

Effect of Mg Molar Concentration on Structural and Optical Properties of CdO Thin Films Prepared by Chemical Bath Deposition Method

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ABSTRACT

In this study, pure and Mg doped CdO thin films were coated on glass substrate using a chemical bath deposition method. Adding different molar concentrations of magnesium (0, 2, 4, 6) % to the chemical bath solution and their effect on the structural and optical properties were studied. Oxidation of films was conducted at 573 K for 60 minutes in the presence of static air. The thin film properties were diagnosed with X-ray diffraction techniques and UV-VIS-spectrophotometer. The results of X-ray diffraction for the thin films showed that all prepared thin films have a face-centered cubic crystal structure (FCC) with a preference for growth at level (111), and the synthetic coefficients calculated from the X-ray spectrum are affected with increasing Mg ratios in the chemical bath solution. The results of the optical tests showed that the absorption of the resulting thin films increases with increasing Mg ratios in the chemical bath solution, have a high absorption coefficient in the visible region of the electromagnetic spectrum, and the transmittance decreases with the increase of Mg in the chemical bath solution. The optical energy gap of the prepared thin films changes with increasing Mg doping rates and extends from (2.46) eV to (2.95) eV. The results showed a significant improvement in the structural and optical properties of Mg ion doped CdO films making it suitable for use in many photovoltaic applications such as reagents and solar cells.

1- Introduction

Cadmium oxide is a semiconductor material located in the (II-VI) group in the periodic table of elements. It has a cubic crystalline structure and its cell unit is face-centered (FCC). It can be obtained by heating the element cadmium [1] and its color ranges from dark brown to yellowish green [2]. It has low electrical resistivity due to the presence of cadmium atoms in compensatory sites or as a result of the presence of oxygen spaces. It is also used in solar energy systems in order to increase its efficiency because it has a high absorption coefficient [3, 4]. It is regarded as a transparent semiconductor oxide which has special characteristics including high transparency in the visible region and the near and high reflective infrared regions of the electromagnetic spectrum [5]. Its energy gap is relatively big of about (2.7) eV [6] and also has high electrical conductivity when compared with the semiconductor conductivity

of the negative type [7]. It is, therefore, used in a wide variety of applications such as projectors and photovoltaic and electro-optical devices [8]. Modern science is based mainly on electronics, which in turn are based on semiconductor materials in the form of thin films, especially in solar cells, computers and integrated circuits [9]. In order to increase the efficiency of solar cells, photovoltaic cells are coated with thin films that have certain properties in order to absorb a certain part of the falling energy and reflect the other part. This property depends on the value of the energy gap being confined. Since the beam from the sun is within the visible part of the electromagnetic spectrum and in order to control the amount of energy absorbed or reflected, the study must choose semiconducting materials for which the energy gap is confined to approximate the energy of photons to the visible part of the spectrum [9].

Several methods were used for the preparation of pure and impure cadmium oxide thin films. One of these methods is the pulsed laser deposition, in which the effect of the addition of Al and Mg on thin film properties was studied [9]. Another is thermal evaporation technique where the thin films were turned into impure by magnesium [10]. In addition, the effect of gamma rays on the properties of CdO thin films tinged with Mg by thermal chemical spray technology was studied [11]. The spray pyrolysis technique was used to prepare cadmium oxide thin films doped with Mg and study the effect of Annealing Temperature on the Electronic Transitions [12]. Cadmium oxide was also doped with magnesium but in a spray pyrolysis technique [13]. In addition, other methods are used. In reviewing previous studies, the study noticed that work indicating the preparation of CdO thin films doped with Mg ions by the chemical bath deposition method.

In this research, a new attempt is made to prepare thin films of pure cadmium oxide doped with Mg with different doping rates, and also investigate the effect of doping on the properties of optical and synthetic thin films for the purpose of knowing the possibility of using it in different electro-optic devices. Different molar concentrations of Mg are added to the chemical bath solution of CdO thin films to improve some of the thin film properties such as energy gap, absorbance, absorption coefficient, permeability, crystalline size, and other properties.

2- Experimental Method

The chemical bath deposition method depends on many parameters, including the chemical composition of the materials used, acidity function pH, the temperature of the solution, the concentrations of the materials inside, the substrate used in deposition of thin films, deposition time, and temperature used in annealing [14]. For the preparation of the chemical bath solution, aqueous cadmium nitrate ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) supplied from (Fluka company) with (99.99% purity) was used as a source of cadmium ions (Cd^{+2}). Magnesium nitrate ($\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) supplied from (Laboratory Rasayan company) with a purity of (99.99%) was also used as a source of positive magnesium ions (Mg^{+2}). Ammonium hydroxide (NH_4OH) solution from (Fluka Company) with a concentration of (25%) was also used to adjust the pH value of the solution and as a source of negative hydroxide ions (OH^-). To prepare the thin film deposition solution of CdO thin films, a solution of (0.05) Molarity of aqueous ($\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$) was prepared by dissolving it in (100) ml of distilled water with magnetic stirring for 10 minutes to obtain a homogeneous transparent solution. Then, ammonium hydroxide was added by gradual distillation until the solution changes color to milky white. With continuous addition, the solution changes color again to transparent white. The pH value is measured at room temperature using a (German-

origin pH-meter) of the type (Itrans Bp3001) and was around (11.5). The doping solution was prepared using aqueous $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ at a concentration of 0.05M in 100ml of distilled water and mixed with a magnetic stirrer at room temperature for 10 minutes until a clear and homogenized solution was obtained. For doping, variable proportions of magnesium nitrate solution (0, 0.02, 0.04, 0.06) are added to the chemical bath solution of the thin CdO films, and stirred for 10 minutes until a clear and homogeneous solution is obtained. Glass micro slides as substrates supplied from (Superior Marienfeld) Company and made in Germany with a size of ($1.5 \times 25 \times 75$) mm^3 were used for deposition. It was cleaned with distilled water and then with laundry detergent and chromic acid followed by rinsing with distilled water again and finally treated with the ultrasound device for 15 minutes. Then, the glass substrates are immersed vertically in the chemical bath solution without magnetic stirring at room temperature for 48 hours. After that, the samples are lifted, dried and annealed in static air using a (Yamato FM27) electric oven at temperature (573) K for 60 minutes. The samples are kept in special cases and ready for measurements. The thickness of the prepared thin films is calculated by the gravimetric method in which a sensitive electronic digital scale of the type (Mettler HK-160) with sensitivity up to (10^{-4}) gm is used. To determine the crystal structure of the prepared thin films, the X-ray diffraction device (Shimadzu XRD-6000) of Japanese origin is used with the following specifications: wavelength (1.5404) Å and source (Cu K_α) voltages 40 volts. Optical properties were diagnosed using Taiwanese-made UV/Vis-Spectrophotometer of the type (SP-8001).

3. Results and discussion:

3.1 X-ray Diffraction:

Figure (1, a-d) shows the X-ray diffraction spectrum (XRD) of pure cadmium oxide thin films as well as magnesium doped at different magnesium ratios from (2%) to (6%). The figure also presents the appearance of peaks (111), (200), (222) and (311), with the prevailing crystal trend as (111). This represents the FCC-based polycrystalline synthesis when conforming to the International Center for Diffraction Studies card (JCPDS: 005-0640). The study also note that the decrease in intensity with the increase of magnesium ion concentration (Mg^{+2}) due to the entry of magnesium atoms (Mg) into the crystal structure of the thin film (CdO). When the concentration of magnesium ion is increased to more than (4%), the study noticed that an increase in the intensity of peaks and the appearance of additional peaks which represents the hexagonal magnesium phase when conforming to the International card (JCPD: 00-045-0946). Doping also increases the full width at half-maximum (FWHM) and consequently decreases the crystalline volume and keeps growth in the dominant direction at level (111) and the peaks creep towards small lengths compared to the sites of the peaks of the

pure thin films. This is due to the difference in the amount of ion diameter in relation to the cadmium ion and magnesium ion, in which the cadmium ion diameter is (0.95 Å) and the magnesium ion diameter is (0.72 Å). This allows magnesium atoms to occupy interfacial sites in the crystal structure of the material and this reduces the distances between the crystalline levels and consequently decreases in the crystalline size. The peaks change and some other peaks are absent due to the relatively low grafting rate This is consistent with previous studies [14,15].

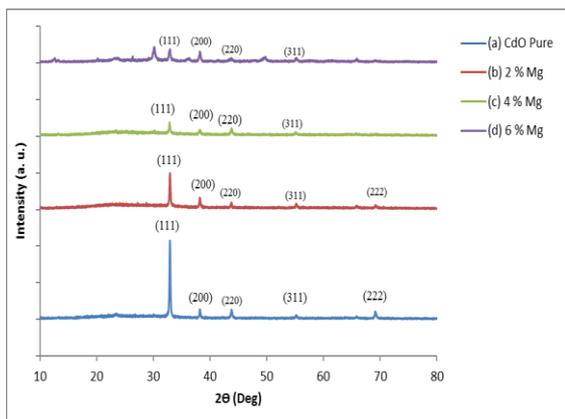


Fig. 1: X-ray diffraction of pure CdO films grafted with variable magnesium ratios.

The average crystalline size (C.S) of pure CdO thin films as well as Mg grafted at the predominant crystalline level (111). It can be calculated by the Scherer's Equation [16]:

$$C.S = \frac{0.9\lambda}{\beta \cos\theta} \dots (1)$$

where: (C.S): average grain size (nm). λ : wavelength of dropped X-ray (1.54 Å). θ diffraction angle and β : full width at half-maximum (FWHM). Figure (2) shows the average crystalline size change with increasing magnesium ions in the chemical bath solution. The study noticed that from Fig. (2), the average crystalline size decreases from (46.7) nm to (21.4) nm with the increase in the molar concentration of magnesium from (0 to 6%) as a result of stresses caused by the entry of magnesium atoms (Mg) and occupying interfaces in the crystalline structure of the CdO thin film leading to reduction in the dimensions of the crystalline granule and thus decreasing the crystalline size This is consistent with previous studies [15,17,18].

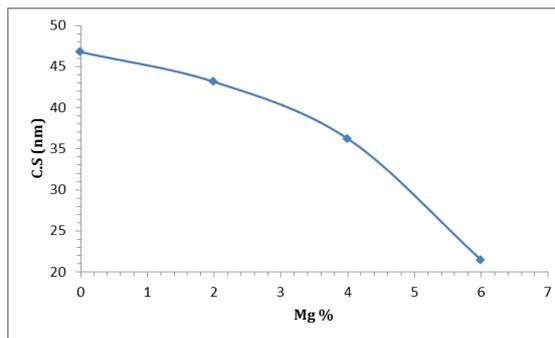


Fig. 2: Crystal size change with increased magnesium doping rates

Table (1) shows a set of structural coefficients for the prepared thin films, including the lattice constant (a), which was calculated using the following equation [19]:

$$d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}} \dots (2)$$

where (d): the distance between the crystalline levels (Å). And (a): the lattice constant (Å). And (hkl): Miller's coefficients. To calculate the curve width at the half-maximum (β), it is important use the following relationship [20]:

$$\beta = FWHM \times \frac{2\pi}{360} \dots (3)$$

where (β): the full width at the half-maximum according to radian units. The density of dislocation line (δ) is calculated using the following relationship [19]:

$$\delta = \frac{1}{(C.S)^2} \dots (4)$$

Where (δ): (Dislocation Line/nm²).

To calculate the number of crystals per unit area (N_o), it is significant to use the following relationships [21]:

$$N_o = \frac{t}{(C.S)^3} \dots (5)$$

Where (N_o): the number of crystals per unit area (Crystal / nm²). And (t): prepared film thickness (nm). It can be calculated thickness of films by using the following relationship [22]:

$$t = \frac{\Delta m}{\rho_t A} \dots (6)$$

Where (t): the thickness of film (nm). And (Δm): The difference in film weight before and after deposition (gm). And (ρ_t): The density of cadmium oxide and magnesium (gm/cm³). And (A): Film area (cm²).

The thickness of films from equation (6) is (207,324,412.518) \pm (15) nm doping ratios (0%,2% , 4%, 6%) respectively.

The results obtained from the above equations listed in Table (1) show that the values of the lattice constant (a) change each time depending on the values of the distance between the crystalline levels (d_{hkl}) according to equation (2). The granule size (C.S) reduces depending on the cosine of the diffraction angle and the beam full width at greatest measured half-maximum intensity ($\beta \cos \theta$) due to the entry of magnesium ions into the crystalline structure

of cadmium oxide according to equation (1). As for the dislocation density line (δ), it increases since the relationship between it and the granule size (C.S) is an inverse relation according to equation (4). The number of crystals per unit area (N_o) also increases depending on equation (5) since the relationship between it and the granular size (C.S) is an inverse relationship.

Table 1: Results of X-ray diffraction examination of thin films prepared by (CBD) method.

Sample	2 θ (Deg)	(hkl)	d (Å)	a (Å)	FWHM (Deg)	FWHM(β) (RAD)	C.S (nm)	$\delta \times 10^{14}$ (1/m ²)	$N_o \times 10^{15}$ (1/m ²)
CdO Pure	32.9470	(111)	2.71642	4.7049	0.17740	0.00309	46.774	4.5707	2.0228
2% Mg	32.9555	(111)	2.71574	4.7037	0.19230	0.00335	43.145	5.3720	4.0341
4% Mg	32.8963	(111)	2.72049	4.7120	0.22920	0.00399	36.219	7.6230	8.6713
6% Mg	32.9066	(111)	2.71966	4.7105	0.38670	0.00674	21.441	21.7525	52.5526

3.2 Optical properties:

To know the properties of optical thin films and the possibility of use in practical applications, these properties must be studied including the absorption spectrum since it depends on the film material and its crystal structure in addition to the amount of energy of photons dropping on the material and this spectrum is considered a distinguishing characteristic of the material [23]. The absorption spectrum of thin films with wavelength ranges between nm (275-900) nm was studied. Figure (3) shows the absorbance spectrum of prepared thin films as a function of wavelength. It is noted that the absorption is less in case the film is not doped. In the case of doping, it gradually increases with magnesium ion levels in the solution and becomes (0.21) for the pure thin film, i.e. when the concentration of magnesium ion is (0%), and increases with the increase in the concentration of magnesium ion of the thin film (0.31,0.49,0.71) for doping ratios (2% ,% 4,% 6) respectively. The increase in doping ratios will be additional levels near the conduction beam, increasing the likelihood that low-energy photons will be absorbed, and in turn increasing the probability of electronic transitions between the valence and conduction beams. This is consistent with previous studies [14].

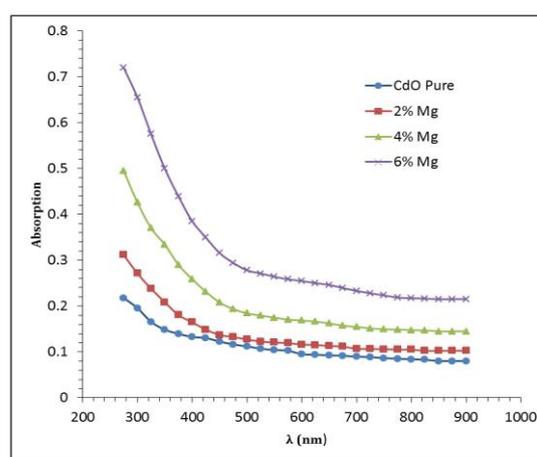


Fig. 3: Absorption spectrum is a function of the wavelength of pure CdO thin films doped with variable magnesium ratios.

Figure (4) shows the optical transmittance spectrum of pure cadmium oxide thin films doped with variable magnesium ratios as a function of wavelength. It is noted that there is an increase in the spectrum in the visible area and near infrared and is a relative change in the basic absorption edge of all grafting ratios towards long wavelengths. Also, by increasing the molar concentration of magnesium, the permeability decreases from (83%) for the pure cadmium oxide thin film to (71%) at doping ratio (4%), and the permeability is lower at doping ratio (6%) reaching to about (60 %). This decrease is due to the increase in thin film thickness by increasing the magnesium doping rates. It is also attributed to the presence of localized levels within the crystal structure of the material above the valence bundle because of doping. These levels receive electrons and generate tails inside the energy gap and consequently reduce the energy gap. In addition, it the increase of wavelength increases the value of permeability and this is consistent with previous studies [24,25].

The optical energy gap is one of the most important optical constants that is depended on within semiconductor physics such as the manufacture of reagents, solar cells, photodiode and other applications. Materials with an energy gap comparable to the energy of the dropping photons are selected from within a specific portion of the electromagnetic spectrum as needed, and photons which are absorbed or reflected by dropping on the film are being controlled [26].

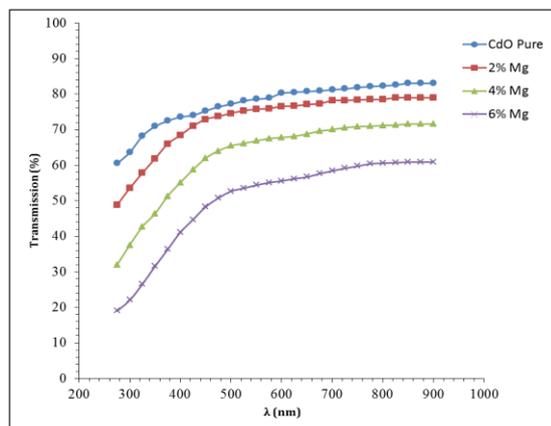


Fig. 4: Optical permeability spectrum of pure CdO thin films doped with variable magnesium ratios.

The optical energy gap of pure CdO thin films grafted with variable magnesium ratios can be calculated using the following direct electronic transition equation [27]:

$$(\alpha h\nu) = B(h\nu - E_g)^r \quad (7)$$

where: B: constant based on photon energy, and α : absorption coefficient, and r: factor determining the type of transition within the energy gap and E_g : optical energy gap. The energy gap can be calculated in practice "for the allowable direct transition ($r = 1/2$) by plotting the relationship between $(\alpha h\nu)^2$ and $(h\nu)$ and taking the straight-line tangent" to the curve of the absorption edge when $0 = (\alpha h\nu)^2$. Figure (5) shows the change in the value $(\alpha h\nu)^2$ with the photon energy ($h\nu$) of the prepared thin films. It is preferable to focus on the figure (5) that the energy gap value increases from (2.46) eV to (2.95) eV with an increase of magnesium ion in the chemical bath solution from (0%) to (6%), respectively. The increase in the energy gap value by increasing magnesium grafting rates is due to the increase in free electron concentration, which shows that most of the (Mg^{+2}) ions will be replaced by (Cd^{+2}) ions in the host lattice[28]. This is consistent with previous studies [13,24].

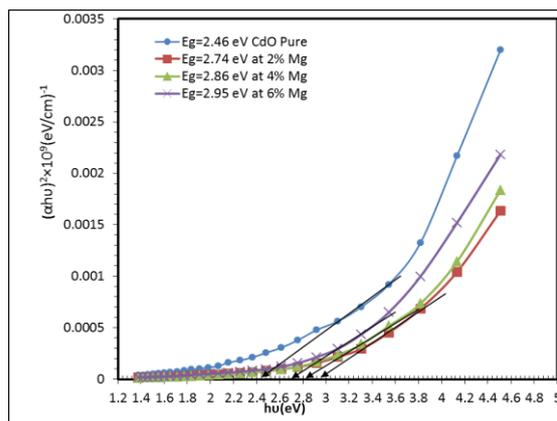


Figure (5): $(\alpha h\nu)^2$ is a function of the dropped photon energy ($h\nu$) of pure CdO thin films doped with variable magnesium ratios.

When drawing between the magnesium doping rate and the energy gap, it is better to observe that when the doping rates increase, the energy gap increases as shown in the following figure(6). This is consistent with previous studies [24].

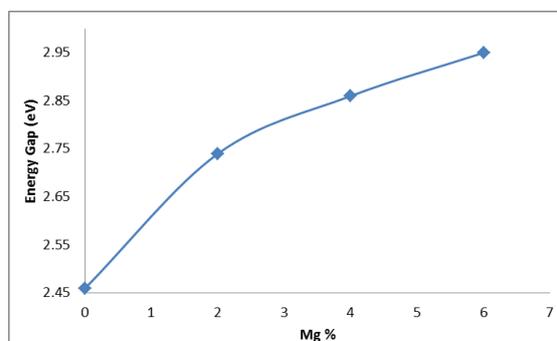


Fig. 6: Increased energy gap values with increased magnesium doping rates.

4. Conclusion

The study suggests that conclude there is a possibility to prepare thin films of cadmium oxide doped with magnesium by the chemical bath deposition method. The increased molar concentration of magnesium in the chemical bath solution of CdO thin films has a pronounced effect on the studied structural and optical properties. The crystal structure of pure CdO thin films doped with magnesium in different molar ratios all have a polycrystalline faced centered cubic (FCC) structure with a predominant crystalline direction at level (111). Structural coefficients such as the rate of crystalline volume, lattice constant, density of dislocations, a number of crystals per unit size and configuration factor change with increasing magnesium molarity in the chemical bath solution of CdO thin films. Optical absorbance increases and permeability decreases with increasing molarity concentration of magnesium. The optical energy gap increases from (2.46) eV to (2.95) eV with an increase in magnesium ion content in the chemical bath solution. There is a clear improvement in the structural and optical properties of the prepared thin films, indicating that they can be used in electronic applications.

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تأثير التركيز المولاري للمغنسيوم على الخصائص التركيبية والبصرية لأغشية CdO الرقيقة المحضرة بطريقة الترسيب بالحمام الكيميائي

فارس صالح عطاالله ، هاني هادي احمد ، وليد خالد جاسم

قسم الفيزياء ، كلية العلوم ، جامعة تكريت ، تكريت ، العراق

المخلص

في هذه الدراسة حضرت أغشية رقيقة من مادة أكسيد الكاديوم النقية وكذلك المطعمة بالمغنسيوم على ارضيات زجاجية بطريقة ترسيب الحمام الكيميائي. وتم دراسة تأثير إضافة تراكيز مولارية مختلفة % (0,2,4,6) من المغنسيوم الى محلول الحمام الكيميائي على الخصائص التركيبية والبصرية. أجريت عملية الأكسدة للأغشية درجة حرارة 573 K لمدة ساعة بوجود الهواء الساكن. تم تشخيص خصائص الاغشية المحضرة بتقنيات حيود الاشعة السينية (XRD) وجهاز (UV-VIS-spectrophotometer). نتائج حيود الاشعة السينية بالنسبة للأغشية بينت بأن الأغشية المحضرة جميعها ذات تركيب بلوري متعدد التبلور من النوع المكعب (FCC) مع أفضلية للنمو عند المستوي (111) وان المعاملات التركيبية المحسوبة من طيف الاشعة السينية تتأثر مع زيادة نسب (Mg) في محلول الحمام الكيميائي. نتائج الفحوصات البصرية بينت أن الامتصاصية للأغشية الناتجة تزداد بزيادة نسب (Mg) في محلول الحمام الكيميائي ولها معامل امتصاص عالٍ بالمنطقة المرئية من الطيف الكهرومغناطيسي وأن النفاذية تقل مع الزيادة في نسبة (Mg) في محلول الحمام الكيميائي. فجوة الطاقة البصرية للأغشية المحضرة تتغير بزيادة نسب التطعيم بالمغنسيوم وتمتد من (2.46eV) الى (2.95eV). أظهرت النتائج تحسن كبير في الخصائص التركيبية والبصرية لأغشية (CdO) المطعمة بأيون المغنسيوم مما يجعلها ملائمة للاستخدام في العديد من التطبيقات فوتوفولتائية كالكواشف والخلايا الشمسية.