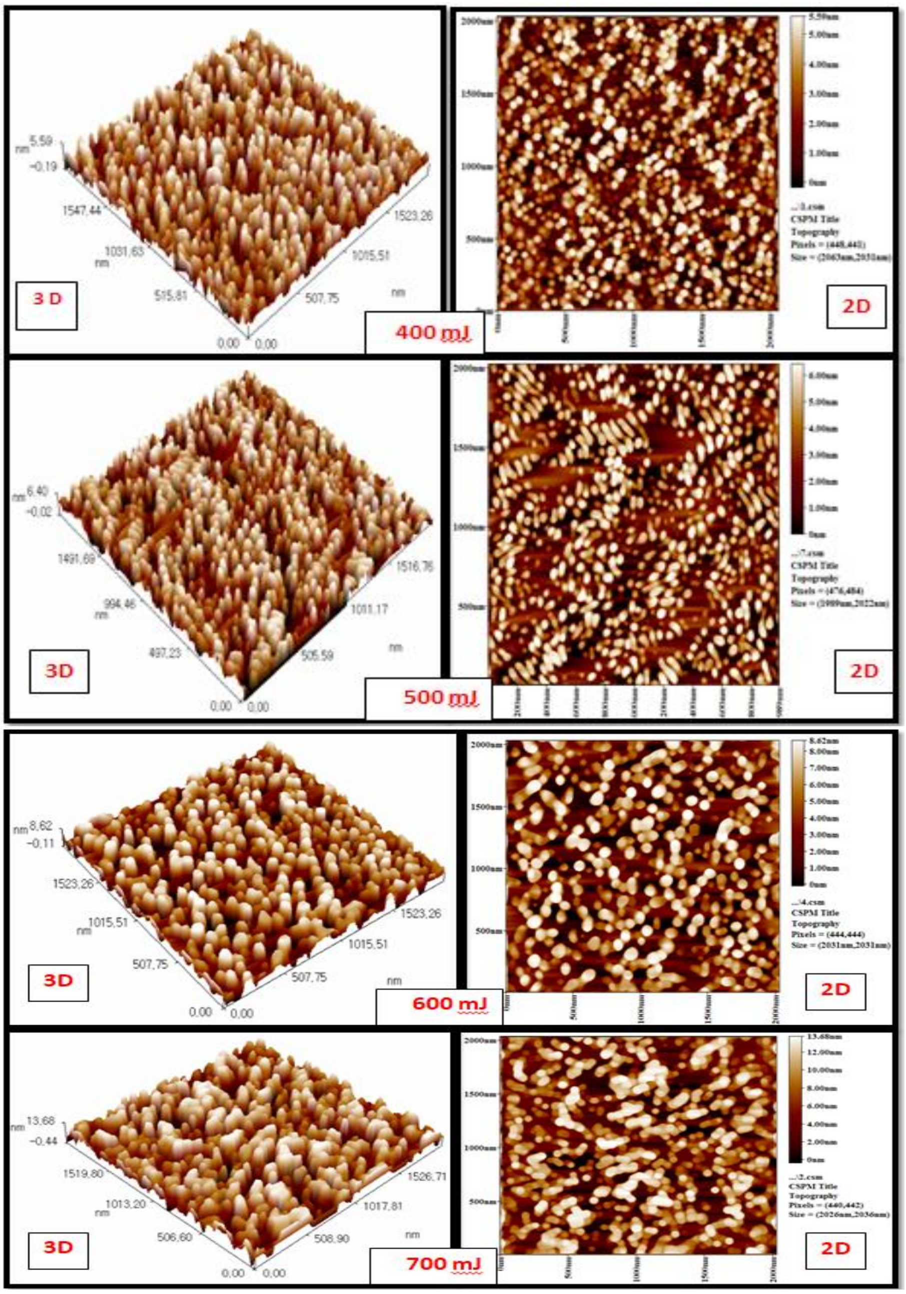

Fig. 5: (AFM) Images in two (2D) and three (3D) dimensions of pure cadmium oxide films prepared at laser energies (400,500,600,700).

Table 2: shows the grain size , S_a ,S_p ,from (AFM) for CdO thin films at laser energies (400,500,600,700) mJ.

Energy (mJ)	Average diameter (nm)	Average roughness (nm)	r.m.s (nm)	Peak-Peak (nm)
400	50.63	1.45	1.67	5.78
500	54.52	1.62	1.87	6.42
600	70.89	2.18	2.52	8.73
700	85	3.42	3.95	14.1

3.2 Optical Properties

Optical properties of CdO films deposited on glass substrate were done in the wavelength rang (300-1100)nm. Figure .6 shows the absorption spectrum samples at different laser energy , absorbance values are the greatest possible at short wavelengths (visible region) and then begins to decrease exponentially with increasing wavelength for all the prepared films. This can be attributed to the fact that the photon does not have enough energy to transfer the electron from the valance band to the conduction band because the energy of the falling photon is less than the value of energy gap of the semiconductor. On the other hand , the absorbance increases with increasing laser deposition energy from 400 to 700mJ .Because of the increasing number of collisions of photons with electrons of matter, which leads to an increase in electronic transfers between the valance and conduction bands, therefore, absorbance will increase with increasing laser deposition energy.

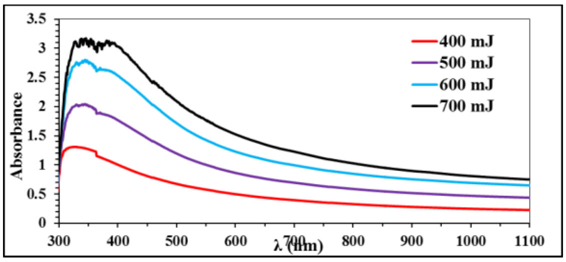

Fig. 6: Absorbance Spectrum of CdO films prepared at laser energies (400,500,600,700) mJ.

The transmittance of the CdO thin films deposited with different of laser energy (400,500,600,700) mJ are shown in fig .7. As the transmittance is as low as possible at short wavelengths and maximum as possible at large wavelengths for all prepared films. It is obvious that the transmission decreases from 26.53 to 1.70% with increase in laser energy from 400 to 700mJ at the wavelength 550nm due to increasing of thickness from (200-250)nm as shown in Table .3. This is agreement with result [16].

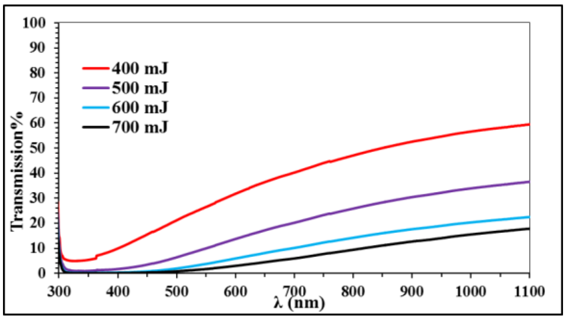

Fig. 7: The transmittance as a function of wavelength for CdO thin films with different laser deposition energies (400,500,600,700) mJ.

To calculated optical energy gap values (E_g^{op}) for CdO thin films were determined using Tauc equation[16]:

$$\alpha h\nu = B(h\nu - E_g)^{1/2} \quad ...(2)$$

where B is a constant, hν the incident photon energy, α the absorption coefficient, E_g the optical energy gap. By plotting the relation between $(\alpha h\upsilon)^2$ versus photon energy (hυ) , as shows in figure .8 the optical energy gap (E_g^{op}) values for CdO films at different laser deposition energy (400,500,600,700)mJ and deposited on glass substrates .In general, the values of direct optical energy gap decrease with increasing laser energy for all samples . The direct (E_g^{op}) decrease from (2.5-2.3) eV. This decrease in the values of optical energy gap may be due to the increased density of state that lead to the formation of secondary levels within the energy gap.This decrease may be due to the expansion of lattice , which causes shift at the edge position of the VB and CB bands through the growth of grain size, as less causes of defects near the bands [17] .

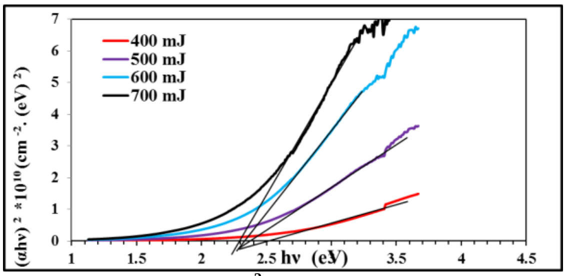

Fig. 8: Variation of $(\alpha h\upsilon)^2$ versus photon energy (hυ) for CdO films deposited at different laser energies (400,500,600,700) mJ.

Figure .9 Shows the absorption coefficient of all CdO membranes prepared at laser energies (400,500,600,700) mJ . The result showed that the absorption coefficient values are greater than 10^4 cm^{-1}, and this means that the transition has occurred. The result are in close agreement with [18]. The absorption coefficient (α) is calculated from the flowing relationship [19]:

$$\alpha = 2.303\frac{A}{t} \quad ....(3)$$

where A is absorbance, t is the thickness of film.

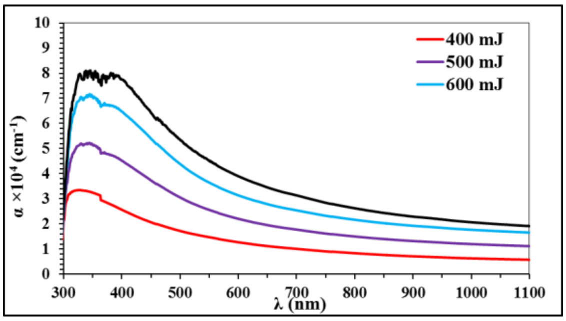

Fig. 9: The absorption coefficient as function wavelength for CdO films with different laser energies (400,500,600,700) mJ.

The relation between the extinction coefficient (k) and wavelength for CdO films at different laser energies (400,500,600,700) mJ is shown in fig.10 . From through this figure, we notice that the extinction coefficient has similar behavior to the absorption coefficient, because they are related to the following equation [20]:

$$k = \frac{\alpha\lambda}{4\pi} \quad ...(4)$$

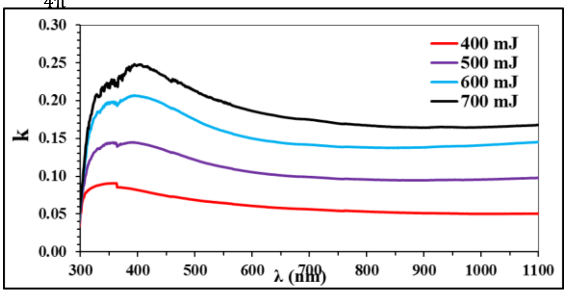

Fig. 10: Extinction coefficient as function wavelength for CdO films with different laser energies (400,500,600,700) mJ.

Table 3: parameters of optical properties for CdO films at laser energies (400,500,600,700) mJ.

	Laser Energy(mJ)	T%	A	α(cm-1)	k	Eg (eV)	Thickness (nm)
CdO	400	26.53	0.53	14744	0.065	2.5	200
	500	9.97	0.99	25616	0.112	2.4	215
	600	3.35	1.42	36487	0.162	2.35	235
	700	1.70	1.75	45282	0.198	2.3	250

Conclusion

In this paper the CdO films that deposited on glass substrates is prepared by pulse laser deposition. The X-ray diffraction demonstrate the film was polycrystalline structure . The architecture of (AFM) samples have large unit of grain size and homogeneous distributing and unified . A grain size of deposited films increase with increasing laser energy. The transmission, direct energy gap of CdO films were found decrease with increasing laser deposition energy from 400 to 700 mJ.

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تحضير ودراسة الخصائص البصرية والتركيبية لأوكسيد الكادميوم (CdO) النانوي باستخدام الترسيب بالليزر النبضي

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الملخص

تم تحضير اغشية رقيقة من حبيبات اوكسيد الكادميوم (CdO) النانوية على شرائح زجاجية باستخدام تقانة الترسيب بالليزر النبضي(PLD). استخدم ليزر النديميوم–ياك وعند الطاقات المختلفة (400,500,600,700) ملي جول، وعدد النبضات يساوي (100) عند تحضير جميع الاغشية الرقيقة. وبعدها درست الخصائص التركيبية والبصرية لهذه الاغشية. اظهرت نتائج حيود الاشعة السينية (XRD) بأن جميع النماذج المحضرة هي من نوع متعددة التبلور وان المستويات البلورية العالية والمهيمنة كانت (111) عند الزاويا ($2\theta=32.986$). وبينت نتائج مجهر القوة الذرية (AFM) بأن الحجم الحبيبي للأغشية المرسبة يزداد من nm(50.63−85) مع زيادة طاقة الليزر من (400mJ) الى (700mJ). كما ان طيف الامتصاصية يزداد من (0.53−1.75) مع زيادة طاقة ترسيب الليزر، بينما طيف النفاذية يقل مع زيادة طاقة الترسيب لجميع النماذج المحضرة بحيث تبلغ النفاذية (% 26.53) عند طاقة الترسيب (400mJ) الى ان تصبح (% 1.70) عند طاقة الترسيب (700mJ) وعند الطول الموجي (550nm)، اما فجوة الطاقة فتقل مع زيادة طاقة الترسيب وتتراوح من (2.5eV-2.3eV).