The Structural and Surface Morphology Properties of Aluminum Doped CdO Thin Films Prepared by Vacuum Thermal Evaporation Technique.

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Abstract

Undoped and Al-doped CdO thin films have been prepared by vacuum thermal evaporation on glass substrate at room temperature for various Al doping ratios (0.5, 1 and 2)wt.% . The films are characterized by XRD and AFM surface morphology properties. XRD analysis showed that CdO:Al films are highly polycrystalline and exhibit cubic crystal structure of lattice constant averaged to 0.4696 nm with (111) preferred orientation. However, intensity of all peaks rapidly decreases which indicates that the crystallinity decreases with the increase of Al dopant. The grain size decreases with Al content (from 60.81 to 48.03 nm). SEM and AFM were applied to study the morphology and to estimate the surface roughness of the obtained films. All films were homogeneous and smooth, with a characteristic spherical grain size depending on Al content. The (RMS) roughness of the films increases with the increase of Al dopant. The improvement of the structural and surface morphology properties of Al-doped CdO has potential applications for different optoelectronic device applications.

Keywords: CdO:Al thin films, structure properties, XRD, AFM, SEM.

Introduction

Transparent conductive oxides (TCO) are a type of semiconductor oxides of high conductivity arising from structural metal interstitials and oxygen vacancies. It is well known that high carrier mobility μ_H is essential for TCOs with good quality electro-optical properties[1]. From other side sometimes it is necessary to hybridize TCO in order to get some properties for various advanced technology applications, the hybridization can be realized by doping with elements having the required wanted property to be appeared in the TCO. Among these TCOs, CdO is an important material for the fundamental studies. It is now well conceived that the CdO shows many excellent properties, which make it suitable as a TCO [2]. CdO is a degenerate n-type semiconductor with a rock-salt crystal structure (FCC). Its high electrical conductivity (10^2-10^4) $\Omega^{-1}cm^{-1}$ and high optical transmittance in visible and NIR spectral regions with a moderate reflective index make it useful for various applications, photo diodes, gas sensors devices, transparent electrodes, photo transistors, photovoltaic solar cells, etc[2-5] with a direct band gap of (2.2-2.7) eV [4-7]. The n-type electrical conduction in CdO and the low resistivity of CdO thin films are due to Cd interstitial (Cd_i) and oxygen vacancies, however the oxygen vacancies is dominate defect acting as doubly ionized (+2) charge shallow donors [6].

The carrier mobility in CdO films could be improved by doping with different metallic ions. It was established experimentally that doping of host CdO by ions of radius slightly smaller than that of Cd^{2+}, like Tl [8], Y [9], In [10], Sm[11] and Al [12-14], could improve the electronic mobility and conductivity of carriers.

In this paper, we investigate both the structural and surface morphology properties for undoped CdO and Al-doped CdO films with various low percentages of Al. Our aim is to improve new nanostructure CdO films to control their structural and surface morphology properties to be utilized in new applications in optoelectronic devices. We conclude that Al as an incorporation element can be used to change structural and surface morphology properties to raise the chemical stability.

Experimental details

CdO:Al films have been deposited on glass substrate by thermal vacuum evaporation using (Edwards- Unit 306) system in vacuum about $4.5x10^{-5}$ mbar at room temperature. The thickness of the films were determined with (Precisa-Swiss) microbalance by using weighing method and found to be about (300 ∓ 10) nm, with deposition rate about (1 ∓ 0.1) nm/sec.

The metal bulk Cadmium thin films (from Fluka A.G company / Germany), were evaporated in vacuum at room temperature onto cleaned glass substrates with (2.5 x 2 x 1)cm^3 size. The distance between the substrate and the boat is (18) cm. A thin layer of CdO is formed on a chemically deposited Cd thin film through reaction with atmospheric oxygen during heating by (VECTOREEN model) oven for one hour at 400 °C. During the heating process, the color of the Cd films changed from silver-grey at room temperature to a black-brown color at a temperature of oxidation. So that Al atomic percentages doping in the films were (0, 0.5, 1 and 2)% wt.

The crystal structure of these films was checked by X-ray diffraction technique, (XRD) patterns were obtained with a (SHIMADZU Japan -XRD600) automatic Diffractmeter using the CuKα radiations (λ=1.54059 Å) in the range of 2θ between 25° and 75°. Surface morphology was studied using (VEGA3-TESCAN model, USA) scanning electron microscope (SEM). Atomic force microscopy (AFM) measurements were carried out using (SPM model AA 3000 Angstrom Advanced Lns., USA) to determine the nanocrystalline topography and grain size of the films.

Results and Discussion

1. Crystal structure:

(XRD) spectra of the undoped and Al-doped CdO films are shown in Fig. 1. The existence of multiple diffraction peaks of (111), (200), (220), (311) and (222) planes indicates the polycrystalline nature of the CdO films compound with cubic NaCl structure. The d-space 'd' and the lattice constant 'a' of the prominent peaks ((111), (200) and (222)) were calculated and presented in Table. 1. The XRD peaks coincide with those of cubic crystal structure according to ASTM card No. 05-0640 [15]. The observed 'd' and 'a' values are in good agreement with standard 'd' and 'a' values taken from (ASTM) data as shown in Table (1).

The lattice constant parameter 'a' of the cubic structure of CdO films is calculated using the following formula [16]:

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

The calculated lattice constant for the dominant peaks of (111), (200) and (222) of CdO is averaged to a = 4.6963 Å which is close to the reported value [13, 16, 17, 18]. However, intensity of all peaks rapidly decreases as shown in Fig. 1 and full width at half maximum (FWHM) increases for the films with the increase of Al content. This behavior can be associated with the presence of Al-Cd compounds in the amorphous phase and with a diminishing of the CdO grain size 'D' [19]. The lattice parameter 'a' decreases from 4.6968 to 4.6763 Å when Al percentage increases from (0 to 2)% wt. for (111) plane as shown in table (2).

The relatively stronger intensity of the peak indicates preferential (111)orientation of the films or indicating a strong orientational growth along that plane, similar behavior has also been reported by other researchers [12, 20-22]. The undoped thin film has shown the highest (111) diffraction peak intensity while the peak intensities of the aluminum doped films decreased with doping concentrations, which indicates that an increase in doping concentration deteriorates the crystallinity of the films. For polycrystalline semiconductor, dopants are known to segregate in grain boundaries in electrically inactive configurations [23-25]. There is always a tendency for the aluminum dopants to segregate at the grain-inter particle boundaries even for a low Al doping concentration [26].

Considering the above mentioned cases, the deterioration of crystallinity in the present CdO:Al films may be attributed to the deformation in the CdO lattice induced by the ion size difference between Cd (ionic radius=0.095 Å) and Al (ionic radius=0.0535 Å) and excess Al may also occupy interstitial positions in CdO lattice resulting in distorted crystal structure, thus decreasing the carrier mobility [27], and an increased segregation of aluminum in grain boundaries inhibiting the crystalline formation as a function of doping concentration. This is evident by the peak width broadening with increasing Al content, which suggests that the grain size of the CdO:Al thin film decreases as a function of Al concentration as shown in table(2). The grain size value 'D' of the films was estimated from the Scherrer's equation[28]:

$$D = \frac{0.9\,\lambda}{\beta \cos(\theta)} \quad \text{..... (2)}$$

Where β (FWHM), is the full width at half maximum of diffraction peak measured in radians units, θ is the Bragg angle and λ is the X-ray wavelength used (λ=1.54059 Å).

The grain size associated to the (111) direction was found to decrease from 60.8 nm (undoped) to a minimum about 48 nm for an Al concentration of 2 wt.% as shown in Fig. 2, this behavior is consistent with previous report of Al-doped CdO films prepared by sol-gel [28], spray pyrolysis [13, 18, 29] and by ultrasonic spray pyrolysis technique [30].

The dislocation density (δ) is defined as the length of dislocation lines per unit volume of the crystal, has been estimated using the equation [31]:

$$\delta = \frac{1}{D^2} \quad \text{..... (3)}$$

The δ-value is criterion of crystallization level. Lower δ-values indicated higher crystallinity levels for the films, since δ is the measure of the amount of defects in a crystal and the value of dislocation density obtained in this work is found to be equal to $2.7x10^{14}$ lines/m^2 for undoped films. The small value of δ obtained in the present work confirms the good crystallinity of the CdO film fabricated by employing this thermal evaporation technique.

The number of crystallites per unit surface area (N) of the film was determined using the formula [31]:

$$N=\frac{t}{D^3} \quad \text{.....} \quad (4)$$

Where, t is the thickness of the films. The value of N was found to be equal to $1.334x10^{15}/m^2$ for undoped films. The variation of δ and N as a function of Al dopant as shown in Fig. 3 and table (2), were increased with the increase of Al dopant concentration in the films.

2. (AFM) analysis:

AFM scans of the surface morphology were carried out to study the change in the surface morphology of the films. AFM images of pure and Al doped CdO thin films are shown in Fig. 4. These images show that the film is homogeneous and it has a large number of vertically aligned (columnar) grains, uniformly distributed features with no pinholes or island structures are observed in all the thin films. It can be seen in Fig. 4 that the surface roughness of the CdO film is changed with the increase of Al-doping level from (0 to 2)% wt. The increase in pyramidal shape with increases Al dopant, indicate that the increase in the surface roughness affects the surface characterization of the films and leads to changes in the optical, electronic, and vibrational transitions of the material. [32].

Nanoscale surface roughness of the films was calculated by section analysis of the height image. The section analysis of the height image indicated increase in nanoscale roughness for films with additives. AFM phase image can be used to map stiffness difference on the surface [33]. The grain size and the root-mean-square (RMS) roughness of the samples were estimated from AFM images and the results have been shown in table (3). The measured (RMS) roughness of the (undoped, 0.5, 1 and 2)%wt. Al doped cadmium oxide films were (0.76, 0.96, 1.03 and 1.18) nm, respectively (table 3). It is less than 1.2 nm over a (2μm×2μm) area for all films; these values are consistent with previous reports for undoped and doped CdO thin films prepared by various techniques [34-36]. It is evaluated that the surface roughness of the films depends on the distribution of particles on surface of the films. Further, the roughness of the samples increases with the increase of Al concentration as shown in Fig.(2). This behavior is similar to CdO:Al thin films of the previous reports [14, 29]. Therefore, the increases in roughness of the film surface indicate the possibility of using the synthesized films as anti-reflection-coatings, which reduce light reflection, but increase light absorption in the visible region of the solar spectrum [37].

It is observed from Table (3) of AFM analysis, that the grain size or the particle size of CdO is decreased with increase in Al-doping percentage. The AFM observation is consistent with the above XRD results. From table (2) & table (3), it seems a lower estimate of the corresponding size determined by the XRD analysis. The disagreement between the grain size determinations from XRD and AFM measurements is expected because the AFM measurement directly visualizes the surface grains only without considering the degree of structural defects [38], while the XRD determination is based on the grain size of the defect-free volume [39] (common lattice defects in metal oxide semiconductors are for instance oxygen vacancies and metal atoms on interstitial lattice sites) [40]. So, we conclude that an average grain observed by the AFM contains other lager size crystallites belonging to

different orientations, as observed in the XRD [38]. AFM images are consistent with pervious report of Al-doped CdO films prepared by spray pyrolysis method [29].

3. (SEM) analysis:

Fig. 5 shows scanning electron micrographs (SEM) of the nanostructured undoped and Al–doped CdO films. From the first image (Fig. 5a) it seems that the particles have rounded shape less than 1 μm diameter. But the diameter decreases in second image (Fig. 5b), while the third and fourth images (Fig. 5c,d), the particles shape is like pyramids and the number of pyramids-like particles increased with Al doping. Furthermore, at the fourth image, we can see that the pyramids-like shape particles are more homogeneous than the third image. All the films were homogenously distributed and have dense uniform spherical crystalline grains for undoped and 0.5%wt. doped films (Fig. 5a,b), and granular surface properties with uniform pyramids shape of single mode size distribution of grains are observed for 1% and 2wt.% doped films (Fig. 5c,d). Also, it is well hold and well adherent with glass substrate surface without cracks.

With the Al-doping, surface becoming homogeneous and fairly smooth. This array completely diminished and surface becomes highly smooth and homogenous for 2%wt. Al-doped sample (Fig. 5d). The SEM images show that the surface morphology of the films is strongly dependent on the concentration of aluminum, that suggests Al acts as an agent provide homogeneity and to improve film quality. Generally, the low ratio doping samples exhibited a porous microstructure and spherical crystalline surface particles. Furthermore, when the doping concentration increases the CdO particle size decreases. These results are consistent with above XRD and AFM results. The change of particle size can be attributed to the almost 43% difference in ionic radius between cadmium (0.095 nm) and aluminum (0.0535 nm) [41]. However, the SEM images confirm the AFM images. SEM images are consistent with pervious report of Al-doped CdO films prepared by sol–gel process [12].

Conclusion

The undoped and Al-doped CdO films were deposited by Physical Vapor Deposition in vacuum technique. The structural and surface morphology properties of these films were investigated as a function of Al doping concentration at room temperature. Well adherent, homogeneous, smooth and crack-free thin films with nano-grain size were successfully grown on glass substrate utilizing a new precursor, by PVD method. XRD patterns shows that it has polycrystalline nature with cubic crystal structure with (111) plane as preferential orientation. SEM images revealed a spherical and pyramids shapes-grain surface with single mode size homogenous distribution of grains. The mean values of nano-clusters size and roughness for pure and Al-doped CdO were determined by using AFM. AFM imaging indicated high smooth surfaces with small RMS roughness values and the results show moderate autosimilarity of the film surfaces and the Surface morphology of CdO film can be improved substantially by Al-doping. The correlation between structural properties and morphology of the films have shown that the grain size decreased with the increase of Al concentration and RMS roughness. The grain size of undoped CdO film is 60.81 nm which decreases with the increase of Al content. Finally, it may be concluded that vacuum thermal evaporation technique is a good technique for producing nano-structure material. In addition, the surface morphology of the films plays an important role on structural, optical and electrical behavior of Al doped CdO thin films.

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Table No.(1): XRD, results of CdO:Al thin films compared with (ASTM) card.

Sample	2θ)ASTM(	2θ Observed	d (Å))ASTM(	d(Å) Observed	)hkl( )ASTM(	a (Å) (ASTM)	Å) a (Observed
CdO (Pure)	33.000 38.283 69.284	32.9807 38.2722 69.2869	2.7120 2.3490 1.3550	2.7117 2.3482 1.3556	111 200 222	4.695	4.6968 4.6964 4.6959
Al (0.5%)/CdO	33.000 38.283 69.284	33.022 38.305 69.312	2.7120 2.3490 1.3550	2.7086 2.3471 1.3540	111 200 222	4.695	4.6914 4.6942 4.6904
Al (1%)/CdO	33.000 38.283 69.284	33.104 38.370 69.315	2.7120 2.3490 1.3550	2.7021 2.3451 1.3521	111 200 222	4.695	4.68017 4.6902 4.6838
Al (2%)/CdO	33.000 38.283 69.284	33.118 38.450 69.361	2.7120 2.3490 1.3550	2.6999 2.3431 1.3508	111 200 222	4.695	4.6763 4.6862 4.6763

Table No.(2): XRD, results of CdO:Al thin films for the (111) preferred orientation peak

Ratio	T_a (^oC)	d (111) (Å)	a (Å)	FWHM (111) (deg)	D (nm)	N_0x10^{15} (m^{-2})	$\delta x10^{14}$ (m^{-2})	Intensity (cps)
CdO(Pure)	R.T	2.7117	4.6968	0.14230	60.81	1.3341	2.7042	7113
0.5 % Al		2.7086	4.6914	0.1494	57.92	1.5439	2.9808	6530
1 % Al		2.7021	4.6801	0.1651	52.429	2.0816	3.6379	5854
2 % Al		2.6999	4.6763	0.1802	48.037	2.7064	4.3336	4765

Table No. (3): AFM analysis, the crystal grain size, RMS surface roughness of the pure CdOand Al doped CdO thin films.

Thin films	Grain size, D (nm)	(RMS) roughness (nm)
Pure (CdO)	79.53	0.76
0.5%Al - CdO	64.67	0.96
1% Al - CdO	57.87	1.03
2% Al - CdO	51.19	1.18

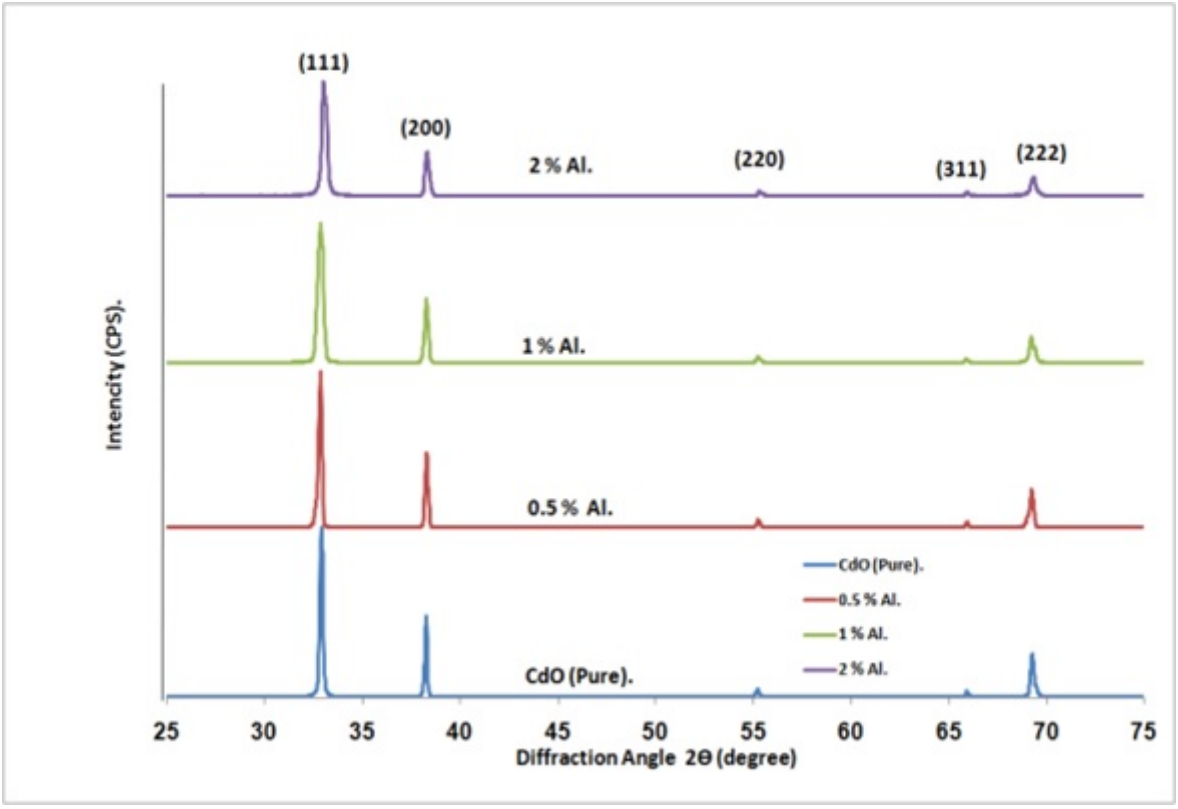

Figure No. (1): X-ray diffraction pattern of Al doped CdO thin films at room temperature.

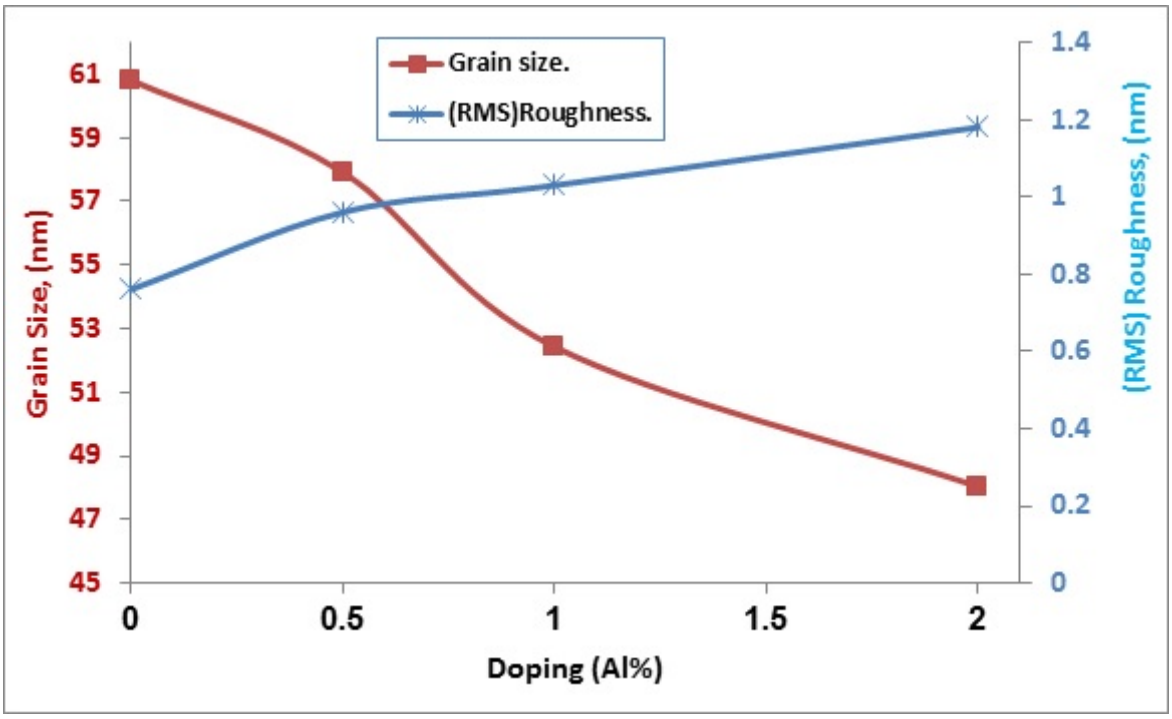

Figure No. (2): Variation of grain size and (RMS) roughness as a function of Al doping ratio.

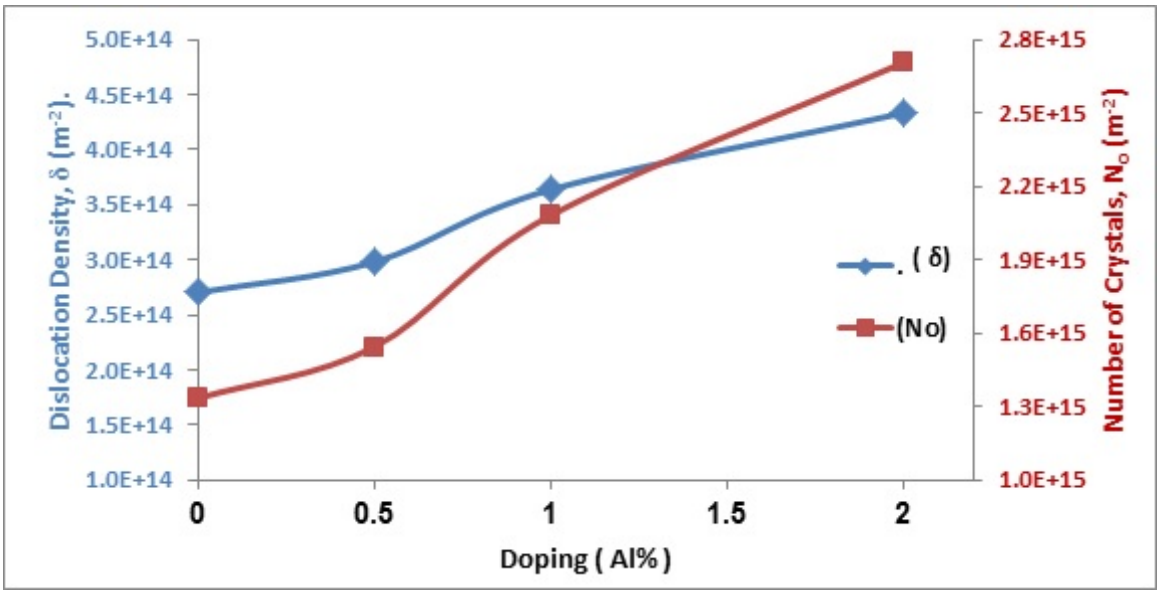

Figure No. (3): Variation of (δ) and (N_o) of CdO:Al thin films as a function of Al doping ratio

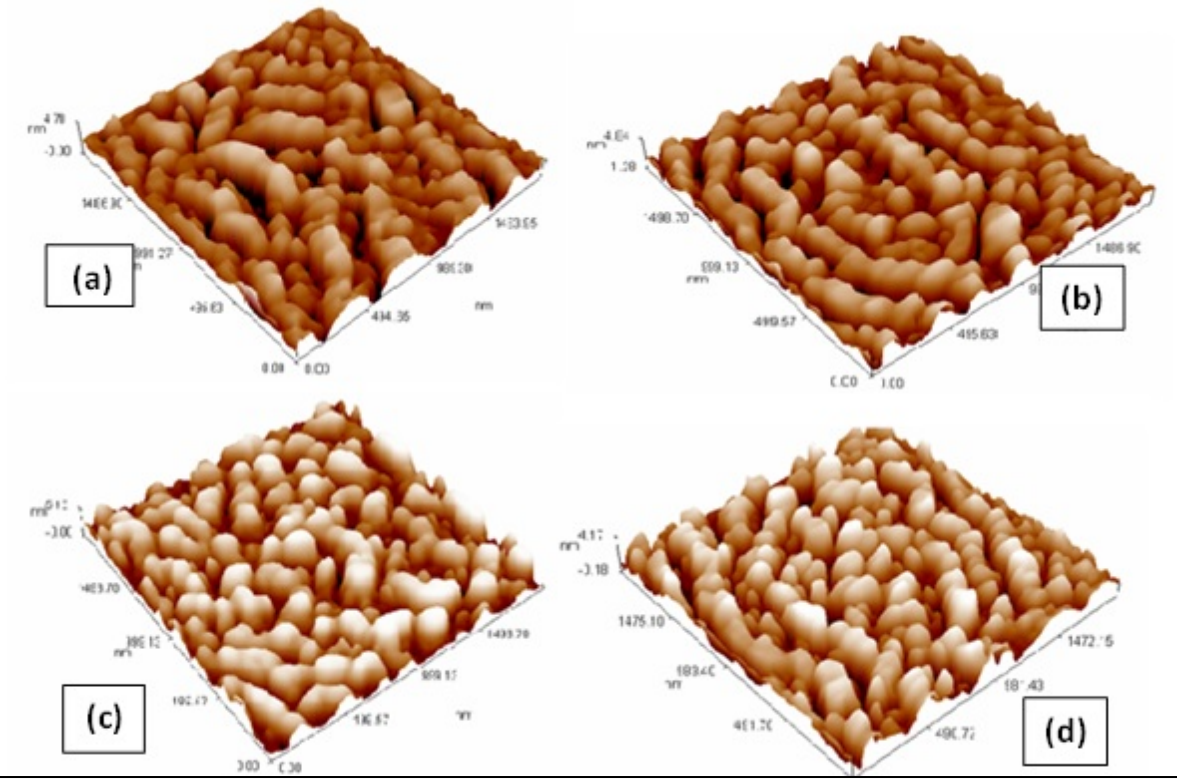

Figure No .(4): AFM-3D images of (a)0%wt , (b)0.5%wt, (c)1%wt, (d)2%wt Al doped CdO films.

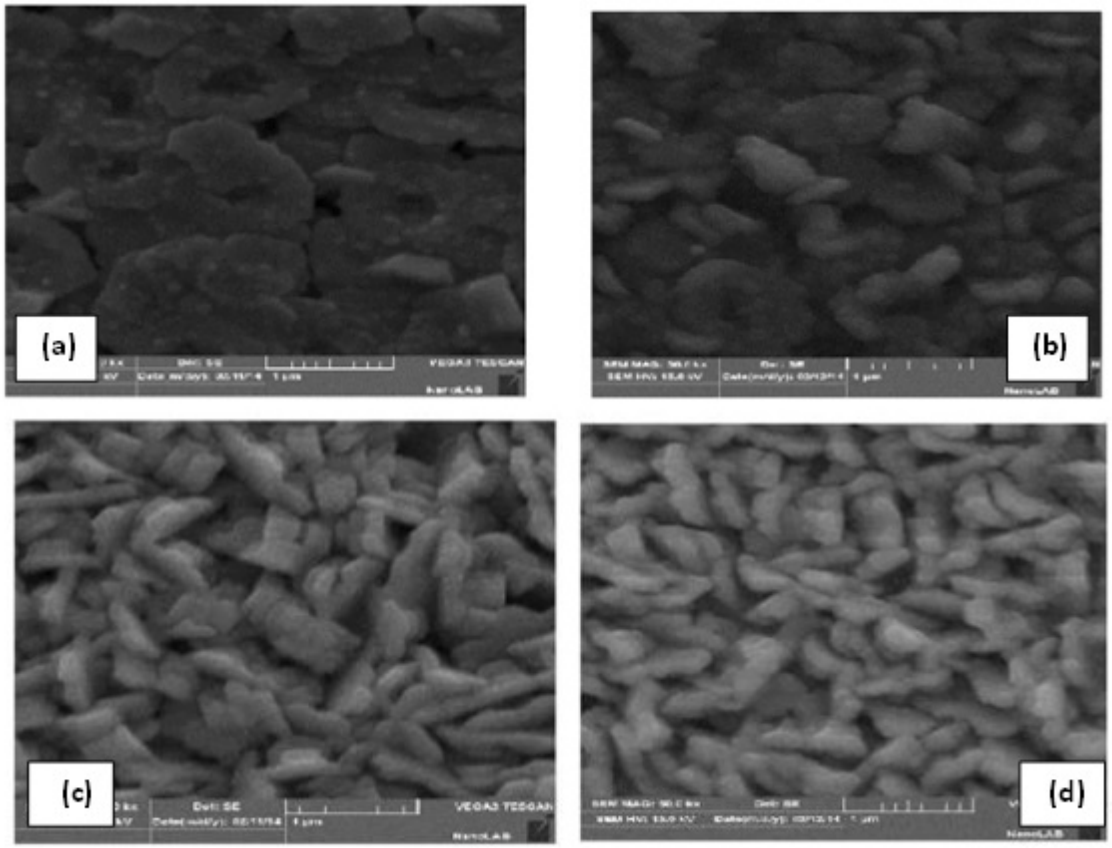

Figure No.(5) : SEM – 1 µm images of CdO thin films with various Al doping ratio (a) undoped , (b) 0.5 %wt. , (c) 1%wt. , (d) 2%wt

الخواص التركيبية ومورفولوجية السطح لأغشية (Al) النقية والمشوبة ب(CdO) والمحضرة بتقنية التبخير الحراري في الفراغ

علية عبد المحسن شهاب
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الخلاصة

حضرت أغشية أوكسيد الكادميوم (CdO) الرقيقة النقية والمشوبة ب (Al) بنسب تشويب %(0.5, 1, 2) وبإعتماد تقنية التبخير الحراري الفراغي على أرضيات زجاجية بدرجة حرارة الغرفة. دُرست الخواص التركيبية بأستخدام تقنية (XRD)، وكذلك مواصفات مورفولوجية السطح للأغشية المحضرة بإستخدام تقنية (AFM) و (SEM). أظهرت نتائج قياسات حيود الأشعة السينية (XRD) إن جميع الأغشية المحضرة النقية والمشوبة كانت ذا تركيب بلوري متعدد التبلور ومن النوع المكعب الذي يمتلك ثابت شبيكة طوله 0.4696 nm مع هيمنة النمو بالإتجاه (111) للأغشية النقية والمشوبة كافة مع إزاحة في زاوية الحيود، وحدوث أنخفاض في شدة القمم المقاسة مما يدل على نقصان التبلور وتناقص واضح أيضاً يظهر في معدل الحجم الحبيبي nm (60.81-48.03) عند زيادة التشويب بالألمنيوم (Al).
أستخدمت تقنية مجهر القوة الذرية (AFM) والمجهر الإلكتروني الماسح (SEM) لدراسة طوبوغرافية وخشونة سطوح الأغشية الرقيقة المحضرة. أظهرت نتائج فحوصات المجهر أن جميع الأغشية متجانسة وناعمة وأن شكل الحجم الحبيبي المتكون أشبه بالشكل الكروي بالأعتماد على نسبة التشويب، مع زيادة معدل خشونة السطح (RMS) بإزدياد نسبة التشويب. وأظهرت النتائج أن الخواص التركيبية ومورفولوجية السطح تتحسن لأغشية CdO بعد تشويبها بنسب قليلة جداً من Al مما يجعل هذه الأغشية واسعة التطبيقات في مجال النبائط الالكتروضوئية.

الكلمات المفتاحية:الأغشية الرقيقة (CdO/Al)، الخواص التركيبية ، XRD, AFM, SEM.