



Effect of the Number of Dipping Cycles for a Cadmium Oxide Film Prepared by SILAR Method

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Abstract

The thin films of cadmium oxide (CdO) were deposited using the SILAR (Successive ionic layer absorption and reaction) method at various deposition cycles. CdO thin films were made on glass substrates at a temperature of 95°C, using a cadmium acetate source material and an ammonium hydroxide solution. One of the main criteria that impact the quality of thin films is the number of deposition cycles. The size of the crystals decreases with the increase in the number of cycles from 33.7 nm at the immersion cycle 10 to 22.7 nm at the immersion cycle 20, as shown by the X-ray diffraction results. The optical band gap energy of the films reduces as the number of deposition cycles increases, while the transmittance of the Cadmium oxide film increases as the dipping cycle increases. FTIR analytics of the sample showed that the transmittance increases with the immersion cycles. The thickness of the sample measured by the Michelson method increases with the increase in sedimentation.

Keywords: thin films, SILAR, X-ray diffraction

1. Introduction

In recent years, transparent conductive oxides have attracted many researchers because of their high transparency in the visible region of the electromagnetic spectrum [1]. Among these TCOs are the following Cadmium oxide (CdO) has been effectively employed for a variety of applications gas



sensors [2], photovoltaic photodiodes [3], and other uses. Due to its low resistivity and high conductivity, it can be used to make transparent electrodes, among other things [4]. Cadmium oxide (CdO) is a widely used substance for its electrical and optical properties. With a direct band gap of roughly 2.3 eV, CdO is an n-type semiconductor [5].

Several processes can be used to make CdO thin films, including sol-gel method [6], pulsed laser deposition [7], and chemical bath deposition [8]. Spray pyrolysis method [9], the growth of the solution method [10], and ionic layer adsorption and reaction in consecutive layers SILAR method [11].

In 2018, Chile prepared thin films of cadmium oxide (CdO) on Borosil glass substrates by successive ionic layer adsorption and a reaction method. The films were found to be amorphous. The thickness of the films increased with the sedimentation cycles and Focus Source Solutions. The band gap of the film dropped from 2.7 eV to 2.5 eV was found [12]. In 2017, Shamim et al. prepared Cadmium Oxide (CdO) thin films with different molar concentrations on a glass substrate with a simple and low-cost SILAR method (Successive Ionic Layer Adsorption and Reaction) [13].

A goal of this research is to see how the number of deposition cycles affects the structural, optical, and morphological properties of as-deposited films. The SILAR method is frequently referred to as a modified variant.

of chemical bath sedimentation. SILAR has several benefits, including low cost and low temperature [14]. SILAR is a two-step chemical bath deposition process. The SILAR method is used in this study to prepare CdO thin films for various dip cycles at pH = 7.2.

2. Experimental

The steps for making cadmium oxide film are shown in the methods below:

- i) Glass substrates are washed with chromic acid and rinsed with distilled water for 15 minutes in an ultrasonic bath.
- ii) Cadmium acetate $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$, this material, which is a colorless, soluble in water substance with a molecular weight (266.52) g / mol (99 percent e-Merck) at the molar concentration (0.1 mol / l), where a certain weight of Cadmium acetate is gradually dissolved in (100) ml distilled water using a magnetic mixer for (two) hours. Finally, a clear, homogenous solution was obtained; it is worth noting that the liquid solutions are prepared using the relationship below [15].

$$M = \frac{Wt}{Mwt} \times 1000 / V \quad (1)$$

Where: M: molar concentration.

Wt: The weight required to melt.

V: Volume of distilled water.

Mwt : molar weight of matter.

Then, with a pH of 7.2, gradually add ammonium hydroxide with a molecular weight of 17.03 g/mol (manufactured in Belgium), and precipitate at a bath temperature of 90°C under optimum conditions.

The glass substrates are immersed in Baker one for 30 seconds before being immersed in Baker two containing hot water at 100 °C. The duration of the immersion cycle of the glass substrate in the cadmium acetate solution is 30 seconds and in the water bath is 15 seconds. Finally, the glass substrate is prepared in this way for the purpose of annealing for an hour until it turns a dark yellow color; this represents the cadmium oxide film.

3. Results and Discussion

The produced CdO thin films were characterized using a variety of analytical techniques, including an X-ray diffraction (XRD), scanning electron microscopy (SEM), and atomic force microscopy (AFM)

a) The Film Thickness

A Helium-neon laser (He-Ne) with a continuous optical power of 1 mW is used to determine the thickness of the prepared films, using equation 2.

Δx : width for Which was fringe dark, X: width for Bright fringe

$$t = \lambda/2 \Delta x/x \quad (2)$$

Where, $\lambda = 632\text{nm}$, the wavelength of the Helium-Neon laser, and The measured thickness = 118 nm, 136 nm.

b) X-Ray Diffraction

The intensity increases with the increase in the number of sedimentation cycles, which shows a clear improvement in crystallization. The Scherrer relationship is used to estimate the Crystal size [16]:

$$D = \kappa\lambda/(\beta \cos\theta) \quad (3)$$

Where κ : is a constant of 0.94, and λ is the wavelength of $\text{CuK}\alpha$ line, and equal to 1.54 nm, θ : is the Bragg's angle, and β is full width half maximum (FWHM) of the preferential plane. The structural parameters of CdO thin films are given in Table 1. The crystallite size is calculated using Scherrer's formula. Figure 1 shows typical XRD patterns for films containing On different immersion cycles. The diffraction peaks can be indexed to cubic CdO phases (JCPDS card number: 05-0640 for CdO). The highest intensity is at (111), then the patterns begin to shift downward.

Table 1. The structural parameters of CdO thin films with different dipping cycles number.

NO.of. dipping	$(\beta \times 10^{-3})$ Rad	Bragg's angle(2θ)	Miller indices(hkl)	Crystallite Size (nm)
Dip10	7.27	32.88	(111)	33.7
	7.26	38.18	(200)	21.2
Dip20	7.97	55.08	(220)	20.6
	6.73	32.84	(111)	22.7
	7.29	38.13	(200)	21.1
	8	55.07	(2200)	20

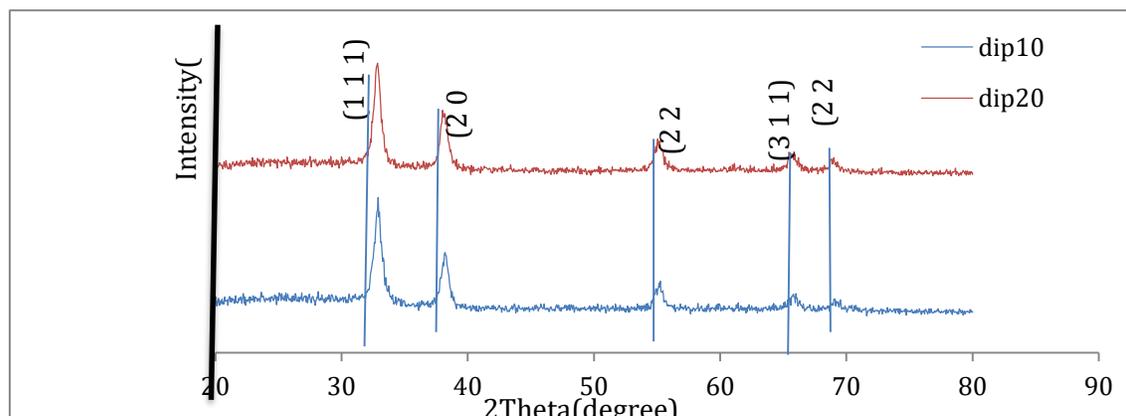
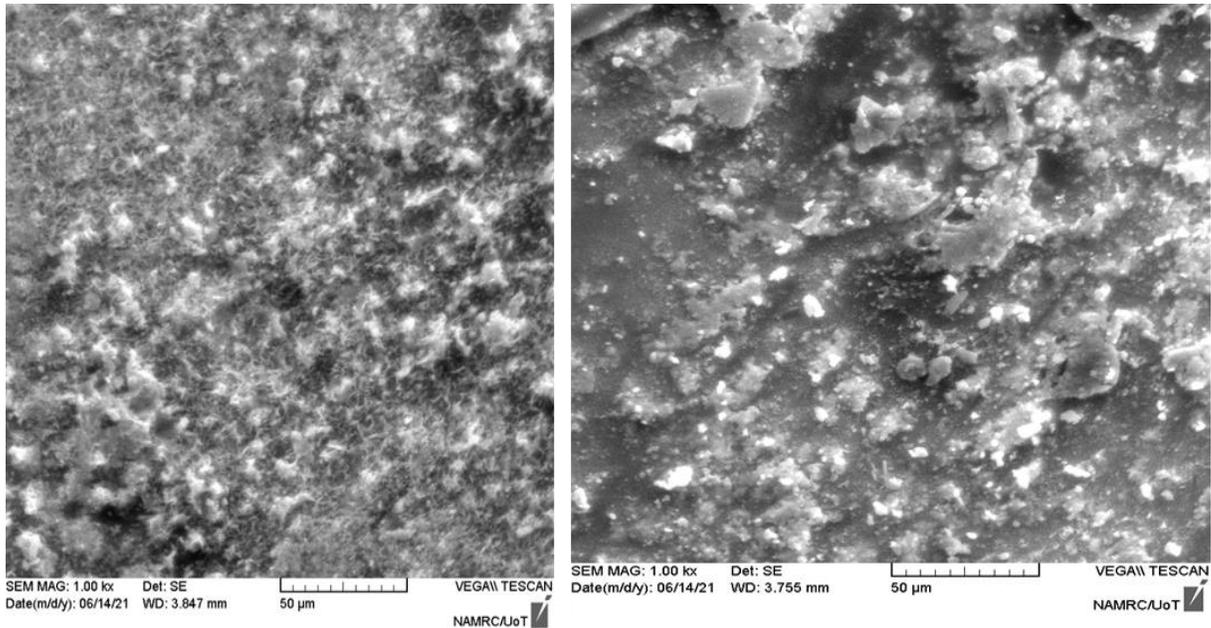


Figure 1. XRD patterns of CdO thin films at 0.1 M, PH=7.2 for different dipping cycles number

C) Scanning Electron Microscopy of CdO

Thin Films in Figure 2 (a, b) show the SEM micrograph of the surface topography of the CdO films. Figure 2 (a) shows the SEM image with 10 dipping cycles. It also shows the collimation of particles, indicating a large particle size, while Figure 2 (b) has spaced particles with small particle sizes. As the number of deposition cycles increased, the grain size shrank. All SEM images appear homogeneous and dense surfaces. Surface shape and grain size. The CdO films are largely changed as a function of changing the number of immersion cycles. The change in particle size is indicative of decreased total particle boundary in films.



a: (dip10)

b:(dip20)

Figure 2 (a,b). Shows the Scanning Electron Microscopy taken in a 10,20 dipping for a concentration of 0.1M at pH 7.2

d. Atomic Force Microscopy (AFM)

Figures (3, 4) show the atomic force micrographs in two and three dimensions of the pure Cadmium oxide with a different number of dipping. Table 2 shows the surface roughness and the root means square of the roughness rate of pure Cadmium oxide films.

Table (2). Average diameter size, Roughness, and Root Mean Square of pure CdO membrane

Average Diameter (nm)	surface Roughness(nm)	Root Mean Square(nm)	No.of.dipping
76.5 nm	4.92	5.8	Dip10
82.4 nm	4.74	5.4	Dip20

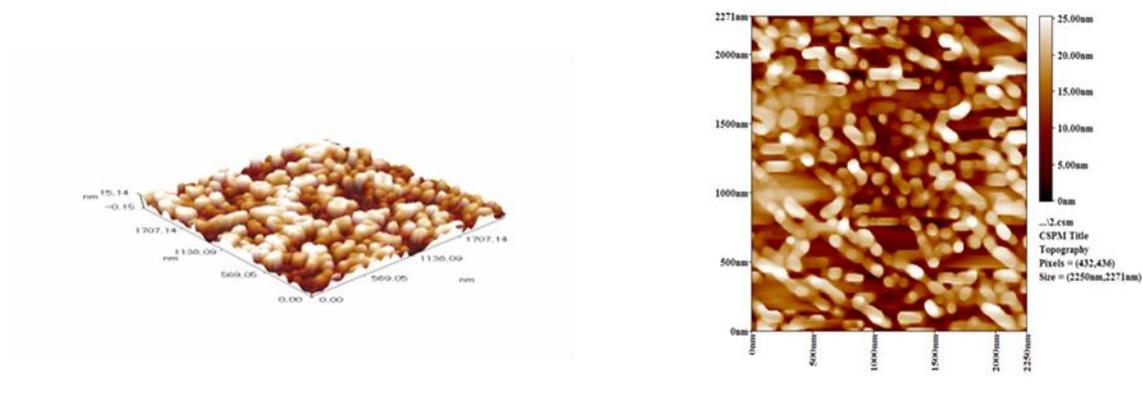


Figure3.AFM of CdO thin films at Dip=10 for a concentration of 0.1M at pH 7.2



Figure4.AFM of CdO thin films at Dip=20 for a concentration of 0.1M at pH 7.2.

4. Optical properties

Under ambient conditions, the optical properties of CdO thin films are investigated, using a single-beam UV-Vis spectrophotometer in the wavelength range from (300-900) nm. The transmittance spectra of CdO thin films deposited for different immersion times are shown in Figure 6. The plot indicates a dramatic increase in transmittance near the band edge, related to the good crystallinity [12] Increasing the number of deposition cycles shows a narrow range of variation It is a useful material for optical coatings because of its high transmittance. Using Tauc's plot, the optical energy band gaps of the films are determined from the transmittance spectra. The equation is used to compute the absorption coefficient (4) [17]: $\alpha = \ln (1/T) / d$ (4)

Where d is the coating thickness and T : is the transmittance. The following formula indicates the relation between the absorption coefficient (α) and the incident photon energy ($h\nu$):

$$(\alpha h\nu)^2 = (h\nu - E_g) \quad (5)$$

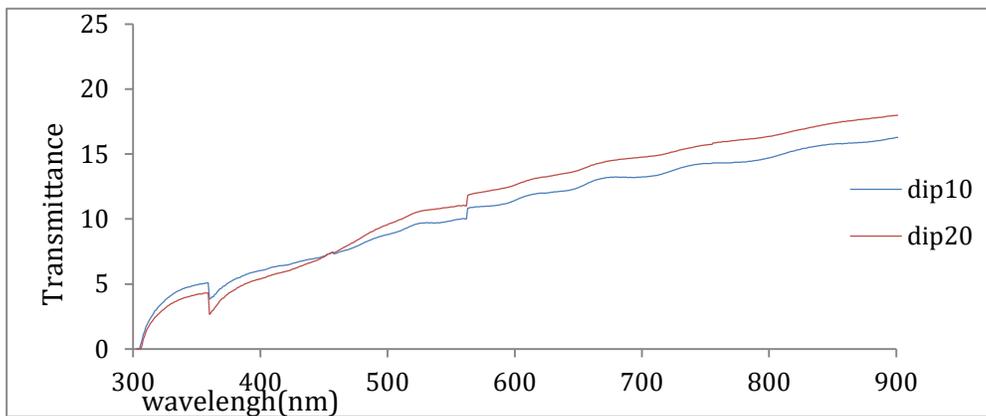


Figure 5. The transmittance of CdO thin films at different number of deposition cycle

Figure 6 shows the spectral data of CdO produced at 90°C with a varied number of deposition cycles as a function of the wavelength range (300-900) nm with glasses as the reference (6). It depicts the optical absorbance values, indicating that the film's absorbance decreases with increasing wavelength. In the visible regions, all the films deposited at the various deposition cycles had good transparency, with little or no change in optical transparency.

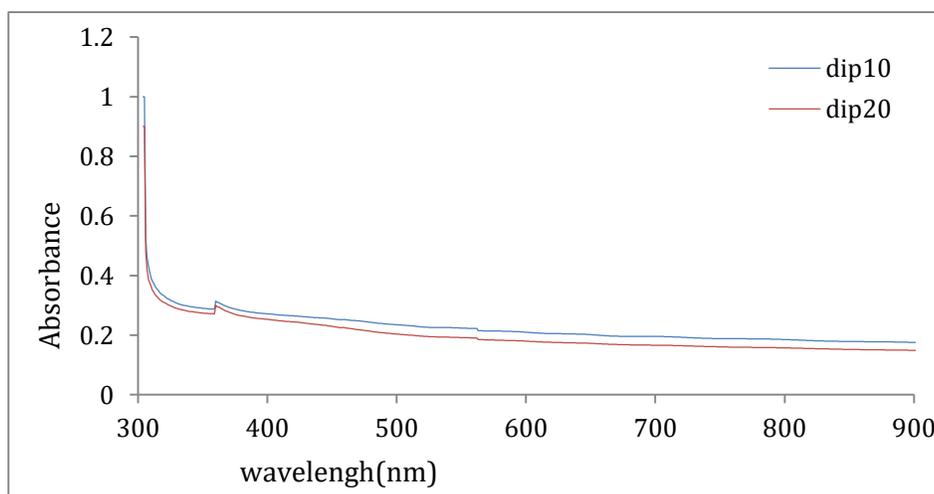


Figure 6. The absorbance of CdO thin films at different number of deposition cycle

Figure 7 clearly shows that increasing the number of dipping cycles does not influence on the band edge in the visible region. This means that the fundamental crystal structure remains unchanged. Figure 7 (a,b) shows a typical plot of $(h\nu)^2$ against h for CdO thin films produced

on a glass substrate with a number of dipping cycles. It is observed that an increase in dipping cycles of CdO precursor yields a slight decrease in optical bandgap from 2.3eV to 2.15eV.

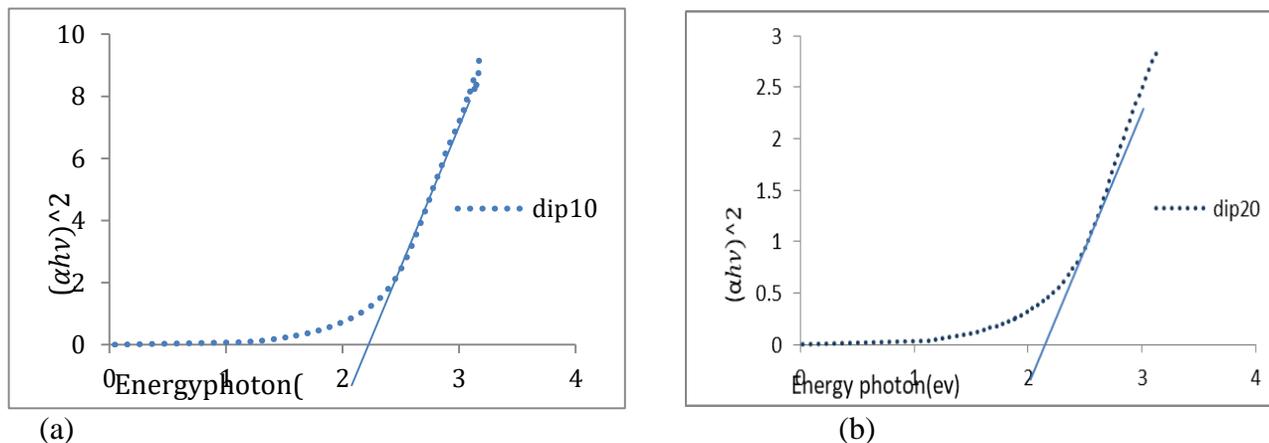
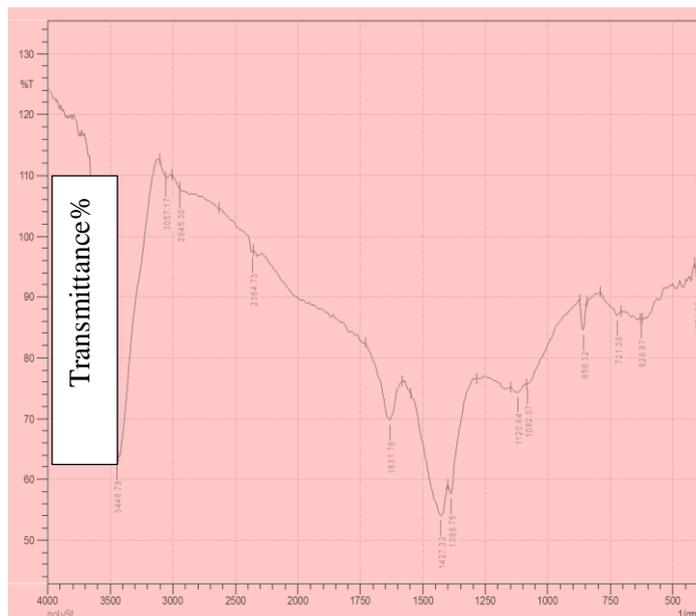


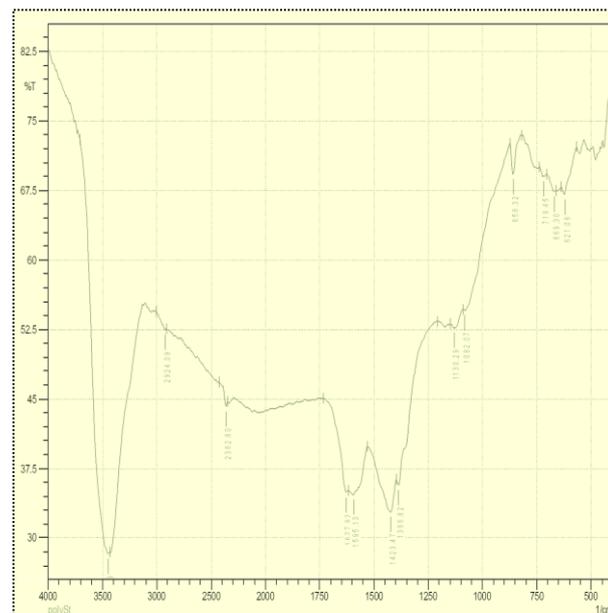
Figure7 (a,b). CdO sample The optical gap energy for different dipping cycles

5. FTIR spectroscopy

The goal of FTIR spectroscopy is to investigate multi-component functional groups to gain insight into the mechanism of contact and determine the composition of the material phase in a variety of bond types seen in all samples. Figure (8a, b) shows the FTIR spectra of the pure cadmium oxide film at different immersion cycles, and the spectra of their nanocomposites, displaying the properties of different vibration bands for stretching and bending and, shifts at some band sites and intensity changes at others. Peaks at: 3377.61 cm^{-1} correspond to OH stretching; 3057.17 cm^{-1} (dip10) and 2924.09 cm^{-1} (dip20) corresponds to C-H stretching; 1631.78 cm^{-1} (dip10) and 1627.92 cm^{-1} corresponds to the characteristic vibration of C = N 1427.32 cm^{-1} (dip10) and 1423.47 cm^{-1} represent the characteristic vibration of C = N and C-C stretching and/or CH₂ deformation, respectively. The OH stretching vibration of the hydroxyl groups causes a large peak at 3446.79 cm^{-1} (dip10) and 3446.79 cm^{-1} (dip20). In addition, the peak at 1654.92 cm^{-1} has dwindled in intensity and broadened in scope.



(a)



(b)

Figure 8(a,b). FTIR of the Cadmium oxide film at different immersion cycles for a concentration of 0.1M at pH 7.2

6. Conclusion

In this study, using a simple chemical process known as the Chain Layer Ion Absorption Reaction (SILAR), polycrystalline CdO films are produced. The sample is void of voids according to scanning electron microscope images. XRD analysis revealed that the cubic polycrystalline phase of the CdO is formed on a glass substrate at 90°C. AFM micrographs of the nanostructure film reveal that the thin film is homogeneous upon changing the immersion cycle, the optical band gap of the CdO film is changed from 2.3 eV to 2.15 eV, and transmittance values for films are changed with the increase in the number of sedimentation cycle.

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