Effect of Gamma Ray Irradiation on Structural and Optical Properties of ZnO Thin Films

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Abstract

In this research, the structural and optical measurements were made on the Zinc oxide (ZnO) films prepared by two methods once by using chemical spray pyrolysis technique, and another by using thermal evaporation technique before and after irradiation by Gamma –Ray (γ – rays) from source type (Cs^{137}) with an energy (0.611)MeV as a function of gamma dose (0.15,0.3 and 0.45) Gy. The thickness of all films prepared by two method was about (300 ± 50) nm.

XRD is used to characterize the structural properties, the results demonstrated that all samples prepared by two method before and after irradiation have polycrystalline structure with a preferred orientation (002).Also it showed that the structural properties are weakly dependent on the gamma dose. The optical measurement shows that all ZnO films prepared by two method have a direct energy gap, and they in general decrease with the increase of Gamma dose while the optical constant such as absorption coefficient, refractive index, extinction coefficient, real and imaginary parts of the dielectric constant and optical conductivity showed an opposite trend, these values increase with the increase of irradiation dose. As well as all optical properties for the samples prepared by thermal evaporation technique is higher than the samples prepared by chemical spray pyrolysis technique.

Key words: Zinc oxide film, Structural properties, Optical Constant, Gamma irradiation.

Introduction

ZnO thin film is one of the II-VI compound semiconductors, composed of hexagonal wurtzite crystal structure, it has a broad band gap (~3.3) eV at room temperature, large excitation binding energy(60)MeV and high lattice constant[1,2]. Zinc oxide thin films have unique physiochemical properties such as chemical stability in hydrogen plasma, high optical transparency in the visible and near-infrared region of the electromagnetic spectrum and high refractive index[3,4,5]. Due to these properties, ZnO is used in various applications such as solar cell window, antireflection coating ,transparent conducting material , flat displays, heat mirrors, chemical sensors, gas sensors, ultraviolet light sensors, light emitting diodes (LEDs), and other optoelectronic devices [2,5,6] . Several growth techniques have been used by different workers to prepare ZnO films such as physical vapor deposition (PVD) [7], sol-gel [2, 4, 8], chemical spray pyrolysis [3], electro-deposition [9], sputtering [5, 10], chemical vapor deposition (CVD) [11], etc.

Ionizing radiations such as X-rays, gamma-rays, beta particles, alpha particles, fission fragments, etc. are present in several fields that include industry, medicine, military, particle accelerator based research, nuclear power plants, etc. Several studies have been made on thin films made up of metal oxides and their mixtures for their use in radiation dosimetry, sensing, and memory cells. It is a well-known fact that the ionizing radiation produces changes in the physical properties (such as optical, electrical, structural, etc.) of the material[12]. One of the most promising applications of thin film solar cells is to work in the outer space, therefore it is very important to investigate the influence of radiation on these films.

In this research, ZnO thin films are prepared by using chemical spray pyrolysis technique once, and thermal evaporation technique in another time. All samples were exposed to gamma ray form Cs^{137}withenergy (0.611)MeV for different doses (0.15, 0.3 and 0.45) Gy. The effect of two different preparation techniques on the structural and optical properties of ZnO thin films before and after irradiation by (γ – rays) at different doses was studied and investigated.

Experimental

The first sets of Zinc oxide (ZnO) thin film samples are prepared by chemical spray pyrolysis method, the solution of (Zin Acetate Dehydrate) $Zn(CH_3COO)_2.H_2O$ was prepared in molarity (0.1) by diluted (2.195) gm of $Zn(CH_3COO)_2.H_2O$ in (100) ml water in accordance with the following equation: [13]

$$M = (W_t / M_{wt}). (1000 / V) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where M = molarity, W_t = weight of sample, M_{wt} = molecular weight, V = water volume.

The obtained solution is used to prepare (ZnO) films of thickness (300 ± 50) nm by spraying the solution on preheated glass substrates at about (623)K which is measured by using thermocouple type NiCr , the distance between sprayer nozzle and substrate of (25)cm, these glass substrates are placed on the hot plate for about (25) min before the spraying process, each spraying period lasts for about (15) sec followed by (2.5) min waiting period to avoid excessive cooling of the hot substrate due to the spraying process, based on the reaction:[14]

$$Zn(CH_3COO)_{2(solid\ near\ substrate)} \xrightarrow{hea} 4Zn(CH_3COO)_{2(gas\ near\ substrate)} + H_2O \xrightarrow{adsorption} Zn_4O(CH_3COO)_{6(adsorbed/\ substrate)} + 2CH_3COOH_{(gas\ near\ substrate)}$$

and

$$\uparrow Zn_4O(CH_3COO)_{6(adsorbed/\ substrate)} + 3H_2O \rightarrow 4ZnO\ (film/\ substrate) + 6CH_3COOH_{(gas)} \uparrow \ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

Another set of samples has been prepared in a high vacuum system of ($3x10^{-6}$) torr where pure metal Zinc thin films deposited on glass substrate at Ts = (423)K, of thickness (300 ± 50) nm by thermal evaporation technique using Edward coating unit model (E 306), molybdenum

boat was used to hold powdered samples, the distance between the substrate and boat was about (15)cm. Then we get ZnO thin films by thermal oxidation processes with exist air flow at temperature of (673) K for one hour by using (Kilns Furnaces).

The thickness of all samples prepared was determined by using weighing method using a sensitive balance whose sensitivity is of the order (10^{-4}) gm, according to the following relation: [15]

$$t = (m / à .\rho) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where t= thickness of film, m= mass of film, à= area of film, ρ= density of films.

Gamma ray form a disc-type Cs^{137} source with an energy (0.611)MeV exposure to ZnO films using different doses (0.15, 0.3 and 0.45) Gy.

The crystal structure characteristics of all samples before and after irradiation by (γ – rays) were measured with X-ray diffraction technique by recording the intensity in the range of Bragg's angle θ from (20-80) using Siemens X-ray diffractometer system (SHIMADZU Japan XRD 600), source Cu K_α radiation of wavelength (λ=1.5405) A was employed with generator setting of current (30) mA and voltage (40) kV. Transmittance (T) and absorbance (A) measurements have been carried out for different samples in the range of wavelengths (300-1100) nm using a double beam spectrophotometer (UV/VIS).

Results and Discussion

Structural Properties

Figure 1(a,b) shows X-ray diffraction pattern of Zinc oxide films prepared by chemical spray pyrolysis method and thermal evaporation technique respectively, before and after irradiation by Gamma dose (0.45) Gy. These figures show polycrystalline nature of the ZnO films with hexagonal phase formation as compared with ASTM , the peaks are observed due to diffraction from different planes with a preferred orientation (002) at around 34° , and small peaks belong also to ZnO phase with orientation at (101) and (100) direction. We can notice from these figures similar plots are obtained before irradiation and after irradiation with different preparation methods which show that no new peak is observed among the irradiated samples in comparison with that of the control sample, unless simple shift in 2θ position due to the gamma rays is not capable of displacement of damage in structure directly indicating that the film composition remains unchanged after radiation and structural properties are weakly dependent on the gamma dose .

The grain size dimension (D) of the films could be calculated from diffraction line broadening using the Scherrer equation: [16]

$$D = k\lambda / \beta\cos\theta \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where k is a constant (0.9), and β is full width half maximum (FWHM) of the preferential plane. We can calculate the dislocation density (δ) and strain (η) values from the equations: [17]

$$\delta = 1/ D^2 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$\eta = (\beta\cos\theta)/ 4 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

While the number of crystallites per unit surface area (N) was calculated using the formula: [17]

$$N = t / D^3 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

The observed d (hkl) values are in a good agreement with the standard values taken from ASTM data[18], the relative percentage error for the observed and ASTM standard d-values for crystalline ZnO thin films are calculated using the formula: [14]

$$\text{Relative percentage error} = \frac{|z_1 - z|}{z} \times 100 \quad \ldots\ldots\ldots\ldots (8)$$

where Z_1 represents the actual d value obtained and Z is the standard d value in JCPDS card file. All these parameters are shown in Table (1). The results obtained have shown that the grain size increase while dislocation density , number of crystallites per unit surface area and

strain values showed an opposite trend after irradiation, which demonstrated the improvement of the crystallinity in the films with the increase of gamma dose.

Optical Properties

The influence of different gamma doses on the optical properties of ZnO thin films grown on glass substrates by chemical spray pyrolysis method and thermal evaporation technique are studied deeply. We measured the transmission and the absorption spectrum in the range (300-1100) nm by using a double beam spectrophotometer (UV).

Fig.2 (a, b) shows the plots of transmittance (T) as a function of wavelength for ZnO thin films before and after irradiation, prepared by chemical spray pyrolysis method and thermal evaporation technique respectively, from these figures we can notice that the transmittance values increase generally as the wavelength increases and have high values in the NIR region, all samples demonstrate (40-99) % transmittance at wavelengths longer than 400 nm which makes these films suitable for solar cell window. Also we can see from these figures that the value of the transmittance of ZnO thin films decreased with the increase of Gamma dose for all values of the wavelength due to the fact that when thin films are prepared, some intrinsic defects are always present. The interaction of gamma radiation induces defects during its passage through the thin film which results in a change in the order at the micro structural level of the film. For small gamma doses, these thin films possess fine uniform grains with no big pores which reduces the transmittance and increase absorbance[12].Transmittance values are observed to be higher for the films prepared by chemical spray pyrolysis method.

The incident photon energy (E=hν) was calculated as a function of wavelength (λ) according to equation: [16]

$$E\ (eV) = (1240 / \lambda) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (9)$$

The variation of absorption coefficient for each wavelength was calculated from equation: [16]

$$\alpha = 2.303\ (A / t) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ (10)$$

Where A = absorbance.

Fig.3 (a,b) shows a plot of the absorption coefficient (α) versus photon energy for two sets of ZnO thin films as a function of irradiation dose, we can notice from these figures that the absorption coefficient values increase with the increase of gamma dose within the whole range of the spectrum, this behavior may be due to the changes in crystal structure of these films after irradiation, improvement of the crystallinity in the films leads to the growth of small grain and decrease of grain boundaries; the absorption is not attributed to the free carriers only, but to impurities or localized electronic states. Also we can notice from these figures that all the films have high values of absorption coefficient ($\alpha > 10^4$ cm^{-1}) this means that the direct transition possibly occurs. This result is in agreement with refs. [4 ,14]

Figure 4 (a,b) shows a plot of the relation $(\alpha h\nu)^2$ versus photon energy (hν) to find the type of the optical transition for all ZnO films prepared by chemical spray pyrolysis method and thermal evaporation technique respectively before and after irradiation, which describes the allowed direct transition and calculation of the optical energy gap (Eg^{opt}) values from Tauc formulas: [19]

$$(\alpha h\nu) = B`\ (h\nu - Eg^{opt})^{r} \quad \ldots\ldots\ldots\ldots\ (11)$$

Where B` is a constant inversely proportional to amorphousity, (Eg^{opt}) is the optical energy gap ,r is constant depending on the material and the type of the optical transition, by selecting the optimum linear part, which is determined by the extrapolation of the portion at (α =0).It is clear from Fig. (5) that the optical energy gap values decrease when the irradiation dose increased. This behavior can be attributed to generate extra energy levels after irradiation between the valance and conduction bands. The value of the optical energy gap agrees with refs. [4 ,14].

The optical constants which include refractive index (n), extinction coefficient (k), and dielectric constant (ε) for each wavelength in the range (300-1100) nm for all ZnO samples prepared before and after irradiation are studied and investigated.

The refractive index value can be calculated from the formula: [20]

$$n = \{[4R / (R-1)] - k^2\}^{1/2} – [(R+1) / (R-1)] \quad (12)$$

Where R is the reflectance which is calculated by using equation: [20]

$$R = 1-T-A \quad (13)$$

The absorption coefficient (α) is related to extinction coefficient (k) by: [19]

$$K = \alpha \lambda / 4\pi \quad (14)$$

The complex dielectric constant can be introduced by: [21]

$$\varepsilon = \varepsilon_1 - i\,\varepsilon_2 \quad (15)$$

Where $\varepsilon_1 = n^2 - k^2$ (16)

$$\varepsilon_2 = 2nk \quad (17)$$

Where ε_1= real part of dielectric constant, ε_2 = imaginary part of dielectric constant.

Figures from (6) to (9) show the variation of these optical constants versus photon energy (hv) for ZnO thin films prepared by chemical spray pyrolysis method and thermal evaporation technique as a function of irradiation dose. We can see from these figures that all optical constants increase with irradiation dose for the same reasons as we mentioned before. Also, it is clear that the optical constants values for the samples prepared by thermal evaporation are higher than the samples prepared by chemical spray pyrolysis method.

The optical conductivity (σ) was calculated using the relation: [22]

$$\sigma = \alpha nc / 4\pi \quad (18)$$

Where (c) is the velocity of light.

Figure 10(a,b) shows the variation of optical conductivity as a function of photon energy of ZnO films before and after irradiation prepared by chemical spray pyrolysis method and thermal evaporation technique respectively .We can notice from these figures that the optical conductivity value increases after irradiated with gamma ray. This behavior is due to the optical conductivity value which depends mainly on refractive index and absorption coefficients according to equation (18).

Conclusion

ZnO thin films were synthesized using two methods once by chemical spray pyrolysis technique, and another by using thermal evaporation technique, the influence of Gamma dose on the structural and optical properties of (ZnO) films has been investigated. XRD pattern for all films prepared confirms the formation of ZnO phase with preferential orientation along (002) plane and the film composition remains unchanged after radiation which indicates that the structural properties are weakly dependent on the gamma dose. The crystallites sizes as measured using XRD data are found to be in the range of (25– 27) nm for the samples prepared by chemical spray pyrolysis while the samples prepared by thermal evaporation technique are found to be in the range of(21– 23) nm. The grain size increases while dislocation density,number of crystallites per unit surface area and strain values showed an opposite trend after irradiation for all samples.

All films prepared have high values of absorption coefficient ($\alpha > 10^4$ cm^{-1}) , the absorbance values increase while the transmittance values decrease after irradiation, the average transmittance is more than 70% for wavelengths in the (400–1100) nm range, indicating these films have high transmittance in the NIR region, which makes it suitable for use as a window material in photovoltaic application. The optical energy gap values decrease when gamma dose increases, while the increase of gamma dose resulted increase values of all optical constant (refractive index , absorption coefficient, extinction coefficient, real and imaginary parts of the dielectric constant and optical conductivity). All optical properties for

the samples prepared by thermal evaporation technique are higher than the samples prepared by chemical spray pyrolysis technique.

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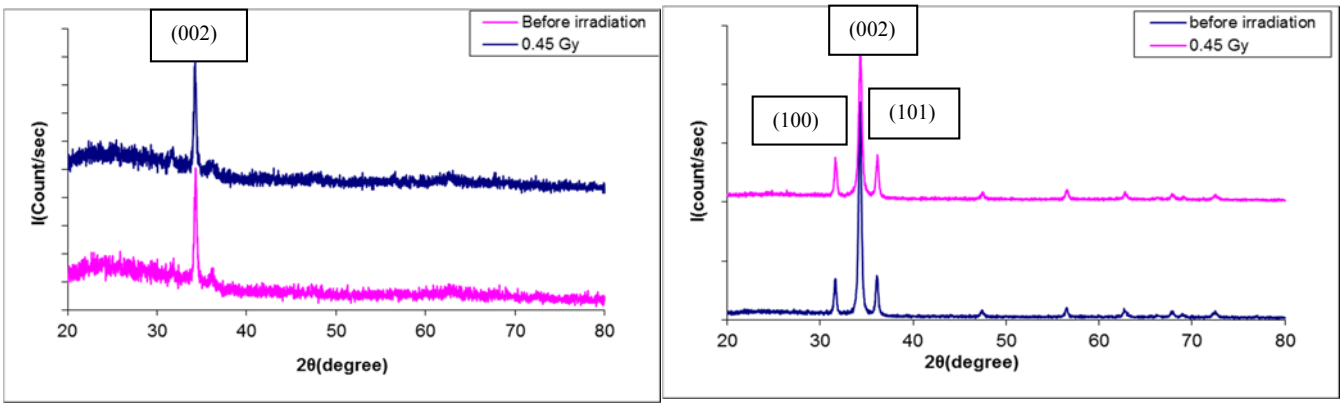
Table (1): XRD results of ZnO thin films for the (002) preferred peak

Sample prepared technique	State	d(A°) (ASTM)	d(A°) Observed	Error%	D (nm)	$\eta*10^{-3}$	$\delta*10^{14}$ (m^{-2})	$N*10^{15}$ (m^{-2})
chemical spray pyrolysis	Before irradiation	2.6033	2.61118	3.027	25.727	1.347	15.11	1.7618
	0.45 Gy	2.6033	2.61528	4.602	27.204	1.274	13.51	1.4901
thermal evaporation	Before irradiation	2.6033	2.61197	3.33	21.639	1.602	21.36	2.9607
	0.45 Gy	2.6033	2.609422	2.352	23.921	1.449	17.48	2.1917



(a) **(b)**

Figure (1): X-ray diffraction pattern of ZnO thin films before and after irradiation
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

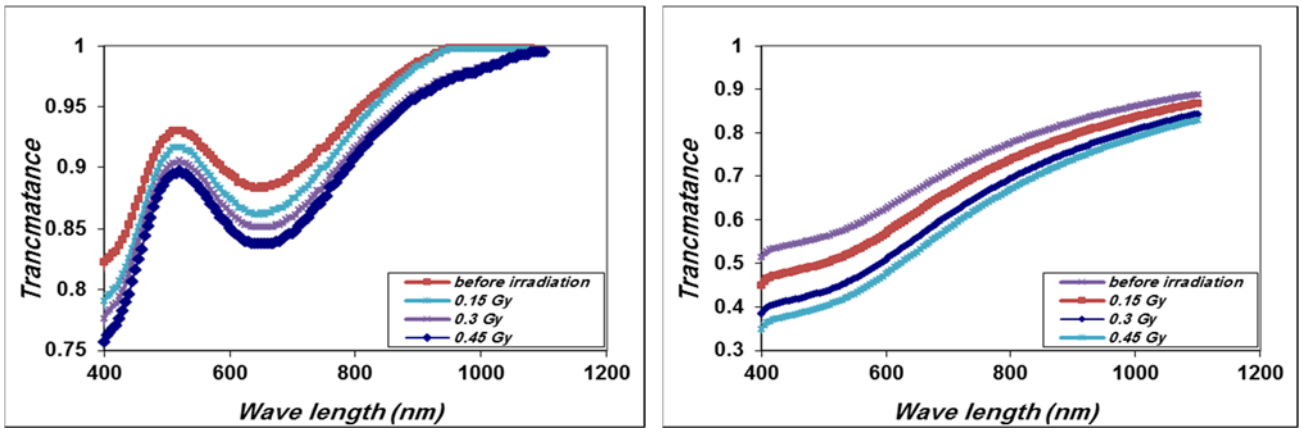


Figure (2): The variation of transmittance versus wavelength for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

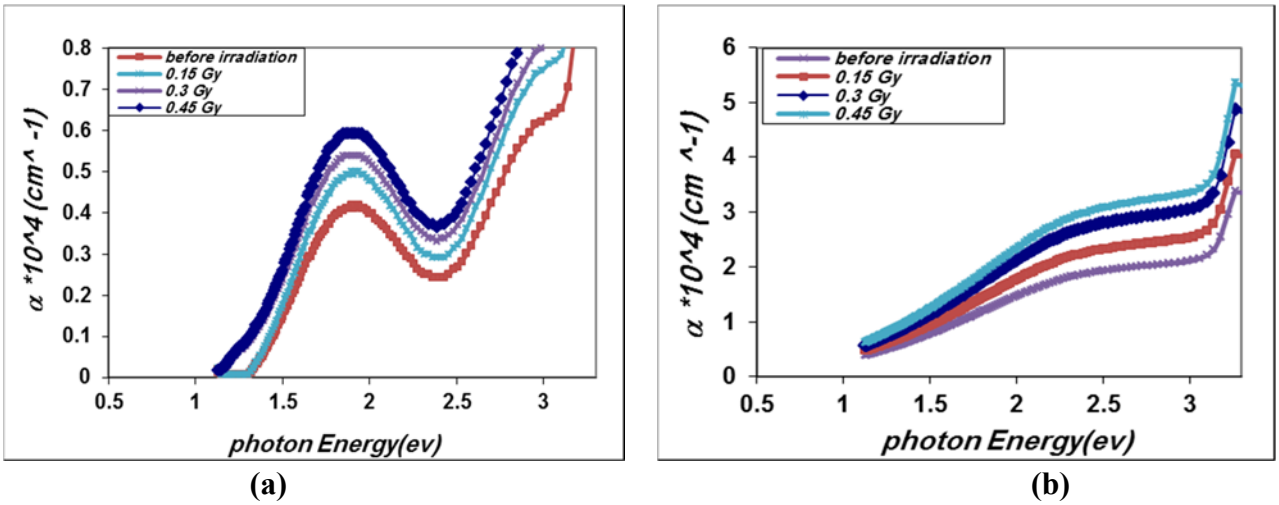


Figure (3): Absorption coefficient & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

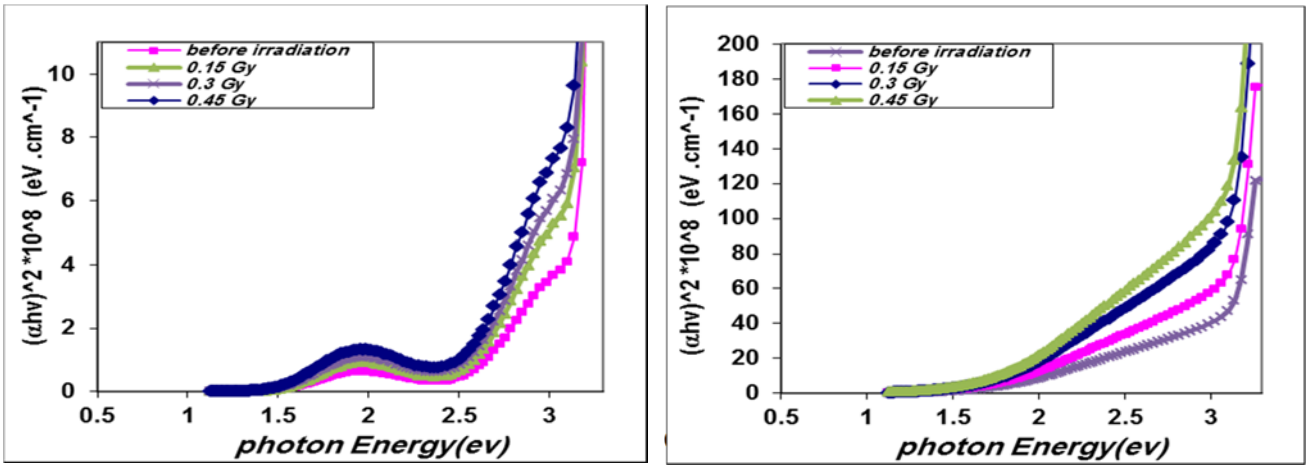


Figure (4):Variation (α hν)^2 & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

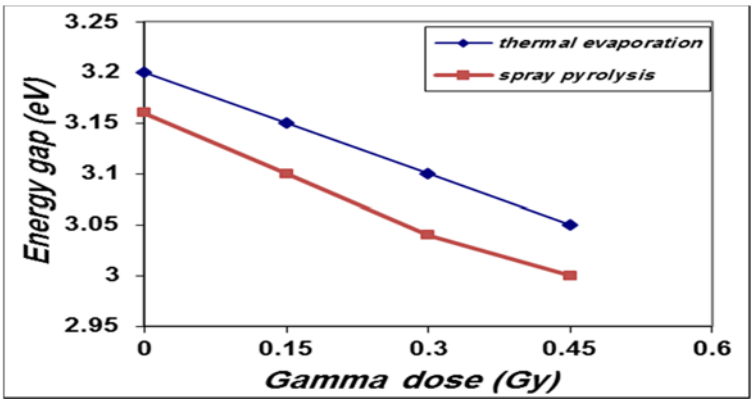
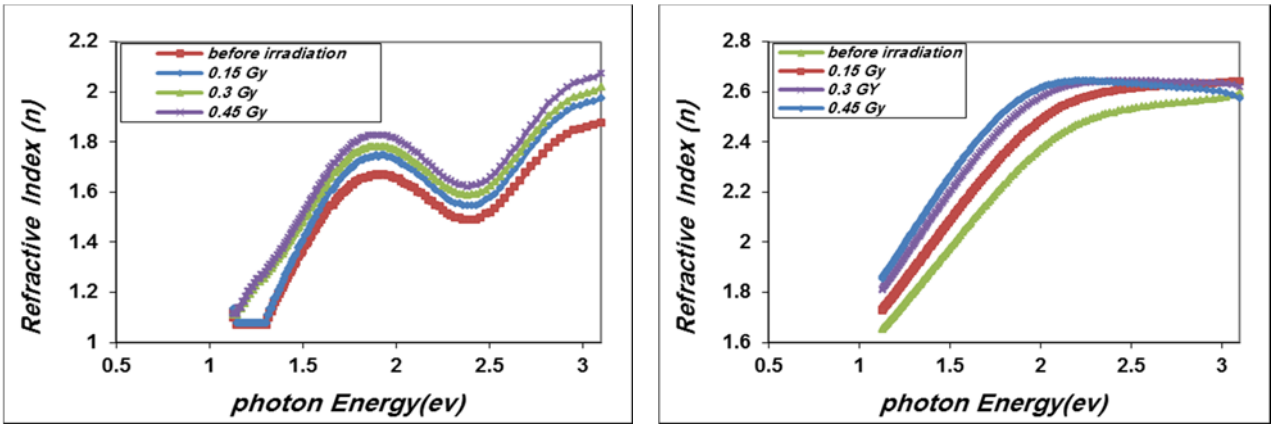


Figure (5): Variation optical energy gap as a function of gamma dose for ZnO thin films



(a) **(b)**

Figure (6): Variation refractive index & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

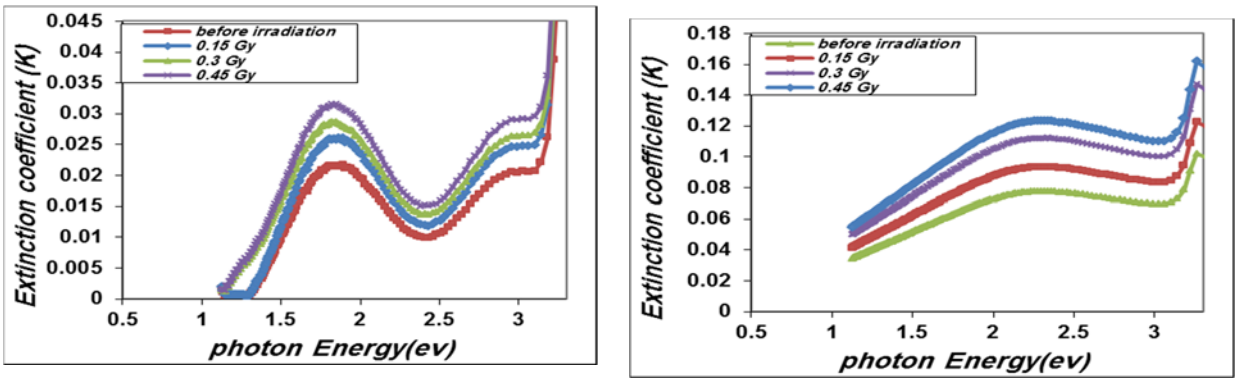


Figure (7): Variation extinction coefficient & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

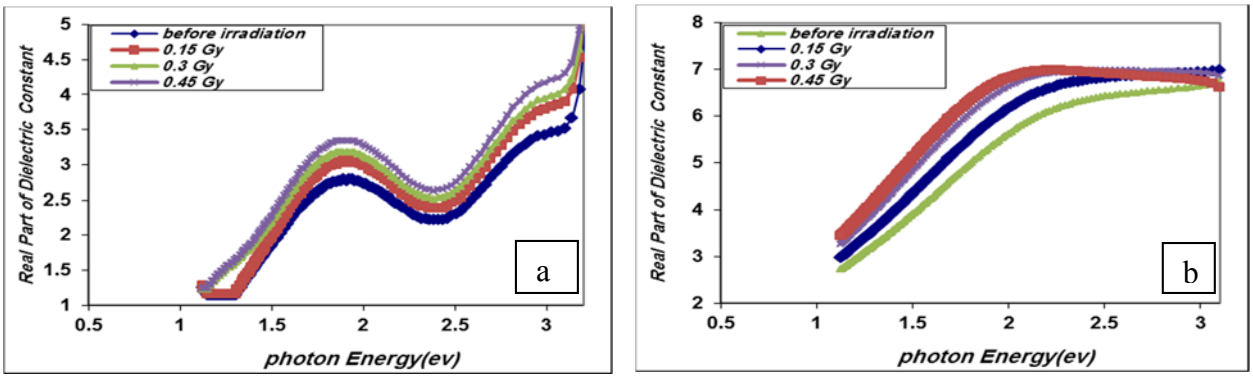


Figure (8): Real part of dielectric constant & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

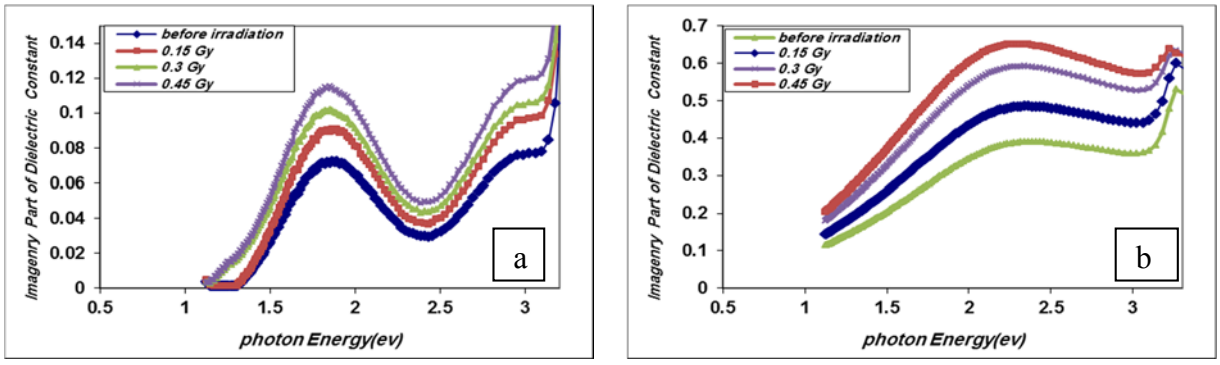


Figure (9): Imaginary part of dielectric constant & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

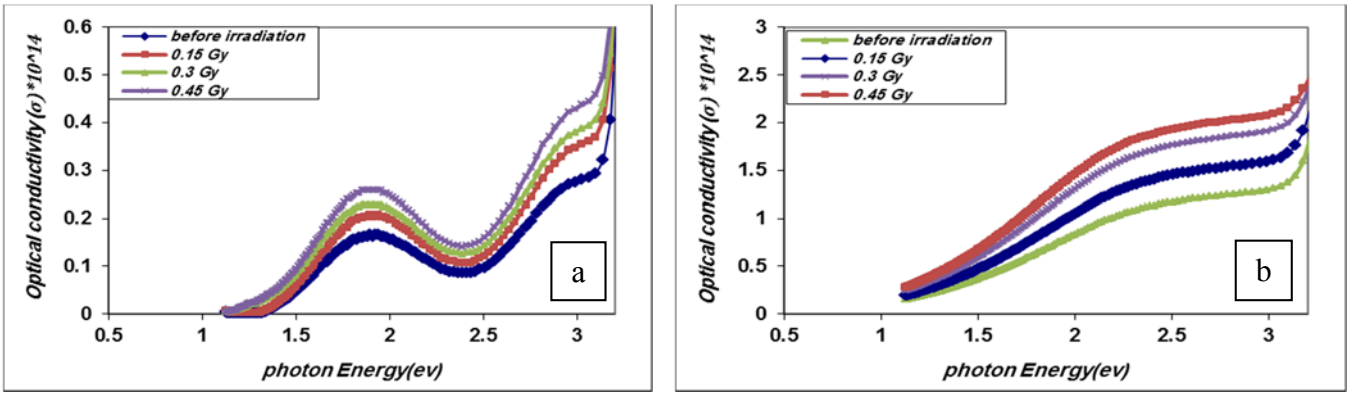


Figure (10): Optical conductivity & photon energy for ZnO thin films
(a) prepared by chemical spray pyrolysis , (b) prepared by thermal evaporation

تأثير التشعيع بأشعة كاما في الخواص التركيبية والبصرية لاغشية ZnO الرقيقة

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الخلاصة

أجريت القياسات التركيبية والبصرية لاغشية أوكسيد الزنك (ZnO) في هذا البحث المحضرة بطريقتين واحدة باستعمال طريقة الرش الكيميائي الحراري والاخرى باستعمال طريقة التبخير الحراري قبل وبعد التشعيع. باشعة كاما من مصدر (Cs^{137}) بطاقة (0.611) MeV بجرع اشعاعية مختلفة (0.15,0.3 and 0.45) Gy. وكانت جميع الاغشية المحضرة بكلا الطريقتين بسمك (300 ± 50) nm واستعملت XRD لايجاد الخصائص التركيبية واكدت النتائج ان لجميع النماذج المحضرة بكلا الطريقتين قبل وبعد التشعيع تركيب متعدد التبلور بأفضلية للاتجاه (002) وان الخصائص التركيبية قليلة الاعتماد على جرعة الاشعاع . وبينت القياسات البصرية ان لاغشية (ZnO) المحضرة بكلا الطريقتين فجوة طاقة مباشرة وتقل قيمتها بصورة عامة بزيادة جرعة الاشعاع بينما اظهرت الثوابت البصرية مثل معامل الامتصاص ومعامل الانكسار ومعامل الخمود وثابت العزل الكهربائي بجزئيه الحقيقي والخيالي والنوصيلية الضوئية سلوك معاكس اذ ازدادت قيمهم بزيادة جرعة الاشعاع .وأظهرت الخصائص البصرية للنماذج المحضرة بطريقة التبخير الحراري قيم اعلى من النماذج المحضرة بطريقة الرش الكيميائي الحراري.

الكلمات المفتاحية :- اغشية أوكسيد الزنك ، الخصائص التركيبية، الثوابت البصرية، اشعة كاما