Effect of deposition time on the structure, direct and indirect energy gap of nanoparticles \( \text{CdO} \) thin films deposited by chemical bath deposition technique

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Abstract. In this research, cadmium oxide (CdO) nanoparticle thin films have been prepared at room temperature using a chemical bath deposition (CBD) technique, and the effect of the deposition time were studied. CdO thin films have been deposited on glass substrate from cadmium chloride (\( \text{CdCl}_2 \)) as \( \text{Cd}^{2+} \) ions source and sodium hydroxide NaOH as \( O^{-2} \) ions source. The pH value (acidity level) of the chemical bath was fixed at about 11. The CdO thin films structures were analyzed by X-ray diffraction. It shows that all the prepared thin films have a cubic polycrystalline structure with a preferential orientation along (111) plane. All structural parameters were calculated. Particle size for the preferential orientation is calculated between (19.1 - 35.5 nm). It is found that the grain size increased with increasing the deposition time. UV-Vis spectrophotometer was used to study the optical properties, and a blue shift in the absorption peaks was noticed. The energy gap values (direct transition and indirect) calculated from the absorption spectrum located between (3.026 - 3.409 eV) for direct transitions and (2.197-2.917 eV) for indirect transitions, and this indicates that all CdO thin films prepared nanoparticles. We found that the energy gap decreased with increasing the deposition time.

Keywords; CdO thin film, chemical bath deposition method, Nanostructured, energy band gap.

1. Introduction

Nanoparticles have attracted great interest in recent years due to their size dependent properties and wide range of applications [1, 2]. Transparent conducting oxides (TCOs), such as pure and doped, \( \text{ZnO}, \ \text{SnO}_2, \ \text{BaO}, \ \text{Fe}_2\text{O}_3, \ \text{BiClO}, \ \text{Cu}_2\text{O} \) and CdO have been studied because of their utilization in optoelectronic device technology. Particularly, \( \text{CdO} \) based transparent conducting oxide (TCOs) are of great interest due to their metal like good optical transparency in the visible region and charge transport behavior with an exceptionally large carrier mobility [3]. CdO compound is reddish brown in color, and is formed by burning of Cd in air. The oxide is not soluble in water, [4]. CdO is a known n-type semiconductor with the direct band gap energy of 2.2 - 2.8 eV [5], 2.5 eV [6], 2.3 eV [7], 2.25-2.9 eV [8], 2.2-2.5 eV [9],
and the indirect band gap of 1.98 eV [6], 1.36 eV [7], 1.36-1.98 eV [9]. In fact, the differences in the band gap energy are due to the lattice’s defects in the crystalline structure [9]. Due to the defect of oxygen vacancies and cadmium interstitials, CdO has special features such as low resistivity (10^{-2} to 10^{-4} \Omega \text{ cm}) [8]. Also, low band gap and high transmission make it applicable solar cells, photovoltaic cell, photodiodes, phototransistors, transparent electrodes, optical communications, smart windows, flat panel display, liquid crystal displays and IR detectors [10, 11, 12]. There are different methods to prepare CdO thin films such as chemical bath deposition [13], Spray pyrolysis [14], Sol-gel [15] vacuum evaporation [16], successive ionic layer adsorption and reaction (SILAR) method [17]. Among these techniques, CBD method is widely used because it does not require vacuum and sophisticated instrumentation, the starting chemicals are commonly available (simple) and cheap (relatively cost effective) and can be applied in a large area of deposition at low temperature [18,19]. The optical absorption of CdO thin films were accomplished using UV-Vis spectrophotometer Cary50 Cm-Exlenain scan software in the wavelength range of [190-1100 nm] at room temperature, and the back correction of the glass substrate has been taken in each measurement. The structural characterization of the CdO thin films were accomplished by analyzing the X-ray diffraction patterns obtained using a computer software (MDI/JADE 5) Pgeneral XD-2 X-ray diffractometer (\lambda= 1.5405 \text{ Å} for Cu-K\alpha, current: 20.4 mA, voltage: 36.3 kV), The range of scan in 20 of (3-75° ) with scan speed 4°/min. The film thickness (t) was found by gravimetric weight difference method. This work focuses on the Synthesis of CdO nanoparticles thin film on glass substrate using (CBD) technique at room temperature and with different chemical deposition time. The structural and optical characteristics of the prepared films were investigated.

2. Preparations of CdO thin films
Microscopic glass slides of (75 × 25 × 1 mm^3) were used as substrates during the deposition process. The substrate was first cleaned with water and soap, after that immersed in chromic acid and boiling for one hour, then cleaned by De-ionized water. Finally it immersed in acetone and rinsed with De-ionized water to remove the surface contaminations and to make the surface more conductive for uniform film deposition. The deposition process was carried out at room temperature using cadmium chloride monohydrate (CdCl_2\cdot H_2O, Merck, 98% purity concentration (0.05 Mol.) as Cd^{+2} ion source and sodium hydroxide (NaOH, Scharlau, 99% purity).as O^{2-} ions as shown in Eq. (1)

\[
\text{CdCl}_2 + 2\text{NaOH} \rightarrow \text{Cd(OH)}_2 + 2\text{NaCl}
\]  

(1)

For the complex formation, an excess ammonium hydroxide (NH_4OH) solution was added (28%) until the clear solution reaches a pH of 11. This clear solution was kept under stirring, and then the substrates were submerged in the solution for (12, 24, 36 and 48 hrs.). Whitish films of Cd(OH)_2 were formed on glass substrates. The (Cd(OH)_2) films were heated in air at (623 K) for (2 hrs), and the films turn to brown. During the heating process the water vapor rises as shown in Eq. (2)

\[
\text{Cd(OH}_2) \xrightarrow{\text{heat}} \text{CdO solid + H}_2\text{O gas}
\]

(2)

These slides were inclined vertically at 20° angle to the wall of the beaker, and a 3 ml of hydrazine hydrate was added to enhance the adhesion under a constant stirrer (80 rpm). It is noted that the fresh solution should be continuously stirred to obtain uniform distribution of the chemical components,

3. Theory and calculation

3.1. Structural parameters

3.1.1. Interplanar distance (d)
Bragg’s equation was used to calculate the inter-planar distance (d) [20]
\[ n\lambda = 2d \sin \theta \]  

where \((n)\) is an integer order of the corresponding reflection, \((\lambda)\) is the incidents wavelength, \((\theta)\) is Bragg’s angle

### 3.1.2. Lattice constant \((a)\)

The cubic structure lattice constant \((a)\) is calculated using the relation [20]

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

where \((h k l)\) are Miller indices.

### 3.1.3. Crystallite size \((D)\)

Debye-Scherrer's formula was used to calculate the crystallite size \((D)\) [21]:

\[ D = \frac{0.9 \lambda}{\beta \cos \theta} \]  

where \((\lambda)\) is the wavelength \((0.15406 \text{ nm})\) for Cu-K\(\alpha\) source, \((\beta)\) is (FWHM) full width half maximum of the peak intensity in radians, and \((\theta)\) is experimental diffraction angle values.

### 3.1.4. Average strain \((\varepsilon)\)

The strain \((\varepsilon)\) is a macroscopic measure of deformation. It is calculated by [21]:

\[ \varepsilon = \frac{\beta \cos \theta}{4} \]  

### 3.1.5. Dislocation density \((\delta)\)

It is the amount of the defects in a crystal. The simple approach of Williamson and Smallman is used to evaluate it by: [22]

\[ \delta = \frac{1}{D^2} \]  

where \((D)\) is the average crystallize size.

### 3.1.6. Texture coefficient \((TC)\)

\((TC)\) of CdO thin films is calculated to quantify the preferential orientation of the films [23]

\[ TC(hkl) = \frac{l(hkl)/I(hkl)}{\Sigma l(hkl)/l(hkl)} N \]  

where \((l)\) is the measured intensity, \((I)\) is the (JCPDS) standard intensity, and \(N\) the reflection number.

\(TC > 1\) implies the preferentially oriented sample.

### 3.2. Optical study

#### 3.2.1. Absorption coefficient \((\alpha)\)

It is calculated for thin films using Lambert law [20]

\[ \alpha = 2.3026 \frac{A}{t} \]  

where \((A)\) is absorbance, \((t)\) is the film thickness.

#### 3.2.2. Energy band gap \((E_g)\)

Energy band gap \((E_g)\) of the films was calculated using Tauc equation [24]:

\[ a\nu = C(\nu - E_g)^n \]  

where \(\nu\) is frequency, \(E_g\) is the energy gap, \(C\) and \(n\) are constants.

\[ a = 2.3026 \frac{A}{t} \]  

where \((A)\) is absorbance, \((t)\) is the film thickness.
where \((hn)\) is the incident photon energy, \(C\) is a constant, \((E_g)\) is the band gap and \((n)\) is an index which can take values \(1/2, 2, 3/2,\) and \(3\) corresponding to allowed direct, allowed indirect, forbidden direct and forbidden indirect transitions respectively.

3.2.3. Particles size of nanoparticles
The particle sizes is calculated by Brus relation [25]

\[
E_g^* = E_g + \frac{\pi^2}{8R^2} \left( \frac{1}{m_e^n} + \frac{1}{m_h^n} \right) - \frac{1.8e^2}{\epsilon R}
\]

(11)

where \((E_g^*)\) is the size dependent band gap, \((E_g)\) is the energy band gap of the bulk sample, \(R\) is the particle size, \((m_e^n)\) and \((m_h^n)\) are the effective masses of electron and hole respectively \((0.21 m_e)\) for \((CdO)\), \((\epsilon)\) is the dielectric constant \((6.07)\) for \((CdO)\) [26].

4. Result and discussion

4.1. Thickness measurement
The film thickness \((t)\) of \((CdO)\) films was found by gravimetric weight difference method by [27]

\[
t = \frac{m}{\rho \times A}
\]

(12)

where \((m)\) is the mass of the deposited films in gram \((g)\), \((\rho)\) is the density of the deposited material in \((g/cm^3)\) and \(A\) is the area of the deposited film in \(cm^2\). Fig.1 shows the variation of the \((CdO)\) films thickness with deposition time. It is clear that the thickness increases with increasing the deposition time.

4.2. Structural study

![Figure 1. Variation of CdO films thickness with deposition time.](image-url)
Fig. 2 shows the XRD patterns of CdO thin films deposited at different time (12, 24, 36) h at room temperature and annealed at 623k for (2 h). Fig. 2 shows that the diffraction peaks for CdO films observed at $2\theta$=(32.91°, 38.313°, 55.2°, 65.858°, 69.211°) are linked to (111), (200), (220), (311), and (222) planes respectively. This indicates that CdO films are polycrystalline in nature with a cubic form as compared with the standard data of CdO (JCPDS file no.75-0592) [28] as shown in Table 1. P. Perumal et al. observed similar results [29]. XRD shows an increase in the crystallinity with increasing the deposition time. Also, as the deposition time increases, the diffraction peaks are slightly shifted in the direction of higher $2\theta$ values and its intensity increases. The lattice constant (a) of CdO films was estimated by Eq. 4, and the observed (d) spacing was calculated from Bragg’s condition Eq. 3. We found a good agreement between the estimated values in this study and the standard data of CdO (JCPDS file no.75-0592) [28] (see Table 1). The FWHM (β) have been evaluated for all diffraction peaks. The FWHM values decreases as the deposition time increases. The Origin software was used in this study for fitting. Texture coefficient (TC) was found to be in the range of (0.591-1.328) by Eq.8, and as the deposition time increases the (TC) slightly increases. The evaluation of the TC indicates the preferred orientation of the films along (111) plane has high crystallinity. Enhancing the preferred orientation is associated with increasing the number of the grains along this plane [30]. The calculated crystallite size (D) was in the range of (19.1- 41.06 nm) by Scherer’s formula Eq.5, and as the deposition time increases, the crystallite size increases as shown in Fig. 3. The average strain ($\varepsilon$) was found to be in the range of (0.00148 - 0.00641) by Eq.6, and as the deposition time increases, the average strain ($\varepsilon$) decreases. The oxygen vacancies and the interstitial atoms incorporated in the CdO lattice may be caused the strain [31]. Dislocation density ($\delta$) was found to be in the range of (0.00059 - 0.00274 nm$^{-2}$) by Eq.7, and as the deposition time increases, the dislocation density decreases.
Figure 2. The X-ray diffraction of CdO films.
Figure 3. Variation grain size with deposition time

Table 1. The experiment and standard values of some XRD parameters for CdO films

| Dep. time (hrs) | 2θ Observe (deg.) | 2θ Stand (deg.) | d Observe (nm) | d Stand (nm) | a Observe (nm) | a Stand (nm) | h k l | I% Observe | I% Stand |
|----------------|-------------------|-----------------|----------------|-------------|----------------|-------------|------|------------|----------|
| 12             | 32.91             | 33.019          | 0.2721         | 0.2710      | 0.4714         | 0.4698      | 111  | 100        | 100      |
|                | 38.313            | 38.321          | 0.2349         | 0.2347      | 0.4698         | 0.4694      | 200  | 80         | 84       |
| 24             | 55.2              | 55.299          | 0.1664         | 0.1659      | 0.4706         | 0.4694      | 220  | 38.5       | 45.2     |
|                | 65.858            | 65.935          | 0.1418         | 0.1415      | 0.4703         | 0.4694      | 311  | 28.5       |          |
|                | 69.211            | 69.271          | 0.1357         | 0.1355      | 0.4702         | 0.4694      | 222  | 8.1        | 12.2     |
|                | 33.02             | 33.019          | 0.2713         | 0.2710      | 0.4698         | 0.4694      | 111  | 100        | 100      |
|                | 38.35             | 38.321          | 0.2347         | 0.2347      | 0.4694         | 0.4694      | 200  | 75         | 84       |
| 36             | 55.36             | 55.299          | 0.165          | 0.165       | 0.4693         | 0.4694      | 220  | 35         | 45.2     |
|                | 66.01             | 65.935          | 0.1415         | 0.1415      | 0.4693         | 0.4694      | 311  | 13         | 28.5     |
|                | 69.358            | 69.271          | 0.1355         | 0.1355      | 0.4693         | 0.4694      | 222  | 9          | 12.2     |
|                | 33.221            | 33.019          | 0.2697         | 0.2710      | 0.4671         | 0.4694      | 111  | 100        | 100      |
|                | 38.38             | 38.321          | 0.2345         | 0.2347      | 0.4690         | 0.4694      | 200  | 79         | 84       |
| 42             | 55.35             | 55.299          | 0.1660         | 0.1659      | 0.4694         | 0.4694      | 220  | 32         | 45.2     |
|                | 66.2              | 65.935          | 0.1411         | 0.1415      | 0.4681         | 0.4694      | 311  | 17.1       | 28.5     |
|                | 69.5              | 69.271          | 0.1352         | 0.1355      | 0.4685         | 0.4694      | 222  | 6.3        | 12.2     |
4.3. Optical study

Fig.4 reveals that the absorbance spectra ($A$) of the prepared films increases gradually with increasing the deposition time which might be due to increase the grain size with increasing the deposition time that improves the crystallinity and increases the thickness of the films [29]. The optical absorbance of the films decreases with the wavelength. These spectra reveal that CdO films have less absorbance in the visible and high wavelength region and more absorbance in the UV region [32].

The absorption band of nanoparticles shows a blue shift due to the quantum confinement compared with bulk CdO particles. This optical phenomenon indicates that these nanoparticles show quantum size effect [33].

Fig.5 shows the absorption coefficient ($\alpha$) as a function of the wavelength calculated from Eq.9. It reveals that in the shorter wavelengths, the absorption coefficient ($\alpha$) exhibits high values which means that there is a large probability of the allowed direct transition. Then ($\alpha$) decreases with increasing the wavelength. The absorption coefficient ($\alpha$) values found to be of the order of $10^4$ to $10^5$ cm$^{-1}$. Fig.6 shows a direct band gap of CdO films estimated from extrapolation the plot of $(\alpha h\nu)^{2}$ versus ($h\nu$) Eq.10. The direct band gap energies are (3.026, 3.096, 3.117 and 3.409) eV, and it decreases with increasing the deposition time as seen in Fig.8. These energies are in good agreement with the earlier results reported in [29], and higher than the bulk (2.3 eV). The increase in the band gap of the bulk is due to increasing the free electron concentration in the films. The shift of the band gap with the change in the carrier concentration can be explained by the Burstein-Moss shift [6]. Fig.7 shows the typical Tauc $(\alpha h\nu)^{1/2}$ vs. ($h\nu$) plot for an indirect band gap energy determination. The indirect band gap energy obtained in this case were (2.197, 2.502, 2.539 and 2.917) eV, and it decreases with increasing the deposition time. This energy is higher than the bulk (1.36 eV). Fig.8 shows the variation of the particle size with deposition time. The particle size increases with increasing the deposition time. This confirms the same effect as in XRD study. The particle size of CdO nanoparticles was estimated from Eq.11 in the range of (11.419-12.895) nm for direct energy gap, and (10.374 -12.923) nm for indirect energy gap, Table 3. There is a slightly disagreement between the particle size calculated from the preferred orientation plans using Eq.5 and that calculated from Eq.11.

| Deposition time (hrs) | $2\theta$ (deg.) | $\beta$ (FWHM, rad.) | $\beta TC$ | Grain size (nm) | $\epsilon$ (average strain) | $\delta$ (dislocation density (nm$^{-2}$)) |
|----------------------|-----------------|---------------------|------------|----------------|--------------------------|---------------------------------|
| 12                   | 32.91           | 0.00757             | 1.209      | 19.1           | 0.00641                  | 0.00274                         |
| 38.313               | 38.31           | 0.00757             | 1.152      | 19.38          | 0.00545                  | 0.0066                          |
| 55.2                 | 65.858          | 0.00757             | 0.806      | 21.81          | 0.00292                  | 0.0021                          |
| 69.211               | 69.21           | 0.00757             | 0.803      | 22.25          | 0.00274                  | 0.00202                         |
| 33.02                | 33.02           | 0.00613             | 1.295      | 23.59          | 0.00517                  | 0.0018                          |
| 38.35                | 38.35           | 0.00613             | 1.156      | 23.95          | 0.0044                   | 0.00174                         |
| 55.361               | 55.361          | 0.00613             | 1.003      | 25.54          | 0.00292                  | 0.00153                         |
| 66.01                | 66.01           | 0.00613             | 0.591      | 26.97          | 0.00236                  | 0.00137                         |
| 69.358               | 69.358          | 0.00613             | 0.955      | 27.8           | 0.0022                   | 0.00123                         |
| 33.221               | 33.221          | 0.00411             | 1.328      | 35.7           | 0.00344                  | 0.0008                          |
| 38.38                | 38.38           | 0.00411             | 1.249      | 35.76          | 0.00295                  | 0.00078                         |
| 55.35                | 55.35           | 0.00411             | 0.940      | 38.13          | 0.00196                  | 0.00069                         |
| 66.2                 | 66.2            | 0.00411             | 0.797      | 40.31          | 0.00158                  | 0.00062                         |
| 69.5                 | 69.5            | 0.00411             | 0.686      | 41.06          | 0.00148                  | 0.00059                         |

Table 2. Experimental values of XRD parameters of CdO films
Fig. 8 shows the variation of the particle size with energy gap. The particle size decreases with increasing the energy gap.

**Figure 4.** UV-Vis Absorbance (A) spectra of CdO films.

**Figure 5.** Absorption coefficient (α) as a function of wavelength (λ) of CdO films.
Figure 6. A direct band gap of CdO films.

Figure 7. The indirect band gap of CdO films.
Figure 8. Vibration of the calculated grain size and direct energy gap of CdO films

Table 3. Experimental values of energy gap, blue shift and the calculated particles size for CdO films

| Deposition time (hrs.) | 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) |
|------------------------|-----------------------------|----------------------------|----------------------|-------------------------------|-----------------------------|----------------------|
| 12                     | 3.409                       | 1.109                      | 11.419               | 2.915                         | 1.555                       | 10.374               |
| 24                     | 3.117                       | 0.817                      | 11.823               | 2.539                         | 1.179                       | 11.532               |
| 36                     | 3.096                       | 0.796                      | 12.29                | 2.502                         | 1.142                       | 12.033               |
| 48                     | 3.026                       | 0.726                      | 12.895               | 2.197                         | 0.837                       | 12.923               |

5. Conclusions
CdO films have been successfully prepared with high quality, homogenous and grain size in the range of nanoparticles on glass substrates by CBD under different deposition time. The XRD tests of these films revealed that the deposited CdO thin films are polycrystalline in nature with cubic structure having (111) preferred orientation. The influence of the deposition time on the structural and optical properties is studied. By increasing the deposition time (thickness increases), the width of the diffraction peaks decreases resulted in increasing the grain size. We found that the average values of the grain size were in the range of (19.1-35.2 nm) for the preferred orientation indicating that the films are nanoparticles. The optical study revealed that the absorption spectra of CBD (CdO) films increased as the deposition time was increased. A blue shift in the optical gap has been observed for the absorption spectrum of the prepared CdO films, as an indication of quantum confinement effect. The absorption edge slightly shifts towards the higher wavelength with increasing deposition time. As the deposition time increases the energy gap decreases and the particle size increases.

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