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RESEARCH ARTICLE

EFFECT OF Co DOPING ON STRUCTURAL AND OPTICAL PROPERTIES OF ZnO THIN FILMS
PREPARED BY CHEMICAL SPRAY PYROLYSIS METHOD

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ABSTRACT

In this work, Zinc-cobalt oxide ($Zn_{(1-x)}Co_xO$) thin films, where $x = 0, 4, 6$ and 8% have been successfully deposited on glass substrates by chemical spray pyrolysis (CSP) technique at substrate temperature of $400\text{ }^\circ\text{C}$ and thickness of about 300 nm . The structural and optical properties of these films have been studied using XRD, AFM, and UV-Visible spectroscopy. The XRD results showed that all films are polycrystalline in nature with Hexagonal structure and preferred orientation along (111),(002),(101),(102),(110) and (103) planes. The crystallite size was calculated using Scherrer's formula and it is found that the undoped ZnO sample has maximum crystallite size (23.1 nm). AFM results showed homogenous and smooth thin films. The absorbance and transmittance spectra have been recorded in the wavelength range of $(300-900)\text{ nm}$ in order to study the optical properties. The optical energy gap for allowed direct electronic transition was calculated using Tauc equation. It is found that the band gap increases as the Co concentration increases and the band gap values were in the range of $3.27-3.32\text{ eV}$. The optical constants including (absorption coefficient, real and imaginary parts of dielectric constant) were also calculated as a function of photon energy. Refractive index and extinction coefficient for Zinc-Cobalt Oxide thin films were estimated as a function of wavelength.

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INTRODUCTION

ZnO is a wide band gap ($\sim 3.37\text{ eV}$) material that has potential applications in the fabrication of devices such as ultraviolet (UV) light-emitters, varistors, transparent high-power electronics, piezoelectric transducers, gas-sensors, smart windows and solar cells (Raidou et al., 2010). It is one of the materials of the II-VI family of Semiconductors which have been studied extensively for many years (Vijayan et al., 2008). Transparent conducting oxides (TCOs) thin films have been extensively studied because of their variety of applications keeping in view the need of low-cost TCOs that are required in various fields of science and technology (Shinde et al., 2008). Transparent conducting ZnO thin films are emerging as the most attractive alternative to ITO and various other TCOs (Tsang et al., 2008). ZnO is a very interesting material for many different applications in both microelectronic and optoelectronic devices (Ashour et al., 2009). It is a wide-band gap oxide semiconductor with a direct energy gap of about 3.37 eV (Wei Lin et al., 2007), with high optical transparency in the visible and near-infrared region of electromagnetic spectrum, and has a high refractive index (1.9).

Due to these properties, ZnO is a promising material for solar cell applications, such as antireflection coatings (López et al., 2008). ZnO thin films can also be used for gas sensing because of high sensitivity to many gases (Kaid and Ashour, 2007). Dilute magnetic semiconductors (DMS) produced by doping transition metal (TM) ions into non-magnetic semiconductors have attracted a great deal of interest (Fitzgerald et al., 2005). Comparing with other TM (e.g., Fe, Ni, V, Cr, Mn) doped ZnO, the identical viewpoint is that the ZnCoO system is a promising material because of the high solubility and the excellent ferromagnetic near room temperature (Ling Wei et al., 2009). ZnO thin films have been deposited by several techniques such as sputtering (Jeng-Lin Chung et al., 2008), pulsed laser (Ngoma et al., 2009), sole-gel (Yaodong Liu et al., 2006), and spray pyrolysis (Young-Sung Kim and Weon-Pil Tai, 2007). In contrast, the spray pyrolysis technique has been only rarely used although this process presents many advantages: (i) it is a low-cost and simple technique, and (ii) it allows the possibility of obtaining films with a large area (Belghazi et al., 2009). In this work, we report the growth of $Zn_{1-x}Co_xO$ thin films ($x=0, 4, 6, 8\%$) on glass substrates by spray pyrolysis technique and the influence of cobalt doping on the structural and optical properties of ZnO thin films. Cobalt (Co) lies with the transition metals on the periodic table. The atomic number of Cobalt is 27 with atomic mass of 58.933195 g/mol (Saleh, 2013). Cobalt is a sturdy, gray metal which resembles iron and Zinc.

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MATERIALS AND METHODS

ZnO and CZO (Co doped ZnO) thin films were deposited using the spray pyrolysis technique on glass substrates at temperature of (400 °C). A homogeneous solution was prepared by dissolving zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$) and cobalt acetate $\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ separately in distilled water at the concentration of 0.1 M. Final solutions were prepared by mixing the two initial solutions in appropriate volumetric proportions to get various concentrations of Co (0, 4, 6, 8) wt. %. The resultant solution was sprayed on glass substrates. The glass substrates were cleaned in acetone, rinsed in distilled water, and subsequently dried before deposition. The substrates are then placed on hot plate which was heated before progressively until the deposition temperature is reached. All films were deposited at 400 °C. Other deposition conditions such as spray nozzle to substrate distance (30 cm), spray time (10 s), spray interval (2 min) and pressure of the carrier gas (1.5 bar) were kept constant for each concentration. The X-ray diffraction patterns for the prepared films were obtained in a (Shimadzu XRD-6000) goniometer using copper target ($\text{CuK}\alpha$, 1.5418 Å) and Atomic Force Microscopy (AFM) micrographs were recorded by using scanning probe microscope type (SPM- AA3000), contact mode, supplied by Angstrom Advanced Inc. Optical properties in the wavelength range of (300-900) nm were investigated by using UV-VIS-NIR spectroscopy (Shimadzu, UV-1800). This spectrophotometer was used to measure the relative transmittance and absorbance of as-deposited ZnO and Cobalt doped ZnO thin films.

RESULTS AND DISCUSSION

Figure (1) shows the photo image of Zinc-Cobalt Oxide ($\text{Zn}_{1-x}\text{Co}_x\text{O}$) thin films, where $x = 0, 4, 6$ and 8% . It is reported that the stoichiometrically correct ZnO thin films are expected to have white color; however, the Co doped ZnO thin film deposited in the present study has light green color which can be attributed to d-d transitions of tetrahedrally coordinate Co^{2+} (Dinia *et al.*, 2005).

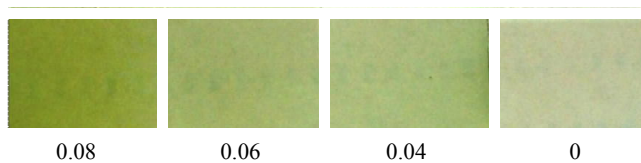


Figure 1. Photo images of ($\text{Zn}_{1-x}\text{Co}_x\text{O}$) thin films, where $x = 0, 4, 6$ and 8%

Structural analysis

The crystallite size was calculated by using the Scherrer's formula, (Barret and Massalki, 1980).

$$D_{av} = \frac{K\lambda}{\beta \cos \theta_B} \quad \dots \dots (1)$$

Where, D_{av} is the crystallite size, K is a constant known as Shape factor (= 0.94), β is the Full width at half maximum (FWHM) given in radians and θ_B is the Bragg's angle. The interplaner spacingd was calculated by using the Bragg's relation (Elliot, 1983).

$$n\lambda = 2d\sin\theta \quad \dots \dots (2)$$

And, for the calculation of lattice parameter, the following formula was used (Elliot, 1983):

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

In order to study the effect of the Co concentration on the structural properties of $\text{Zn}_{1-x}\text{Co}_x\text{O}$ thin films, various dopants levels were used to obtain x values of (0, 4, 6 and 8) %. The XRD patterns of the Codoped ZnO films are shown in Figure (2). As can be seen, the observed peaks are located at (100), (002), (101), (102), (110) and (103) favorite directions respectively which is in agreement with the Joint Committee of Powder Diffraction Standards (JCPDS) card number 36-1451. The diffraction patterns reveal a good crystalline behavior without any appreciable changes from pure ZnO films and are genuinely polycrystalline with the hexagonal wurtzite structure. This indicates that Co doping did not change the crystal structure of the ZnO samples which implies that there were no secondary phases such as Co clusters or oxides. It may be due to very small amount of Co doping which was not enough for changing the crystal structure significantly. This behavior shows good agreement with the study done by Li *et al.* (2007). It can be noticed that 2θ for (002) direction decreases with increasing the Co concentration as shown in Table (1) expect for the sample where $x=0.08$ which has (100) favorite direction. It can be noticed also that the lattice constant (a_o) increases as the Co concentration increases but (c_o) decreases. It should be mentioned here that the standard a_o value for ZnO is 3.249 Å and the standard c_o value is 5.206 Å.

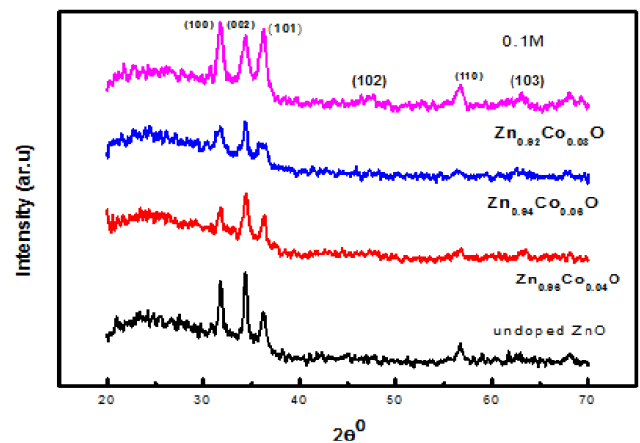


Figure 2. XRD patterns of Co doped ZnO thin films

The crystallite size decreases with increasing Co concentration expect for the sample where $x=0.08$ which has maximum crystalline size of 61.20nm. The values of the other structural parameters are shown in Table (1).

The texture coefficient (T_c) represents the texture of a particular plane, in which greater than unity values imply that there are numerous of grains in that particular direction. The texture coefficients $T_c(hkl)$ for all samples have been

calculated from the X-ray data using the formula (Pankove, 1971):

$$T_{c(hkl)} = \frac{I_{(hkl)}/I_{o(hkl)}}{\frac{1}{M} \sum I_{(hkl)}/I_{o(hkl)}} \dots\dots (4)$$

Table 1. Structural parameters of Co-doped Zinc Oxide thin films

Cobalt concentration	0	0.04	0.06	0.08
2 θ (deg)	34.3785	34.4284	34.3585	31.7337
hkl	(002)	(002)	(002)	(100)
d (Å)	2.60651	2.60285	2.60799	2.81746
(FWHM) (rad)	0.00628	0.0066	0.0069	0.0023
(Dav) nm Scherrer	23.11	21.99	21.03	61.20
Micro strain S	-0.12	-0.24	-0.26	-0.20
Lattice Constants a _o (Å)	3.2497	3.2476	3.2426	3.2534
Lattice Constants c _o (Å)	5.2130	5.2057	5.2159	5.1852
$c = c/a$	1.604	1.602	1.608	1.593
Tc	2.01	2.02	2.14	1.13

Where $I(hkl)$ is the measured intensity, $I_o(hkl)$ taken from the JCPDS data, M is the reflection number and hkl are Miller indices. The texture coefficient is calculated for crystal plane (002) and (100) of the undoped and Co doped ZnO films. All values of texture coefficient were greater than 1 which indicates the abundance of grains in the (002) and (100) directions. The lattice constants a_o and c_o are in a good agreement with the standard values (3.249Å) and (5.206Å) taken from (JCPDS) card file data. In hexagonal ideal hcp structure, the ratio of the lattice constants, c/a , is 1.602. In the present work two cases can be noticed, first the ratio is less than 1.602, which means that the atoms are compressed in the c-axis direction and second, the ratio is greater than 1.602 which means that the atoms are elongated along the c-axis.

AFM results

The 3D AFM micrographs of Co doped ZnO thin films are shown in Figure (3).

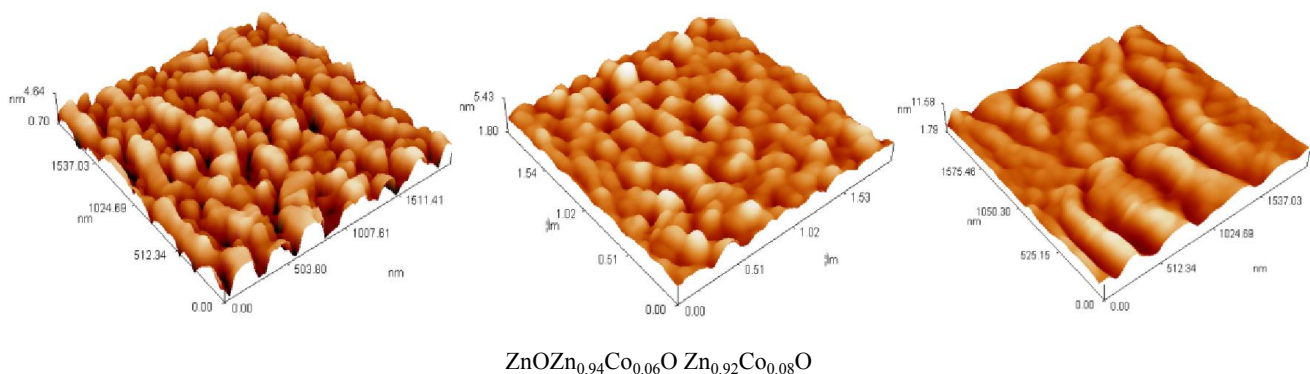


Figure 3. 3D AFM images of some of deposited films

The size of the scanned area was $(2 \times 2) \mu\text{m}^2$. The images show homogenous and smooth films. The average grain size obtained from the AFM granularity report, average roughness and root mean square (RMS) roughness for some samples are given in Table (2).

Table 2. The average grain size, average roughness and root mean square (RMS) roughness for some samples estimated from AFM

undoped ZnO			
Samples	Surface roughness (nm)	RMS (nm)	Grain size (nm)
Undoped ZnO	0.653	0.782	83.04
Zn _{0.94} Co _{0.06} O	0.421	0.321	113.72
Zn _{0.92} Co _{0.08} O	0.592	0.727	91.52

Optical properties

Optical absorption spectra of the films in spectral range of (300-900 nm) were recorded by using UV-visible spectrophotometer. The analysis of the dependence of absorption coefficient on photon energy in the high absorption region is performed to obtain the detailed information about the energy band gaps of the films (Sahin et al., 2014). Figure (4) shows the relation between transmittance for CodopedZnO thin films. It can be noticed that the transmittance increases rapidly as the wavelength increases in the range of (350- 450) nm for all films, which correspond to absorption band edge. Furthermore, small absorption bands can be noticed in the range of (590-690) nm for the Co doped ZnO samples. The absolute strength of these absorption bands increases almost linearly with the increase in Co concentrations and since the ionic radius of Co^{2+} (72 pm) being similar to that of Zn^{2+} (74 pm) in the tetrahedral coordinated structure, this phenomenon can be attributed to the d-d transitions of tetrahedrally coordinate Co^{2+} (Dinia et al., 2005). These transitions causes to change of sample color from white to green. The spectrum shows high transmittance in the visible and infrared regions, and low in the ultraviolet region.

It can also be observed that the fundamental absorption edge is sharp in the region at the wavelength (~ 390 nm) of the spectrum. The maximum transmittance observed of was about 95% for undoped ZnO sample. Figure (5) shows the relation between absorbance (A) and wavelength for the deposited thin films. The absorbance decreases rapidly at short wavelengths corresponding to the energy gap of the film. This evident increase of energy is due to the interaction of the material electrons with the incident photons which have enough energy for the occurrence of electron transitions. It is observed that the absorbance increases as the Co-concentration increases.

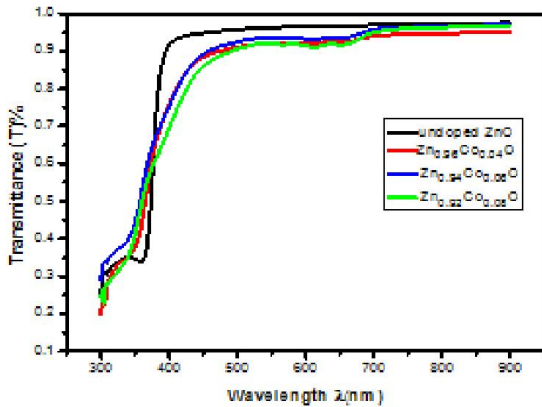


Figure 4. Transmittance versus wavelength for Co doped ZnO thin films

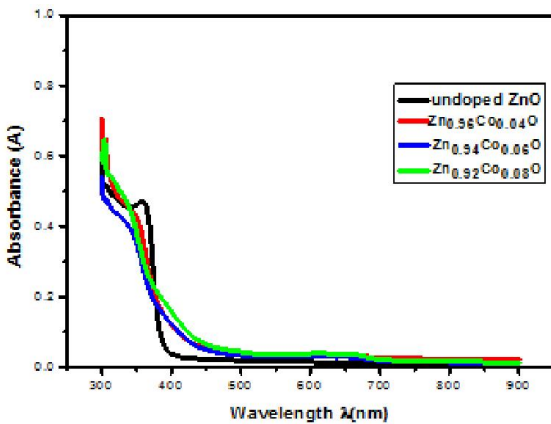


Figure 6. Absorbance versus wavelength for Codoped ZnO thin films

The absorption coefficient can be estimated from the absorbance using the well-known formula:

$$\alpha = (2.303 \times A) / t \tag{5}$$

Where A is the absorbance, t is the thickness and α is the absorption coefficient. It has been noticed that all the prepared thin films have high absorption coefficient in visible range of spectrum, and this could be seen in Figure (7). The absorption coefficient increases with increase in photon energy (hv). The absorption coefficient of the deposited thin films at different Co doping levels has values of ($\alpha > 10^4 \text{ cm}^{-1}$) which implies the increase of the probability of the occurrence of direct transitions.

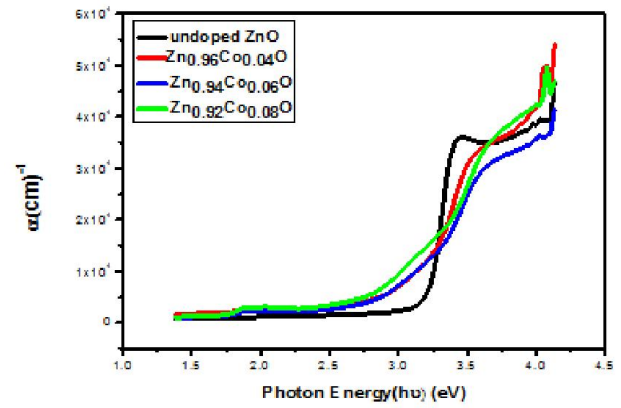


Figure 7. Absorption coefficient versus photon energy for Codoped ZnO thin films

The optical energy band gap (E_g) is given by the classical relation (Omer, 1975):

$$\alpha h\nu = B(h\nu - E_g)^r \tag{6}$$

Where α is the absorption coefficient, $h\nu$ is the photon energy, E_g is the optical band gap, A is a constant which does not depend on photon energy and r has four numeric values (1/2) for allowed direct, 2 for allowed indirect, 3 for forbidden direct and 3/2 for forbidden indirect optical transitions. In this work, direct band gap was determined by plotting a graph between $(\alpha h\nu)^2$ and $(h\nu)$ in eV, a straight line is obtained which gives the value of the direct band gap. The extrapolation of straight line to $(\alpha h\nu)^2 = 0$ gives value of the direct band gap of the material, and this could be seen in Figure (8). It is noticed that the band gap value increases with increase in Co doping concentration (4, 6, and 8) wt. %. This may be attributed to Burstein-Moss Shift (Kaiser and Fan, 1955). The refractive index has been calculated using the relation (Chopra and Kaur, 1983):

$$n_o = \left[\frac{(1+R)^2}{(1-R)^2} - (k_o^2 - 1) \right]^{1/2} + \frac{(1+R)}{(1-R)} \tag{7}$$

Where n_o is the refractive index, R is the reflectance and k_o is the extinction coefficient. The relation between refractive index and wavelength for ZnO thin films at different molarities is shown in Figure (9). It can be seen that the refractive index of the prepared films have values in the range of (2.71-1.41). The relation between extinction coefficient and wavelength for Co doped ZnO thin films is shown in Figure (10). The extinction coefficient K_o decreases rapidly at short wavelengths (300-400) nm and after that the value of K_o remains almost constant. The rise and fall in the value of K_o is directly related to the absorption of light. The lower value of K_o in the wavelength range (400-900) nm implies that these films absorb light in this region very easily. Results show that the extinction coefficient values of Co doped ZnO thin films are in the range of (0.012 - 0.13). The dielectric constant can be represented by the following equation (Sudad S. Ahmed et al., 2014):

$$\epsilon = \epsilon_1 - i\epsilon_2 \quad \dots\dots (8)$$

Where ϵ_1 is the real part of the complex dielectric constant and ϵ_2 is the imaginary part of it. For the calculation of the dielectric constant in its two parts one can use the following expressions:

$$\epsilon_1 = n_o^2 - K_o^2 \quad \dots\dots (9)$$

$$\epsilon_2 = 2n_o K_o \quad \dots\dots (10)$$

The relation between real and imaginary parts of dielectric constant and photon energy for Co doped ZnO thin films is shown in Figure (11). It can be seen that both the real and imaginary parts of the dielectric constant increase as photon energy increases in the range of (1.25-3.5) eV and after that the value of the real and imaginary parts decrease. It is observed that both the real and imaginary parts of the dielectric constant increase with increase in Co doping concentration.

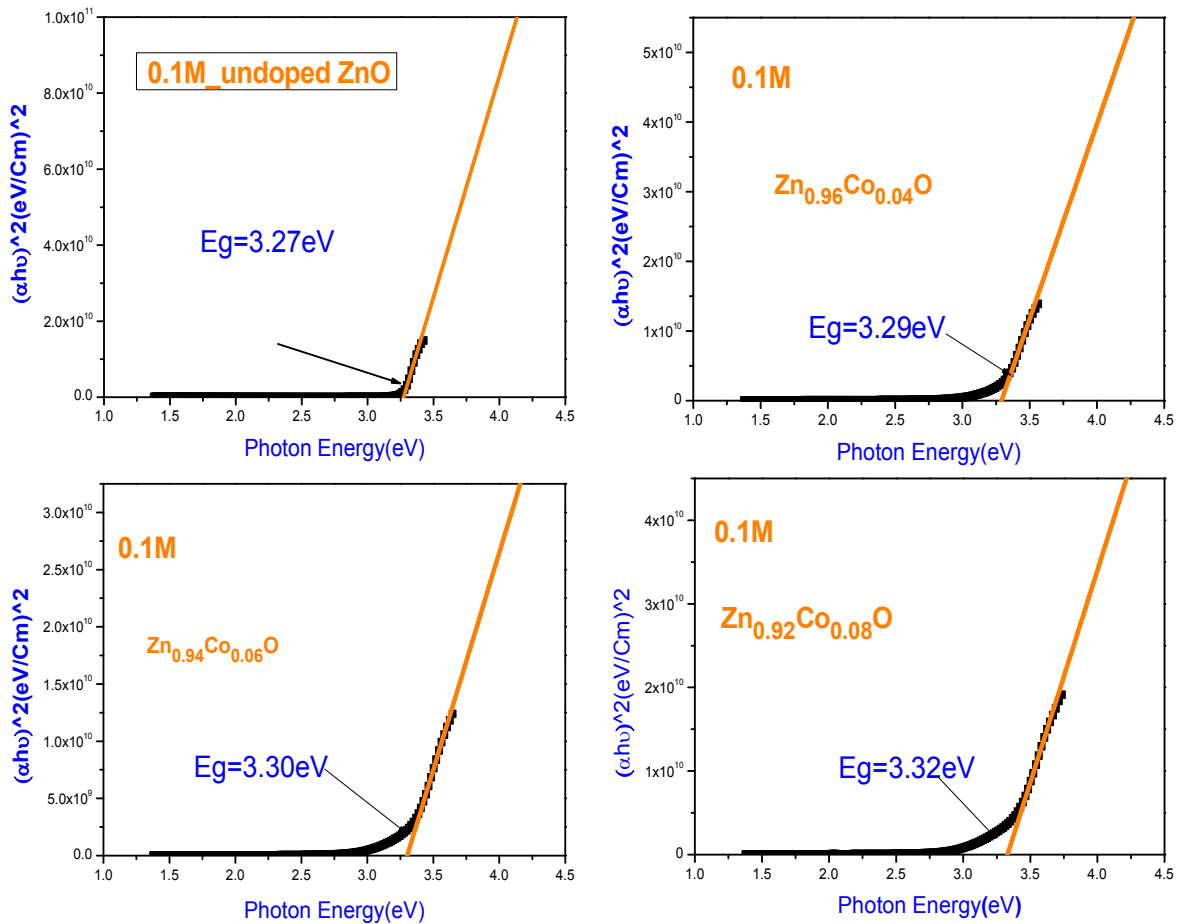


Figure 8. The relation between $(\alpha h\nu)^2$ and $(h\nu)$ for Co doped ZnO thin films

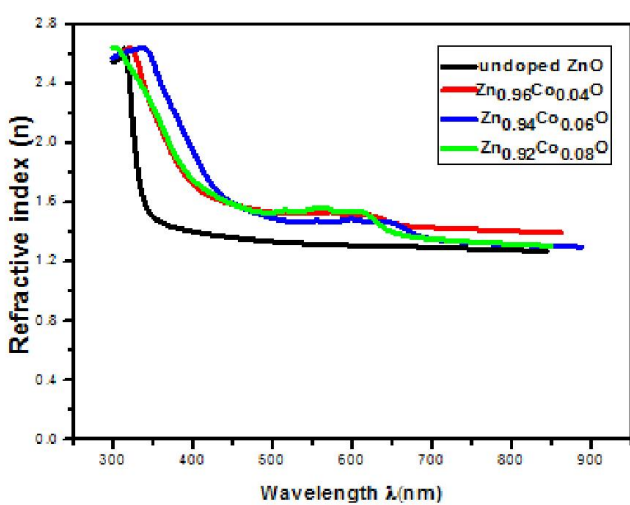


Figure 9. Refractive index versus wavelength of Co doped ZnO thin films

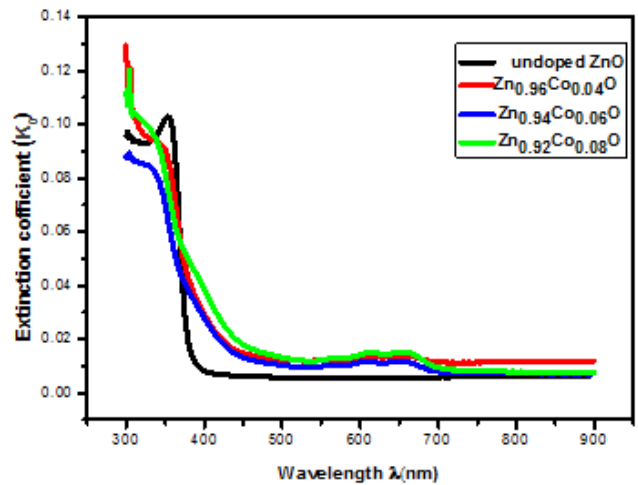


Figure 10. Extinction coefficient versus wavelength of CodopedZnO thin films

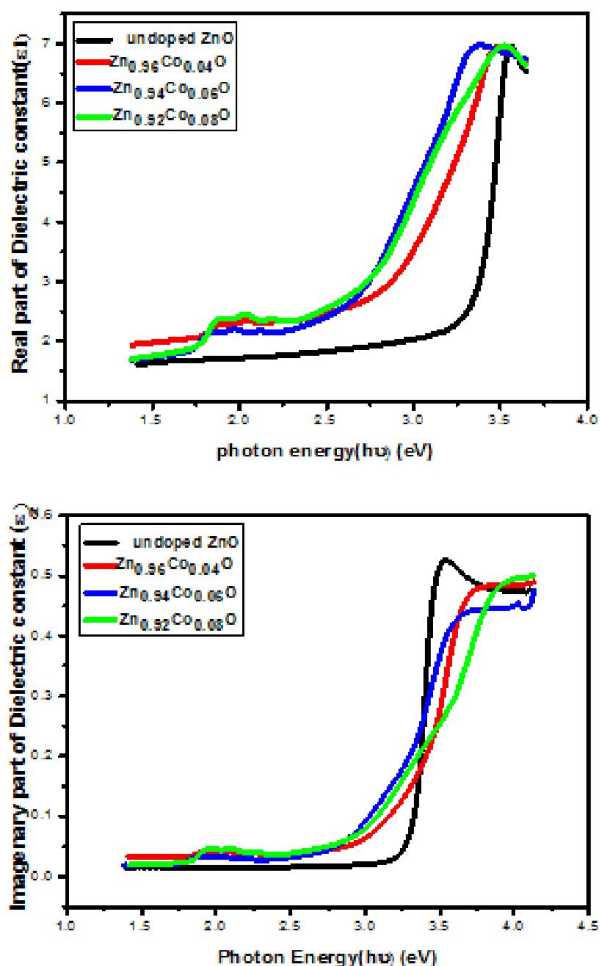


Figure 11. Real and imaginary parts of dielectric constant versus photon energy of Co doped ZnO thin films

Conclusion

In this study Zinc-Cobalt Oxide ($Zn_{(1-x)}Co_xO$) thin films, where $x = 0, 4, 6$ and 8% were successfully deposited on glass substrates at ($400^\circ C$) by chemical spray pyrolysis technique using Zinc and Cobalt acetate as the Zn and Co source. XRD patterns of the Co doped Zinc Oxide thin films reveal a good crystalline behavior with the hexagonal wurtzite structure. The main characteristic peaks are assigned to the (100), (200) and (220) planes. The adding of Co dopant led to a decrease in the average crystalline size and a stable maximum increase in the transmittance in (IR) region which can be useful in optoelectronic devices. The transmittance increases rapidly as the wavelength increases in the range of (350- 450) nm for all films, which correspond to absorption band edge. Small absorption bands had been noticed in the range of (590-690) nm for the Co doped ZnO samples. The absolute strength of these absorption bands increases almost linearly with the increase in Co concentration. This phenomenon can be attributed to the d-d transitions of tetrahedrally coordinate Co^{2+} . These transition causes to change of sample color from white to green. AFM results showed homogenous and smooth thin films. The undoped ZnO thin film has highest grain size, average roughness and RMS roughness. The band gap increases as the Co concentration increases and the band gap values range between 3.27 eV and 3.32eV and this may be attributed to Burstein-Moss Shift.

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