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Study the impact of different annealing temperatures on synthetic Title: properties, surface topography, and optical properties of compounds (50% ZnO: 50% CdO) prepared by chemical deposition method

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ABSTRACT

Studying on the structural properties, surface topography of compounds (ZnO 50%) and (CdO 50%) nanoparticles prepared by the method of chemical deposition, Before and after annealing at (600°C) . Thin films with the same weight ratios were also prepared and deposited by the method of rotational coating and indicating them at different temperatures and the (600°C, 400°C, 200°C) extent of their impact on On optical properties. The results of the X-ray diffraction showed (XRD) that all the results are of hexagonal crystal structure, and the topography of the surface was studied with the scanning electron microscope technique (SEM) and the impact of the annealing time on the loss of atomic weights of the chemical elements used in preparing the samples according to the technique (EDAX), or from the visual results, the value of the energy gap appeared if it appeared (3.614 eV) before annealing, but its value decreases with the increase in the annealing temperature, from which it changed to (3.551 eV3.349 eV), and (3.40 eV) with the change in the annealing temperature from (200°C, 400°C, 600°C) respectively. This research aims to develop new materials with improved properties by controlling annealing processes, which enhances their use in many areas of advanced technology.

1. Introduction

Metal oxides are semiconductor nanomaterials of great importance owing to their size-dependent properties and their wide applications in materials science, engineering, chemistry, and electricity during the past years [1] Thin films of these oxides are also used as sensors for the reactions of chemicals and gases, which are the result of the union of divalent chemical elements with oxygen to obtain them, such as ZnO and CdO [2], these oxides are characterized by an optical energy gap ranging from (4 eV- 3 eV), they have received much attention from physicists and chemists, and can be prepared by various methods, including sol-gel, thermal evaporation, pulsed laser deposition, and chemical precipitation, and because of the ease of manufacture and low cost of the chemical precipitation method, which prompted the researchers Amanpal Singh and Amanpal Singh [4] and researchers (Waleed E. Mahmoud) [5] studied the effect of annealing on the structural and optical properties of composites with the chemical formula (ZnO-CdO); the results showed that annealing improves the structural and surface topography properties of these materials. Thin films of nanocomposites of these oxides were also synthesized by the spin coating method because this technique has many advantages, including the ease of obtaining the necessary equipment for the coating process, and with small amounts of solution covering the entire surface of the sample of the liquid to be coated, and more than one layer and different solutions can be deposited on the same base to study their optical properties [6], and to study its structural properties represented by the lattice constant using the X-ray diffraction technique in the range (2θ =20-80), and the granular size as well as the stacking in the crystal system and to know the chemical elements used in the preparation of the samples under study and the effect of the manufacturing stages on their mass weight was achieved by SEM and EDXA technique. The aim of the study is to study the structural and optical properties and determine the optical constants of (ZnO:CdO) nano-semiconductors prepared by chemical deposition method.

2. Methodology

The Zn-Cd-O solution was added in the form of drops using a burette to the sodium hydroxide solution with continuous stirring for half an hour at a temperature of (60 ℃). Then we obtain a clear solution that is filtered through filter paper and washed several times with distilled water to get rid of insoluble nitrates and dried in a drying oven to (100 ℃) to get rid of moisture and add a binder of polyvinyl alcohol (PVA) and then equal proportions of the two materials were taken by a sensitive balance and put the powder in molds (Stainless-(steal) with a diameter (10 mm) and a sample thickness of (2 mm) and then these models were sintered at annealing temperature (200 ℃), (400 ℃) and (600 ℃) for two hours respectively to obtain more solid models and then practical measurements were carried out. The structure of the films was determined by X-ray diffraction measurements with 40 kV, 20 mA (XRD, Bruker/D8-advance with Cu-Kα radiation (λ= 1.54178 Å), Iran), in the scan range of 2θ between 20˚-80˚. The surface morphology of the ZnO films was investigated by scanning electron microscopy (SEM) (FESEM, MIRA3 LMU, Iran). Then the Prepared model surfaces were characterized by using the energy dispersive X-ray analysis (EDXA, HORIBA EMAX Energy EX-950, Iran).

To study the optical properties, thin films were prepared on glass substrates with dimensions of (2 × 2 cm2) by spin coating at the same temperature and annealing time of half an hour Checked by (T92 +uv Spectrophotometer PG INSTRUMENTS). With a variable spectral range of (0.1-5)nm. The devices mentioned above were used due to their great importance in providing accurate information for examining and studying the structural and optical properties of the composites.

In order to study the structural properties at annealing temperature, X-ray diffraction (XRD) pattern of the samples before and after annealing at annealing temperature (200℃, 600 ℃) was done by calculating the interfacial distance between their crystal planes using Brack's law (1) [7].

$$2\lambda = 2d_{hkl}\ sin\ \theta \ldots\ldots (1)$$

Where:

n is an integer representing the order of reflection.

λ represents the wavelength of the incident X-rays and has a value of (1.54060)

θ is the angle of incidence of the X-rays or Brack's angle.

d_{hkl}: The interfacial distance between two neighboring crystal planes.

The lattice constants were also calculated for all

samples according to the crystal system they belong to, according to the following mathematical relationships (2) and (3), respectively [8].

$$\frac{1}{d^2} = \frac{4}{3}\left(\frac{h^2+hk+k^2}{a^2}\right)\frac{l^2}{c^2} \dots\dots(2)$$

$$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \dots\dots(3)$$

(hkl): Miller Indices

The average crystalline size of the granules was also calculated using the Debye-Scherrer method according to the relationship (4) [9].

$$D_{av} = \frac{k\lambda}{\beta co\, s\, \theta} \dots\dots(4)$$

k: The molding constant with a value of 0.9

λ: wavelength of the ray's incident on the sample.

Brack's diffraction angle

β represents the average of the greatest absorption intensity in radial units.

To study the surface morphology, two-dimensional SEM images were taken, through which the crystal size was calculated, and the proportions of the materials used in the preparation of the samples were determined by EDXA.

The optical properties, represented by the energy gap of the direct transmission allowed for all the models, were calculated using the relationship (5) [10].

$$\alpha h\nu = A\left(h\nu - E_g\right)^n \dots\dots(5)$$

Where:

$h = 6.626 \times 10^{-34}\ \frac{m^2\, kg}{s}$ Planck's constant

ν: Photon of incident light,

A is a constant that depends on the mass of the charge carriers.

n: depends on the nature of the dominant electron transitions.

The absorbance of the material was also calculated and the absorption coefficient of the samples under study was determined.

The relationship (6) [11]

$$\alpha = 2.3026\frac{A}{t} \dots\dots(6)$$

A: the absorption spectrum

t: is the thickness of the model

α: Absorption coefficient

The damping coefficient can be determined according to the relationship (7) [12]

$$k^{\circ} = \frac{\alpha\lambda}{4\pi} \dots\dots(7)$$

3. Results and Discussion

3.1 Structural Characterization

Figure (1) shows the X-ray diffraction of the models (ZnO:CdO) and the weight ratio (50%) before and after heat treatment at an annealing temperature (200℃,600), as it appears from the previous figure that an increase in the intensity of X-ray diffraction when treating the models at 600 ℃ indicates the crystallization of the models as a result of the transition of atoms to their correct locations, and this is consistent with what the researcher stated [13] In addition, it is clear from the previous figure how well our results match the results of the international X-ray diffraction card shown in Table (1); the crystalline phase was determined by calculating the lattice constant for all samples shown in Table (2), which shows that all samples have a compact hexagonal crystal structure, which is consistent with the card number (JCPDF 96-900-8610). As for the SEM images of the models under study before and after heat treatment, as shown in Figure (2), the images showed crystalline shapes in the form of nanorods in addition to semi-spherical shapes interspersed with gaps that are almost absent when the models are treated with annealing temperatures of 600 ℃ with an increase in the size of nanorods and nanobodies with the appearance of dendritic shapes in the form of wires, and very fine nanorods begin to appear as the models under study appear smooth after annealing and that the average grain size and particle diameters of the mixture increase after annealing, which is consistent with the results (XRD) is consistent with the sources [8, 18]. Figure (3) refers to the EDX results, which show the chemical elements included in these samples with their weight ratio, indicating that the annealing temperature has no effect on the loss of the ratio of all elements before annealing.

Table 1. The $dhkl$ values of the peaks in the X-ray patterns of the prepared samples with dhkl. values according to the international standard card.

Sample	$2\theta\ (Deg.)$	$d_{hkl}\ Exp.(\text{Å})$	$D\ (nm)$	hkl	ZnO Lattice parameter		CdO Lattice parameter a
					c	a	
Before	36.43	2.46	18.0	5.24	3.22	101	4.69
200℃	36.17	2.48	27.9	5.19	3.26	101	4.70
600℃	36.52	2.45	31.4	5.18	3.22	101	4.65

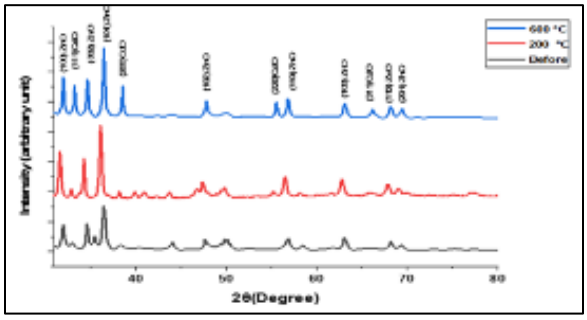


Figure 1: X-ray diffraction patterns of ZnO:CdO samples before and after annealing to 600 ℃

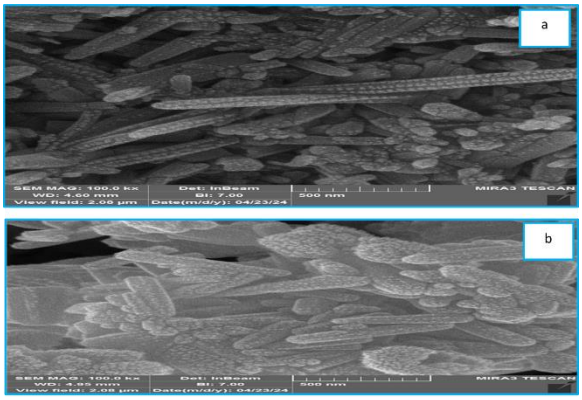
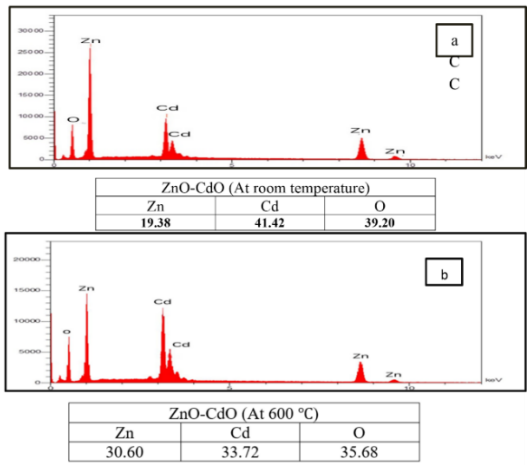


Figure 2: Scanning electron microscope images of the samples a- before b- annealing at 600 ℃.

The EDXA analysis of the ZnO-CdO mixture (50%) shown in Figure 3 shows the presence of the elements of the mixture. Figure 3a shows the presence of the elements before heating, while Figure 3b indicates the presence of the elements at an annealing temperature of 600℃ and according to the weight ratios of the chemical elements.



ZnO-CdO (At room temperature)		
Zn	Cd	O
19.38	41.42	39.20

ZnO-CdO (At 600 ℃)		
Zn	Cd	O
30.60	33.72	35.68

Figure 3: EDXA of ZnO:CdO (a) at Room Temperature, (b) at 600 ℃

3.2 Optical Properties

Figure (4) shows the absorption spectrum as a function of wavelength at different annealing temperatures of 600 ℃, 400 ℃ and 200 ℃, 200 ℃, respectively, for all samples. It appears from the previous figure that the values of the absorption spectrum as a function of wavelength are very small and almost constant, while the values of the absorbance spectrum increase with increasing annealing temperature, reaching its peak (1%) while increasing at the wavelength, which indicates the presence of a large variation in particle size, which is consistent with the interpretation of the researchers [6, 19, 20]. Figure (5) shows the absorption coefficient of the complexes as a function of wavelength for the models before and after annealing. It was found that the absorption coefficient increased after annealing in varying proportions, which may be due to the change in the crystal structure of zinc oxide, as all models showed, and this is due to the type of electronic transition that occurs [21, 22]. Figure (6) shows the relationship between the quenching coefficient and wavelength, as it appears that the quenching coefficient clearly increases after annealing and increases with increasing wavelength, which is consistent with what the researchers stated [22]. While Figure (7) shows the relationship between (αhv)^2 and the energy of the incident photons (hv) and using the relationship (5), the optical behavior of the samples under study was studied by drawing a tangent to the straight part of the photon energy is located at the point $(\alpha h\nu)^2 = 0$, since this intersection point represents the energy gap for direct electron transfer, as the results indicate that the energy gap before annealing $(3.614\ eV)$ and then decreases in a general way after annealing, reaching $(3.551\ eV)$ at 200 ℃ and $(3.349\ eV)$ at 400 ℃, as well as its value (3.40 eV) at 600 ℃, and the reason for the change in the values of the optical energy gap as a result of changing the annealing temperature may be attributed to the increase in the size of the crystals, which is consistent with the researchers' explanation [22, 23].

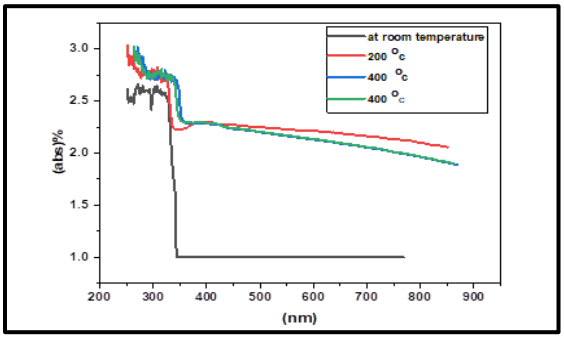


Figure 4: Absorption spectrum of samples before and after annealing 600℃, 400℃ and 200℃.

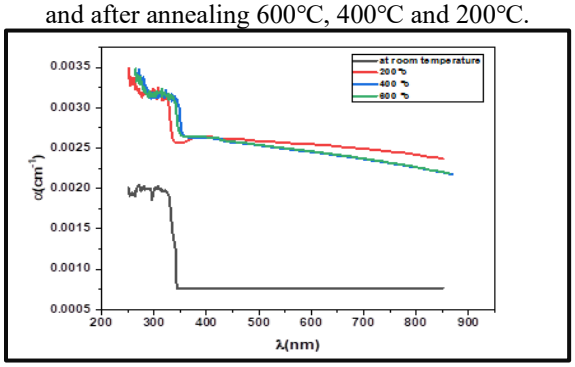


Figure 5: Absorption coefficient spectrum of samples before and after annealing 600℃, 400℃ and 200℃.

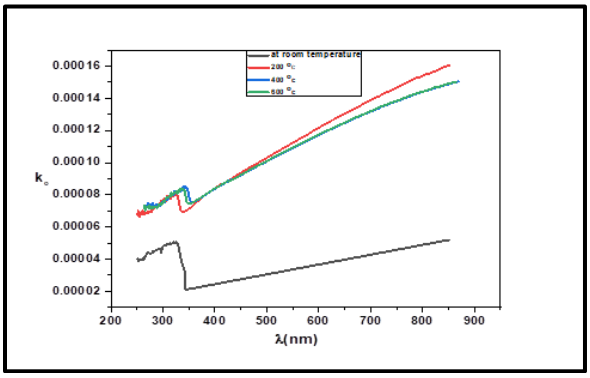


Figure 6: Inertness coefficient of samples before and after annealing 600℃, 400℃ and 200℃.

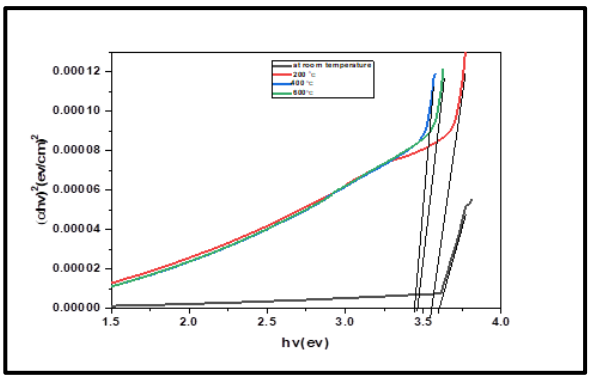


Figure 7: Optical power gap before and after annealing 600℃, 400℃ and 200℃ respectively.

4. Conclusions

ZnO:CdO nanocomposites were successfully prepared by a chemical precipitation method and sintered at different temperatures (600, 400, and 200 °C) for different applications. X-ray diffraction (XRD) results showed that the grain size increased with increasing annealing temperature, which is consistent with SEM. The optical properties showed a clear effect of annealing temperature, where the energy gap decreases with increasing temperature.

Recommendations and Suggestions: The devices mentioned above were used due to their great importance in providing accurate information for examining and studying the structural and optical properties of the composites. The effect of irradiation on the physical properties of composites and then comparing them with the results of annealing for different time periods.

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