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Effect of Composition and Annealing on Structural and Optical Properties of $(ZnO)_{1-X}$ $(TiO_2)_X$ Thin Films Prepared by Sol-Gel Method

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ABSTRACT

The study describes effect of composition parameter, x, and annealing-temperature, T_a , on structural, and optical properties of $(ZnO)_{1-x}(TiO_2)_x$ thin-films prepared by sol-gel method. Experimental results are explained and theoretical equations are suggested for optical-energy-gap, E_g, and Urbach-energy, E_u in terms of their variation with (x) and (T_a). With increased x and Ta, E_g decreases whereas E_u increases. The behavior of E_g was explained by evoking that increasing x will randomize the crystal periodicity, and that annealing temperature (400,500, and600) ℃ relaxes the structure more towards better crystalline forms. The increase of E_u with increased x was attributed to the lower mass of Ti ions as compared to that of Zn ions. Generally, making composites destroys the crystalinity observed in pure ZnO and pure TiO_2 films but annealing does improve the quality of the film in terms of its degree of crystal structure.

Keywords: *$(ZnO)_{1-x}(TiO_2)_x$ thin-film, composition, annealing, energy-gap, Urbach-energy.*

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تأثير التركيب والتلدين على الخصائص التركيبية والبصرية لأغشية $(ZnO)_{1-x}(TiO_2)_x$ المحضّرة بطريقة السائل الهلامي

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الملخص

الدراسة تصف تأثير نسبة المزج (x) ودرجة حرارة التلدين (T_a) على الخصائص التركيبية والبصرية لأغشية رقيقة من ($(ZnO)_{1-x}(TiO_2)_x$) محضّرة بطريقة سول– جل. تم تفسير النتائج التجريبية واقتراح معادلات نظرية لفجوة–الطاقة البصرية (E_g) وطاقة أورباخ (E_u) بدلالة تغيرهما مع كل من (x) و(T_a). مع زيادة (x) و(T_a)، تقل (E_g) في حين تزداد (E_u). لقد تم تفسيرتصرف فجوة–الطاقة بالأشارة إلى أن زيادة (x) سيزيد من عشوائية تكرارية (أو دورية) االتركيب، وأن درجة حرارة التلدين (600،500،400) ^{0}س تجعل التركيب يتراخى بشكل أفضل نحو الصيغ البلورية . زيادة (E_u) مع زيادة (x) فسرت بكون كتلة أيونات التيتانيوم (Ti) أصغر من كتلة أيونات الزنك (Zn). بشكل عام فإن عمل التركيب يهدم الصيغ البلورية التي تلاحظ في ألاغشية النقية لـ (TiO_2) و لـ (ZnO)، ولكن التلدين يعمل على تحسين نوعية الأغشية من حيث درجة التركيب البلوري.

الكلمات الدالة : أغشية رقيقة ($(ZnO)_{1-x}(TiO_2)_x$)، التركيب، التلدين، فجوة الطاقة، طاقة أورباخ.

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1.INTRODUCTION

Oxides of zinc (ZnO) and titanium (TiO_2) attracted the attention of researchers for years for their potential use in various applications. The applications cover a wide range including Surface-Acoustic-Wave (SAW) Devices, LEDs, solar-cells, transparent- electrodes and displays [1-6]. Composites and/or bi-layers of ZnO and TiO_2 are also considered for these applications by various groups of researchers [7-9]. Efforts to improve characteristics and/or performance is still required and ongoing [10-15].

The method of preparation and post annealing are considered as important parameters to improve film quality and device performance.

In this study we report results of investigations carried out in order to study the effect of composition and annealing-temperature on structural, and optical properties of composite thin-films of $(ZnO)_{1-x}(TiO_2)_x$ prepared by sol-gel method for values of x from x=0.05 up to x=0.25 and annealing-temperatures of (400, 500, and 600)^{o}C. The sol-gel process has the advantage of simplicity and it is cost effective. X-ray diffraction (XRD), atomic-force-microscopy (AFM), and UV-VIS-NIR spectroscopy were used for characterization.

2.EXPERIMENTAL DETAILS

2.1.Sample preparation:

Thin films of oxides of ZnO,TiO_2 and $(ZnO)_{1-X}$ $(TiO_2)_X$ with x=(5%,10%,15%,20%,25%) were deposited on glass substrates(76.2×25.4×1 mm) using sol-gel spin coating technique. The glass substrates were cleaned with acetone and then in ethanol for 20 min. The slides were removed from the ethanol and dried with special paper at room temperature.

ZnO solution with 0.5 molarities was prepared by dissolving Zinc acetate { $Zn(CH_3Coo)_2$:$2H_2O$} in ethanol solution and stirring it at 60˚C for 5 min after that we add Diethanolamine; DEA ($C_4H_{11}NO_2$) at a rate of one drop /sec, the mixture solution stirred at 60˚C for 2.0 h to obtain clear light solution which statically aged at room temperature for 24 h. Similarly, TiO_2 solution was prepared by dissolving Tetra Butyl Titanate (TBT) in ethanol solution instead of Zinc acetate. For the composite structure; $(ZnO)_{1-x}$/$(TiO_2)_x$ we add (0.85 , 1.7 , 2.55 , 3.4 , 4.25

ml) of TiO_2 solution to 10 ml of ZnO solution in order to prepare (5%, 10% ,15% ,20% ,25%) of ZnO/TiO_2 composites respectively .

After preparing the sol-gels we deposit it on the glass substrate using a spin coater with 3000 rps for 30 sec. After deposition, the films were dried at 300˚C for 10 min to evaporate the solvent and remove organic residuals. Then, the films were finally post annealing at three temperatures; 400, 500, and 600˚C.

2.2.Characterization:

X-ray diffraction records were obtained using X-Ray diffraction- SHIMADZU (Cu-Kα).in Nanotechnology &Advanced Materials research center, university of technology. The atomic force microscopy (AFM) measurements were done using model (AA3000) scanning prop microscope through the courtesy of college of science, university of Baghdad. The spectrophotometric data were recorded using double-beam Jenway 7315 spectrophotometer. The thickness of the films was measured by the method of fringes using sodium light.**[16]**

3. RESULTS AND DISCUSSION

3.1.Structural properties:

Figure (1) shows the XRD-spectrum of some ZnO films post annealed at(400, 500, and 600)^{o}C. The figure shows that annealing does improve quality of the film interms of its degree of crystal structure. Similar results for TiO_2 films are shown in Figure (2). Figure (3) displays XRD-spectrum for $(ZnO)_{1-x}(TiO_2)_x$ composite films post annealed at 600 oC for values of x from 5% to 25%

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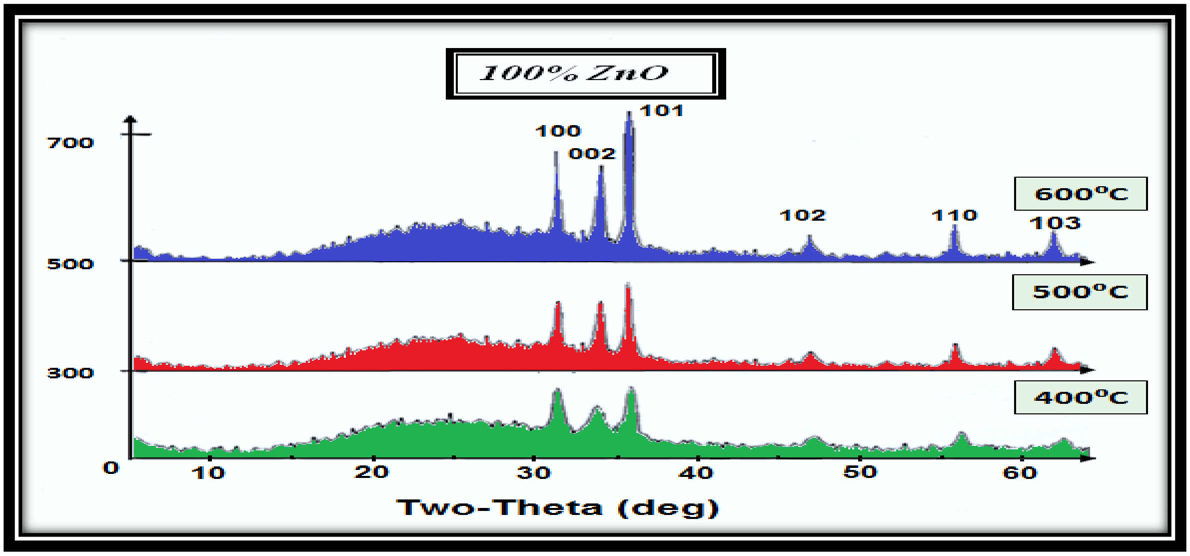

Figure (1): XRD-spectrum for pure ZnO films annealed at (400, 500, and 600)^{O}C. The degree of crystallinity improves as the annealing temperature increases.

From figure it is observed that increasing annealing temperature led to an increase in peaks intensity with a decrease in the width of the peaks middle (Full Width at Half Maximum) due to enhances in crystal structure as a result of heat treatment which reduced the crystalline defects, also it's observed that all films have polycrystalline structure with the presence of several peaks belongs to ZnO wurtizite hexagonal structure with dominate (hkl) equal to (101). The results are compatible to JCPD No (36-1451) [17] and in good agreement with [18], and [19]. Figure (2) for pure TiO_2 diffraction peaks are (101,004,200, 221, and 204) for anatase phase and (110,101, and 211) for rutile phase, no Brookite phases are observed in XRD-pattren, results are in good agreement with those given in JCPD data card (JCPDS No. 21 – 1272 & 21 – 1297) [17] for TiO_2 anatase and rutile .It was observed that the intensities of TiO_2 peaks increased slightly with the increase of annealing temperature. This means that TiO_2 films have been crystallized in a tetragonal mixed anatase and rutile form. However, the Full Width at Half Maxima FWHM of the (101) peaks was hardly changed with increasing film annealing temperature, this goes in agreement with the previous works [20], [21] , and [22] . The XRD of composite (ZnO/ TiO_2) films with pointed characteristic peaks of anatase, rutile, and zinc oxide is presented in Figure (3), The peaks of TiO_2 phases that appear in XRD pattren are for anatase, (101) at 2θ =25.281,(112)

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at 2θ = 38.575,(200) at 2θ=48.049,and (105) at 2θ =53.89. For rutile phase peaks are (110) at 2θ =27.446,(111) at 2θ = 41.225,(210) at 2θ=44.05,and (211) at 2θ =54.322. The appearance of TiO_2 phases can be explained by the high composite percentage of TiO_2, so Ti−O entered as substitution atom within the crystalline Zn−O oxide pattern. Results are in good agreement with [23] who study pure ZnO & TiO_2 and composite (ZnO) 90% with TiO_2 (10%). In addition, the location of the stronger peaks is shifted to lower 2θ angles because of the increasing in interplanar spacing caused by crystal defects like strain as shown in Table (1)&(2).

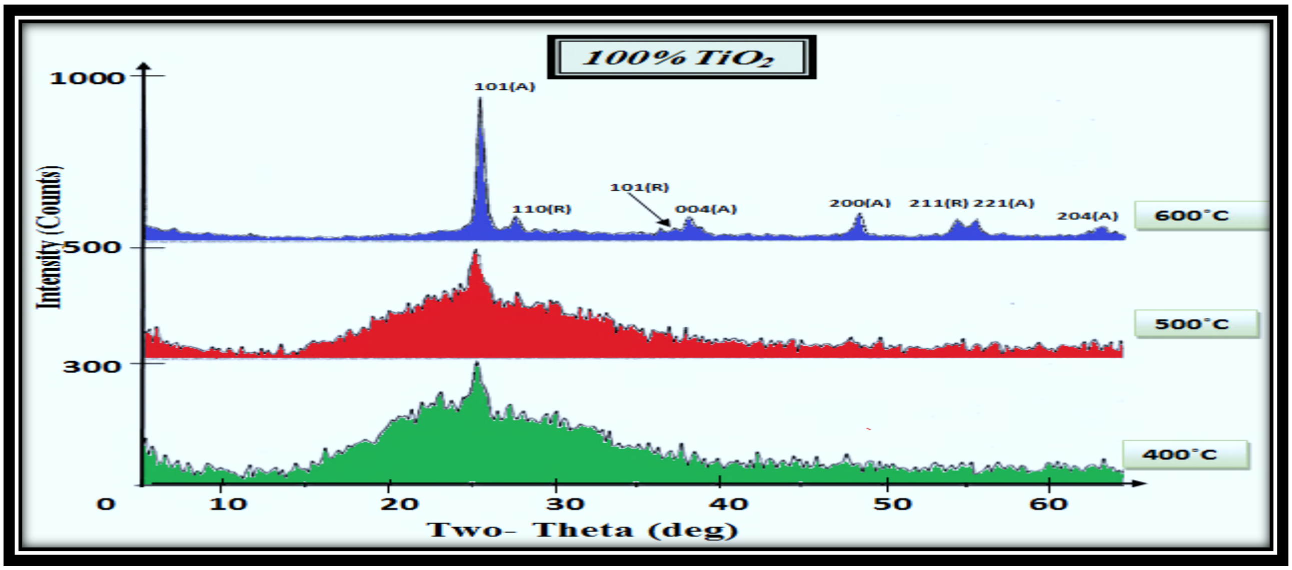

Figure (2): XRD-spectrum for pure TiO2 films post annealed at 400 ^{O}C, 500 ^{O}C, and 600 ^{O}C. The degree of crystalinity improves as the annealing temperature increases

Table (1): Inter planner spacing with corresponding diffraction angle for pure ZnO&TiO_2 films annealing at *(400,500, and 600)* ℃.

Samples	T=400 ℃		T=500 ℃		T=600 ℃	
	2θ	d(101) Å	2θ	d (d(101) Å	2θ	d(101) Å
100%ZnO	36.290	2.4737	36.278	2.4745	36.281	2.4743
100%TiO_2	25.566	3.4813	25.475	3.4936	25.251	3.5242

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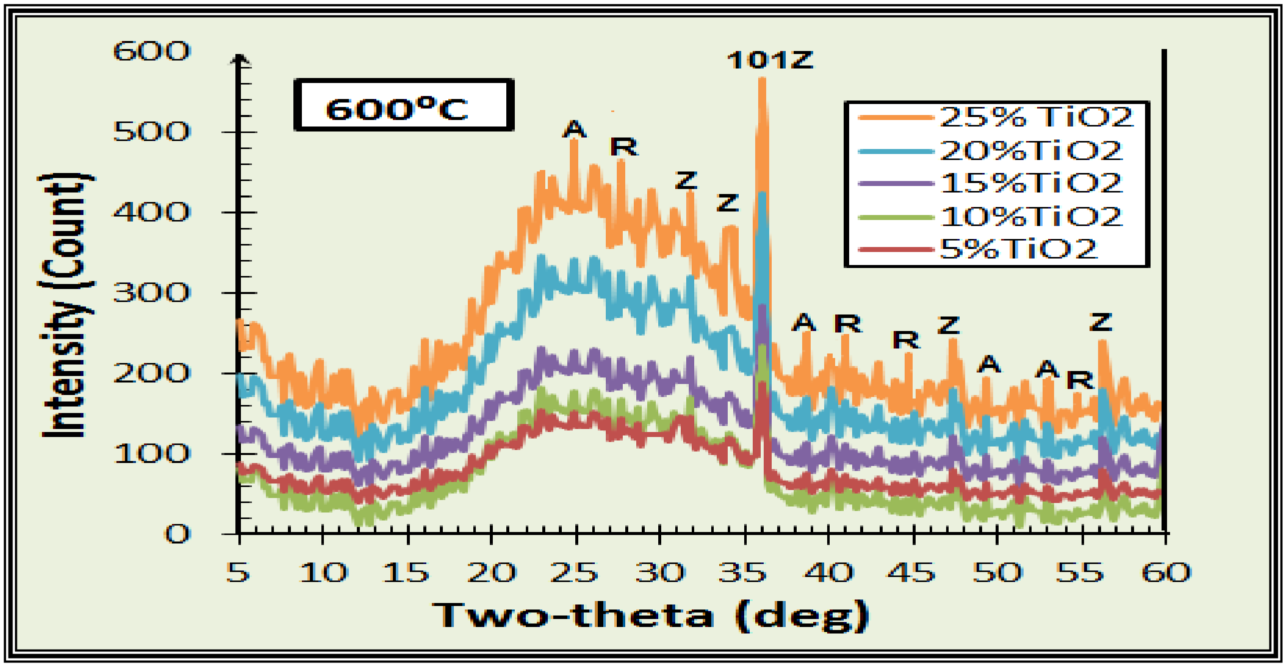

Figure (3): XRD-spectrum for $(ZnO)_{1-x}(TiO_2)_x$ composite films post annealed at 600 ^{o}C for values of x ranges from 5% to 25% in steps of 5%. The composite films are obviously non-crystalline. (A&R represents Anatase & Rutile phases of TiO2, Z represent ZnO phase).

Table (2): Inter planner spacing with corresponding diffraction angle for $(ZnO)_{1-x}(TiO_2)_x$ films annealing at 600^{o}C.

Sample $(ZnO)_{1-x}(TiO_2)_x$	Composition-parameter x					
	0	**0.05**	**0.10**	**0.20**	**0.25**	**1.0**
2θ	36.281	36.264	36.253	36.243	36.215	25.251
d (101) Å	2.4743	2.4754	2.4762	2.4768	2.4787	3.5242

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The AFM-scans for some samples post annealed at 600 ^{o}C are shown in Figure (4).The samples include (100%ZnO), (100% TiO_2), (95%ZnO/5% TiO_2) and (75%ZnO/25% TiO_2). These scans support the XRD results. The average-diameter of the grains and the average-roughness of the surface are best for the 5% sample. Table (3) displays some representative data for average-diameter of grains and the average value of surface-roughness, in nanometers, for x= 0, 0.05, 0.10, 0.20, 025, and x=1.0 for films post annealed at 600. Our results are in good agreement with **[24]**

Table (3): Values of average-diameter of grains (crystallites) and the average - roughness parameter as obtained from AFM-scans for a number of samples annealed at 600 ^{o}C.

Sample $(ZnO)_{1-x}(TiO_2)_x$	**Composition-parameter x**					
	0	**0.05**	**0.10**	**0.20**	**0.25**	**1.0**
Average-diameter [nm]	72.92	83.54	90.69	73.39	78.68	74.74
Average-Roughness [nm]	5.63	0.90	1.75	1.89	1.35	1.42

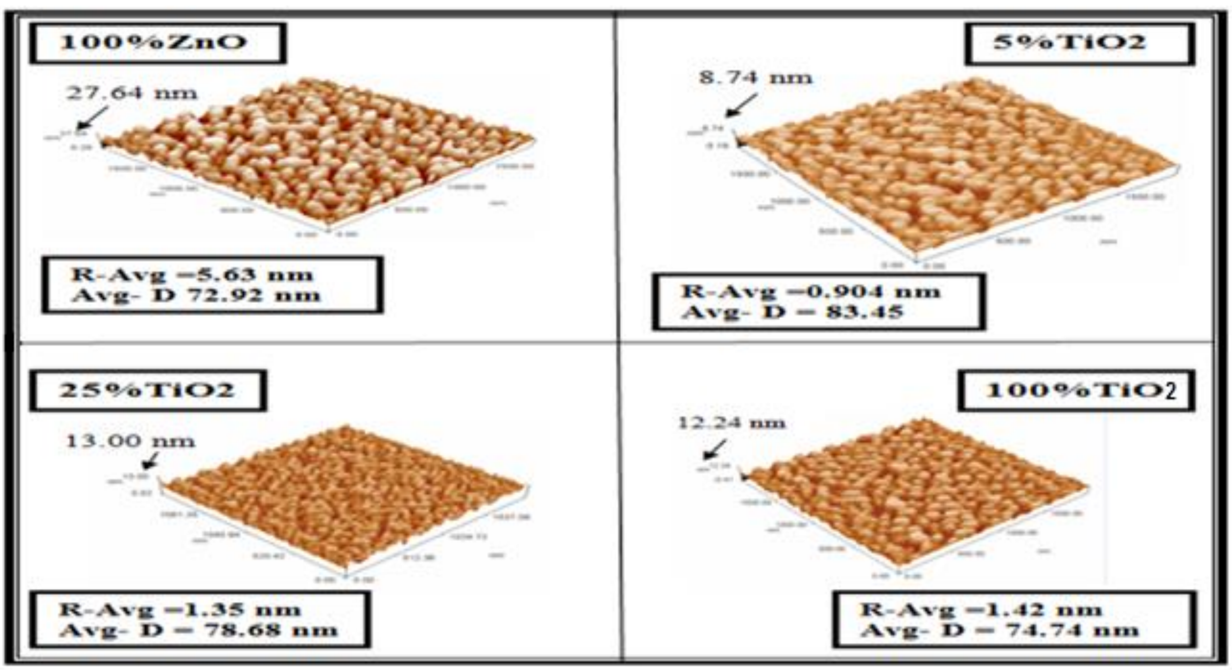

Figure (4): AFM-scans for $(ZnO)_{1-x}(TiO_2)_x$ films post annealed at 600 ^{O}C for values of x=0.0,0.05,0.25,and 1.(R-Avg=Roughness average and Avg –D=Average diameter).

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3.2. Optical properties:

The measured absorbance, transmittance and reflectance of the films which were post annealed at 400 ^{O}C, 500 ^{O}C, and 600 ^{O}C were plotted, it was found that the properties deteriorates; both the absorbance, and reflectance increases while the transmittance decreases as the temperature of annealing is increased, due to heat treatment increase the regularity of the internal arrangement of the atoms and reduce structural defects ,or could be explained by the increased surface roughness of the deposited films as confirmed by AFM[18] The absorption-coefficient (α), refractive-index (n), and extinction coefficient (κ) are calculated from the following relations [25]:

$\alpha = 2.303(\frac{A}{t})$ .... (1).

$n = \frac{1+\sqrt{R}}{1-\sqrt{R}}$ ……….. (2).

$\kappa = \frac{\alpha\lambda}{4\pi}$ .... (3).

Where, λ is the wavelength, A is the absorbance, R is the reflectance and t is the film thickness.

Plots of (α),(κ) , and (n) (not shown because of lack of space) shows that both α and κ increases with increasing annealing temperature, this behavior attributed to the formation of localized levels near connectivity edge,. The absorption- edge (the cutoff wavelength λ_C) shifts marginally towards longer wavelengths. A peak shows in the plot of the refractive-index n, in the range of 370-380 nm wavelengths A similar peak was observed by [18] for pure ZnO films prepared by sol-gel method.Values of α, κ, n, and λ_C for $(ZnO)_{1-X}(TiO_2)_X$ films,; at λ =330 nm in the absorption-band, as shown in Table (4) .

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Table (4): Values of α, κ, n, and λ_C for $(ZnO)_{1-x}(TiO_2)_x$ films at a wavelength λ =330 nm in the absorption-band.

	P p parameter	Films				
		x=0.0	x=0.05	x=0.10	x=0.20	x=0.25
400 °C	α[cm⁻¹]	68869	80839	78445	72859	70465
	n	1. 27	1.4	1.46	1. 8	1.9
	K	0.1809	0.2124	0.2061	0.1914	0.1851
	λ_C[nm]	376	378	379	380	380
500 °C	α[cm⁻¹]	74455	86425	84031	78445	76051
	n	1.57	1.68	1.8	2.2	2.4
	K	0.1956	0.2271	0.2207	0.2061	0.1998
	λ_C[nm]	378	379	380	381	382
600 °C	α[cm⁻¹]	82276	90416	88022	84031	81637
	n	1.94	1.8	2.04	2.26	2.6
	K	0.2103	0.2379	0.2313	0.2208	0.2145
	λ_C[nm]	379	380	381	383	384

3.3.Energy gap and Urbach energy:

The Optical energy gap Eg and absorption coefficient α are related by the equation

$$\alpha = (\frac{\beta}{h\nu})\,(h\nu\text{-}Eg)^{\gamma} \ldots\ldots\ldots (4)$$

Where k = a constant, h = Planck's constant ,hν = The incident photon energy and γ is a number which characterizes the nature of electronic transition between valance band and conduction band. Our films have $\alpha > 10^4$ so they have a direct allowed band gap and for direct allowed transitions β =1/2. Therefore the formula used is

$$\alpha = (\frac{\beta}{h\nu})\,(h\nu\text{-}Eg)^{1/2} \quad \text{which gives} \quad (\alpha h\nu)^2=\beta(h\nu - Eg) \quad \ldots\ldots\ldots (5)$$

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Where β is a constant independent of photon energy .The optical energy-gap E_g, can be deduced from plots of $(\alpha h\nu)^2$-versus-(photon energy $h\nu$), where α is the absorption-coefficient, ν is the frequency of the electromagnetic-wave[26], while values of what is called Urbach energy E_u , can be deduced from plots of $ln(\alpha)$-versus-$h\nu$, in accordance with the following relation [27] $\alpha = \alpha_o \exp(h\nu / E_u)$ .... (6).

Figure (5) shows plots suggested by equation 5, plots suggested by equation 6 were also done but not shown for lack of space. Values of E_g and E_u obtained from the corresponding plots are shown in Table (5). We note from Table that Eg generally decreases slightly with increasing annealing temperature. The value of Eg obtained is close to the values reported by [28] (3.26-3.29 eV), by [29] (3.24-3.40 eV), by [30] (3.24 eV), by [31] (3.27 eV) and by [32] (3.26-3.34 eV).

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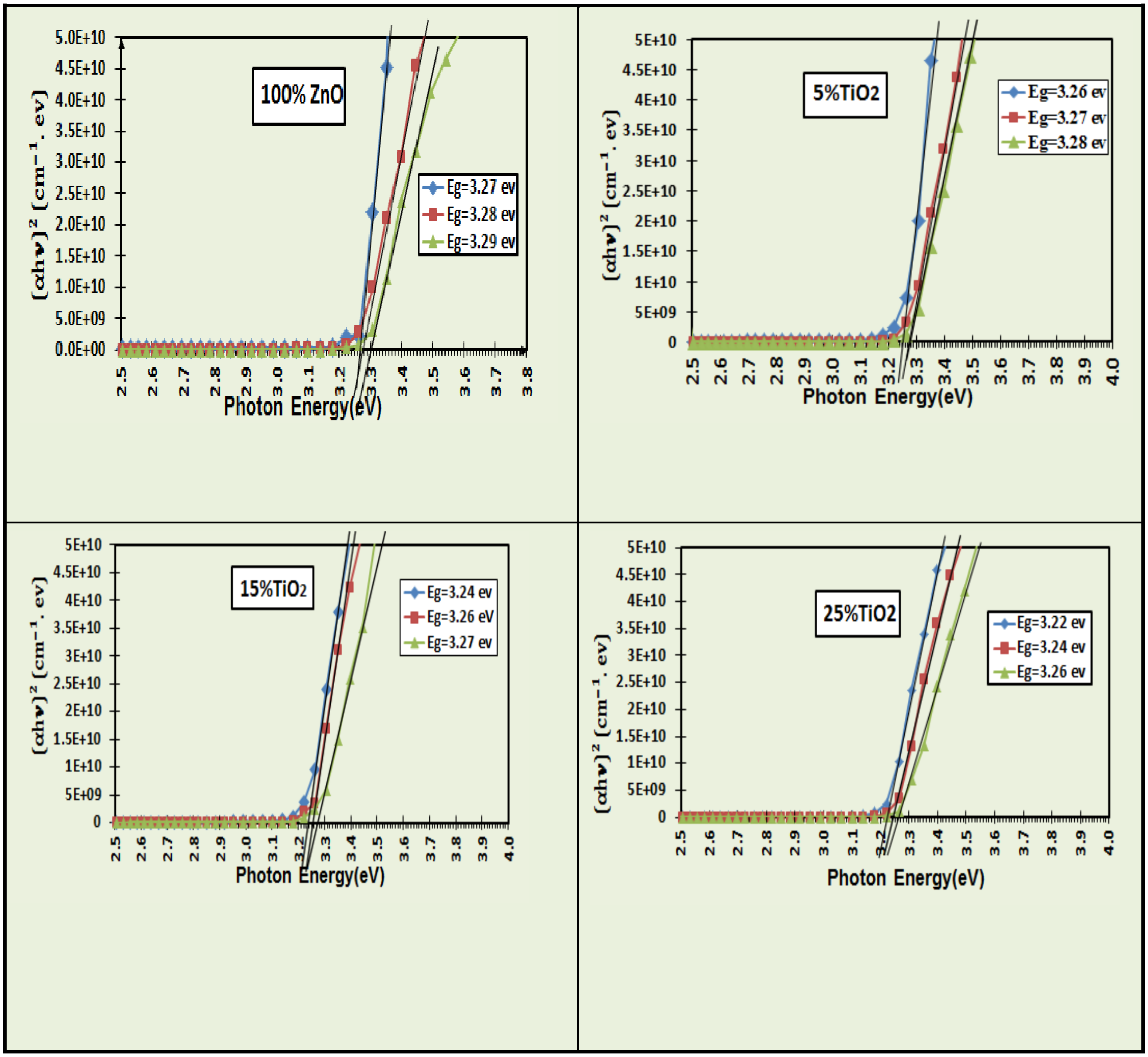

Figure (5): Plots of $(\alpha h\nu)^2$-versus-(photon energy $h\nu$) for $(ZnO)_{1-x}(TiO_2)_x$ composite films post annealed at 400 ^{O}C, 500 ^{O}C, and 600 ^{O}C, for x=0.0, 0.05, 0.15 and 0.25 .

Reference [33] reports somewhat lower values for ZnO films (2.81-2.98 eV) and composite 1:1 ZnO/TiO_2 films (2.71-2.88 eV) as a function of film-thickness (t) ; t=897-4884 nm for ZnO and t=890-3920 nm for the composite 1:1 ZnO/TiO_2 films. Also E_g values generally decreases

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with increasing TiO_2 content in the $(ZnO)_{1-x}(TiO_2)_x$ composite films, because composition process led to the emergence some of the crystalline defects which appear tails (Urbach Tails) in the area between the valence and conduction bands within forbidden energy gap (Eg) which mean that composition process led to existence topical energy levels(Urbach energy) inside energy gap, that make the necessary energy (hν) to obtain electronic transitions less, and electrons moving from the valence to conduction band become easierE_g .Result are in agreement with [34](for ZnO).

Table (5): Values of energy-gap E_g and Urbach-energy E_u, for $(ZnO)_{1-x}(TiO_2)_x$ films annealed at 400 ^oC, 500 ^oC, and 600 ^oC, and for various compositions.

film	Eg [eV]			Eu [meV]		
	400 ^oC	**500 ^oC**	**600 ^oC**	**400 ^oC**	**500 ^oC**	**600 ^oC**
x=0	3.29	3.28	3.27	52	59	73.4
x=0.05	3.28	3.27	3.26	56	60	75.1
x=0.10	3.275	3.265	3.25	58	61	80.8
x=0.15	3.27	3.26	3.24	59	63	82.5
x=0.20	3.265	3.25	3.235	64	70	83
x=0.25	3.26	3.24	3.22	77	78	90

4.CONCLUSIONS

Noting the results of this study as presented in the Tables and figures shown; one may conclude that, the titania and zinc oxides in the composite film exist as separate phases in form of anatase, rutile(TiO_2phases) and wurtzite modification(ZnO phase), the dominating phase can be classified as hexagonal wurtzite ZnO, (101) peak at 36.5° ,and annealing improve the quality of the film in terms of its degree of crystal structure.Also, E_g decreases with both composition-parameter x and annealing-temperature T_a which can be explained by evoking that increasing x will randomize the crystal periodicity, and that the higher annealing temperature relaxes the structure more towards a better crystalline structure. The increase of Urbach-energy, E_u with increased percentage of TiO_2 in the ZnO matrix was attributed to the lower mass of Ti ions as compared to that of Zn ions.

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