

Effect of Substrate and Growth temperature on Zinc Oxide nanorods via Hydrothermal Processing

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Abstract:

In this paper, the hydrothermal processing (HTP) was employed to synthesize Zinc oxide nanorods (ZnO-NRs) arrays on p-type Si(100), and glass substrates at 95°C, and 120 °C. The X-ray diffraction(XRD) measurements confirm that all prepared films are polycrystalline with hexagonal structure and orientation on plane c-axis (002) as shown in FESEM images. The intensity of peak ($2\theta = 34.2^\circ$) was increased and its full width at half maximum (FWHM) decreased with the increase of temperature due to the increase in their particles size. The images of field emission scanning electron microscopy (FESEM) show that ZnO-NRs on Si substrate gives a better result from the glass substrate with an aspect ratio (17.95, and 17.43) for the prepared samples at (120°C, and 95°C) , respectively. The growth temperature effect is clear onto average diameter, average length and aspect ratio, where they increased with increasing temperature. The optical measurement shows that the peak of absorption increased with increasing temperature while the reflection spectra decreased. The band gap of ZnO-NRs on Si was determined from the reflection spectra while for glass substrate was determined from absorption spectra.

Keywords: Zinc Oxide (ZnO); Nanorod arrays; Hydrothermal processing (HTP); various substrates; various temperatures.

تأثير القاعدة و درجة حرارة الانماء على قضبان اوكسيد الخارصين النانوي المحضرة بالطريقة الهيدروحرارية

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الخلاصة:

في هذا البحث تم استخدام الطريقة الهيدروحرارية (HTP) لتحضير مصفوفات اوكسيد الخارصين النانوية (ZnO-NRs) على قواعد السليكون و الزجاج بدرجة حرارة 95°C و 120°C. بينت قياسات حيود الاشعة السينية (XRD) لكل الاغشية المحضرة ان اتجاه النمو المفضل لمركب اوكسيد الخارصين (ZnO) باتجاه المستوي (002) و المحور C و ان الاغشية المحضرة ذات شكل سداسي كذلك لوحظ ذلك من خلال صور المجهر الالكتروني الماسح لانبعاث المجال (FESEM). ان شدة قمة الزاوية ($2\theta = 34.2^\circ$) تزداد بزيادة درجة حرارة الانماء و يقل عرض منتصفها عند قيمته العظمى (FWHM) بزيادة درجة حرارة الانماء بسبب الزيادة الحاصلة بحجمها الحبيبي. بينت فحوصات المجهر الالكتروني FESEM ان اغشية القضبان المحضرة على قاعدة السليكون افضل من قاعدة الزجاج اذ كانت نسبة طولها

لقطرها حوالي (17.95,17.43) المحضرة بدرجة حرارة (120°, 95°) بالترتيب , ان تأثير درجة الحرارة الانماء كان واضح على معدل القطر والطول و النسبة بينهما اذ زادت بزيادة درجة حرارة الانماء. بينت الفحوصات البصرية ان قمة الامتصاص تزداد بزيادة درجة الحرارة بينما نقصان طيف الانعكاسية بزيادة درجة حرارة الانماء. ان قيمة فجوة الطاقة للقضبان المرسبة على قاعدة السليكون تم حسابها بالاعتماد على طيف الانعكاسية و على قاعدة الزجاج من طيف الامتصاصية.

الكلمات المفتاحية: أكسيد الزنك ; مصفوفات نانوية , الطريقة الهيدروحرارية; قواعد مختلفة ; درجات حرارية مختلفة.

1. Introduction

High surface area – volume ratio and other features made semiconductor nanomaterials employ in various devices such as solar cell, light emitting diode, and gas sensor, etc.[1]. A high free exciton binding energy of about (~60 meV) [2] and direct wide band gap (~3.37 eV)[3] made Zinc oxide (ZnO) usage widely compared with another material, especially in laser diodes, solar cells. Generally, ZnO is an n-type semiconductor due to an existence of oxygen vacancy in ZnO and it acts as an electron donor [2]. Otherwise, ZnO can exist in various nanostructures such as nanoparticles, nanorods[4], nanowires[5], and nanotubes[6], etc. These variety nanostructures made ZnO the better candidate in various applications especially in a solar cell due to the high surface to volume ratio. A variety methods used to prepare ZnO nanorods (ZnO-NRs) [7], which are vapor-liquid-solid [8], metal organic chemical vapor deposition [9], pulsed laser deposition [10], hydrothermal [10], and chemical bath deposition (CBD) method [11]. Recently, hydrothermal processing (HTP) has become widely used due to its low cost, easy technique, suitability, the most energy-efficient, and does not need a complex vacuum system [12]. Further, HTP method is an economical and simple for a growth of

ZnO and it obtains diverse morphologies and diverse properties in ZnO nanostructures with high quality [12, 13]. Recently, many studies have been conducted to study the influential factors on ZnO-NRs such as growth temperature [14], time [15], PH [16], seed layer [17], and substrate[18-20], etc. In this study, ZnO-NRs arrays were fabricated with aligned uniform on p-type Si (100), and glass substrates using the HTP at various temperature for the same time without any metal catalyst. The main aim of this work was to study the effect of growth temperature, and substrate type on surface, structure, and optical properties of ZnO-NRs.

2. Experimental Details

2.1 Materials

In this paper, all chemical materials were purchased commercially and used without further purification. The zinc acetate dehydrate (ZAD), zinc nitrate hexahydrate (ZNH), and Diethanolamine (DEA) was equipped by (Himedia Laboratories Pvt-Ltd.). Ammonia solution (NH₃) was equipped by (Sinopharm Chemical Reagent Co., Ltd). Acetone, Ethanol, Hydrochloric Acid (HCl), and Hydrofluoric acid (HF) was equipped by (Honeywell Co., Ltd) with a purity of (99.9%).

The glass and p-type Si (100) substrates were ultrasonically cleaned with HCl, Acetone, ethanol and deionized water. A drying process was then performed in an oven at 200°C for 30 minutes. Then, Si substrates are immersed into a dilute HF solution (20%) for 30 seconds to remove any native oxide layer.

2.2 Preparation of ZnO Seed Layer

The sol-gel solution prepared from dissolved a 0.05M of (ZAD/DEA) into 100 ml ethanol by stirring for 1 hour at 75°C. Then, 8ml of DAE was gradually added to the solution, while stirring constantly at 75°C for 1 hour. After 24 hours, The sol-gel solution was deposited by spin-coating 4 times at 2400 rpm for 1minute on top of clean (Si(100), and glass) substrates. Between layers, the substrates were dried on a hot plate at 180°C for 7 minutes. Finally, all the films were annealing through using an oven at 350 °C for 2 h under atmospheric air to form a homogeneous ZnO seed layer, which acts as a nucleation layer. Finally, the ZnO growth process was conducted by using HTP. The preparation of ZnO Seed Layer (ZnO-SLs) is shown in Figure 1a.

2.3 Growth of ZnO nanorods

After ZnO-SLs deposition, the seeded substrates were placed vertically in an autoclave contains a growth solution at (95, and 120) °C for 6 hours as shown in Figure 1b. The growth solution prepared from dissolved a 0.01M of ZNH, which act as the Zn^{+2} source, into 100 ml deionized water by stirring for 20 minutes at room temperature (RT) by using quartz beakers. Then, 1ml of NH_3 , which act as the OH^- source, was gradually added to the solution until (PH=10), while stirring constantly at RT for 30 minutes. After the growth

operation, the films were thoroughly washed with deionized water to eliminate the residual salts from the surface of the films and then dried with a blower. Finally, all the films were annealed through using an oven at 350 °C for 2 h under atmospheric air. The prepared ZnO-NRs on Si(100) substrate at (95, and 120) °C were defined as (X1, and X2), respectively, and on a glass substrate at (95, and 120) °C is defined as (X3, and X4), respectively.

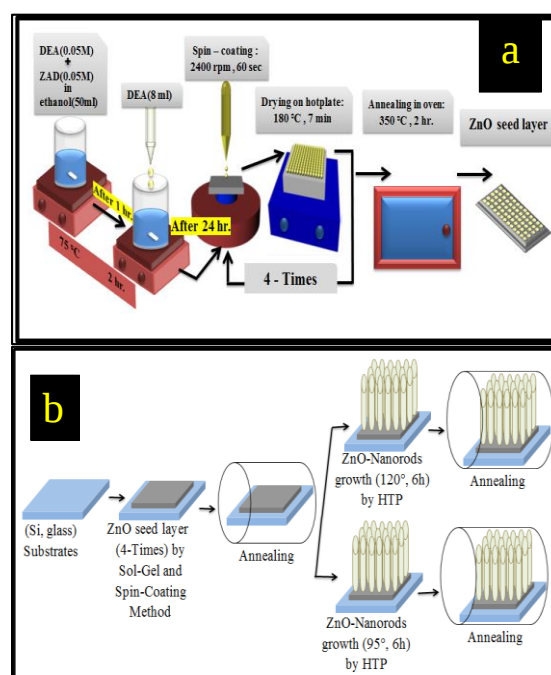


Figure 1: (a) Preparation of ZnO seed layer, (b) Growth of ZnO nanorods arrays.

3. Characterization Methods:

The crystalline structure characteristics of the films (X1-X4) were investigated using X-ray diffraction (XRD) analysis from 20°- 60° (PANalytical X'Pert PRO-MRD with CuK α ($\lambda = 1.54056 \text{ \AA}$) radiation). The surface morphology of the films was analyzed by field-emission scanning electron microscopy (Zeiss Supra 55VP, Germany). The elemental composition of films was studied by using energy dispersive spectrometers (energy

dispersive X-ray spectroscopy EDXS). The optical properties of films were revealed by using a UV-Vis spectrum in the wavelength range of about 300–800 nm with the help of spectrometer (Shimadzu 1800 UV-Vis spectroscopy).

3.1 Structural Studies of ZnO Nanorod Arrays

The XRD patterns of the films (X1-X4) as shown in Figure 2(a-d), respectively, which are a polycrystalline hexagonal (wurtzite) structure of ZnO according to the standard database JCPDS No.36-1451[21]. The diffraction peaks at 31.5°, 34.4°, and 36.1°, which refer to the (100), (002), and (101) planes, respectively. In addition, the diffraction peaks at 47.5, and 56.6° has appeared in X1 and X2, which refer to (102), and (110) planes, respectively. All films have a high sharp intensity of the (002) plane, which indicates the preferred orientation of the nanorods are in the c-axis trend, which indicates that the ZnO-NRs tend to grow perpendicular to the substrate surface [22]. The intensity of (002) plane for the films (X2, and X4) become higher and its full width at half maximum (FWHM) is narrower than (X1, and X3) respectively, due to increasing a growth temperature, this result agrees with [14]. By comparison, The intensity of (002) plane for the films (X1, and X2) as shown in Figure 2(a, and b), are higher increased than (X3, and X4) as shown in Figure 2(c, and d), this result agrees with [20]. The average grain size (D) of all films were calculated by using Scherrer's formula using information on the FWHM (β) of the XRD, the Bragg diffraction angle (θ) of the XRD, and X-ray wavelength ($\lambda = 0.154$ nm), as follows[23]:

$$D = \frac{0.9\lambda}{\beta \cos \theta} \quad (1)$$

In addition, the lattice constants (a, and c) of all films calculated by using the following [3]:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left[\frac{h^2 + hk + l^2}{a^2} \right] + \frac{l^2}{c^2} \quad (2)$$

The Lattice parameters, and c/a ratio of all films inserted in Table 1, which are in agreement with the standard database JCPDS No.36-1451 ($a=0.3249$ nm; $c=0.5206$ nm; $c/a=1.602$) [21]. The average grain size of all films as shown in Table 1, which is increased when growth temperature increased, this result agrees with[14].

3.2 Morphology and distribution of ZnO Nanorod Arrays

Figures 3(a, and b), 4(a, and b), 5(a, and b) and 6(a, and b) shows the FESEM micrographs of the films X1, X2, X3, and X4, respectively. As expected, the coverage density and morphology of the ZnO-NRs arrays on the Si, and glass at (95, and 120) °C are significantly different. The nanorods have hexagonal shapes with a uniform diameter. Figures 3(c), 4(c), 5(c) and 6(c) show the FESEM micrographs cross section of the films X1, X2, X3, and X4, respectively. In figure 5(c), we can find that the nanorods having a uniform hexagonal pyramidal shape while in figure 5(c), we can find that the nanorods having a hexagonal shape. In addition, Figure 7(a), (b), (c) and (d) exhibit the diameters distribution of the films X1, X2, X3, and X4, respectively, in the range of 25-80 nm, 20-100 nm, 20-140 nm, and 20-240 nm, respectively. Figure 8(a), (b), (c) and (d) exhibits the lengths distribution of the films X1, X2, X3, and X4, respectively, in the range of 0.8-1.1 μ m, 0.7-1.4 μ m, 0.54-0.68 μ m, and 0.6-1.6 μ m, respectively. The average diameter, average length and

aspect ratio (average length /average diameter) were inserted in Table1, which are increased with increasing growth temperature due to increasing growth rate of formation of $\text{Zn}(\text{OH})_2$ nuclei and its conversion to ZnO crystal with temperature and ZnO -NRs growth become faster with fewer crystal defects[24, 25]. This result agrees with[14, 26, 27]. The substrate is an important factor affecting the chemical adsorption and nucleation and growth due to the lattice structure and defects on the substrate surface [20]. The ZnO seed layer controls the ZnO nanorods properties such as arrangement, and alignment on the substrate surface, which is affected by the substrate. This effect was studied by many researchers [18, 19]. In our study, we noticed that the ZnO -NRs on Si substrate better than on the glass substrate due to the lattice match between the cubic unit cell of Si substrate and the hexagonal cell of ZnO [28]. There is another reason, the glass has an amorphous structure that means the arrangement of atoms on the substrate surface is random. Thus, the orientation of ZnO -NRs on a glass substrate is random, which is opposite to the ZnO -NRs on Si substrate. This explanation agrees with [29-31].

3.3 Elemental Composition Using EDS

Figures 3(d), 4(d), 5(d) and 6(d) show the (EDX) spectrum analysis of the films X1, X2, X3, and X4, respectively, which contains Zn, O, Si and C peaks. The Si peak appeared in the EDX spectrum in the films X1 and X2 are due to the substrate, while in the films X3 and X4 are due to the glass substrate, that also noticed by[32]. The C peak appeared is caused by the carbon contamination, which happens due to the interaction of the electron beam with the hydrocarbon molecule vapors that exist in the vacuum chamber or on the substrate surface [33]. Thus, the only Zn and O atoms appeared in the EDX spectrum, which confirms that all films are pure of ZnO , this agrees with [34]. The results that obtained from EDX spectra show that Zn/O atomic ratio of all films was wide as shown in Table 1. The substrate effect on ZnO -NRs could be explained depending on the topography, structure, and nature of the substrate that effect on dense ZnO -NRs which causes spaces between the nanorods that allows the high energy electrons of the FESEM to interact with the substrate that produces X-ray fluorescence (XRF) from the constituents of the substrate surface.

Table 1. Lattice parameters, grain size, c/a ratio, average diameter, average length, aspect ratio, Zn/O ratio, and band gap of ZnO -NRs.

Sample	Unit cell (nm)		Grain size (nm)	c/a ratio	Average Diameter (nm)	Average Length (nm)	Aspect ratio	Zn/O	Band Gap (eV)
	a	c							
X1	0.3252	0.5206	59.22	1.601	53	924	17.43	0.68	3.300
X2	0.3250	0.5203	61.18	1.601	54	970	17.96	0.80	3.293
X3	0.3260	0.5216	62.03	1.600	67	604	9.01	0.69	3.268
X4	0.3271	0.5234	70.41	1.600	107	1257	11.74	0.66	3.257

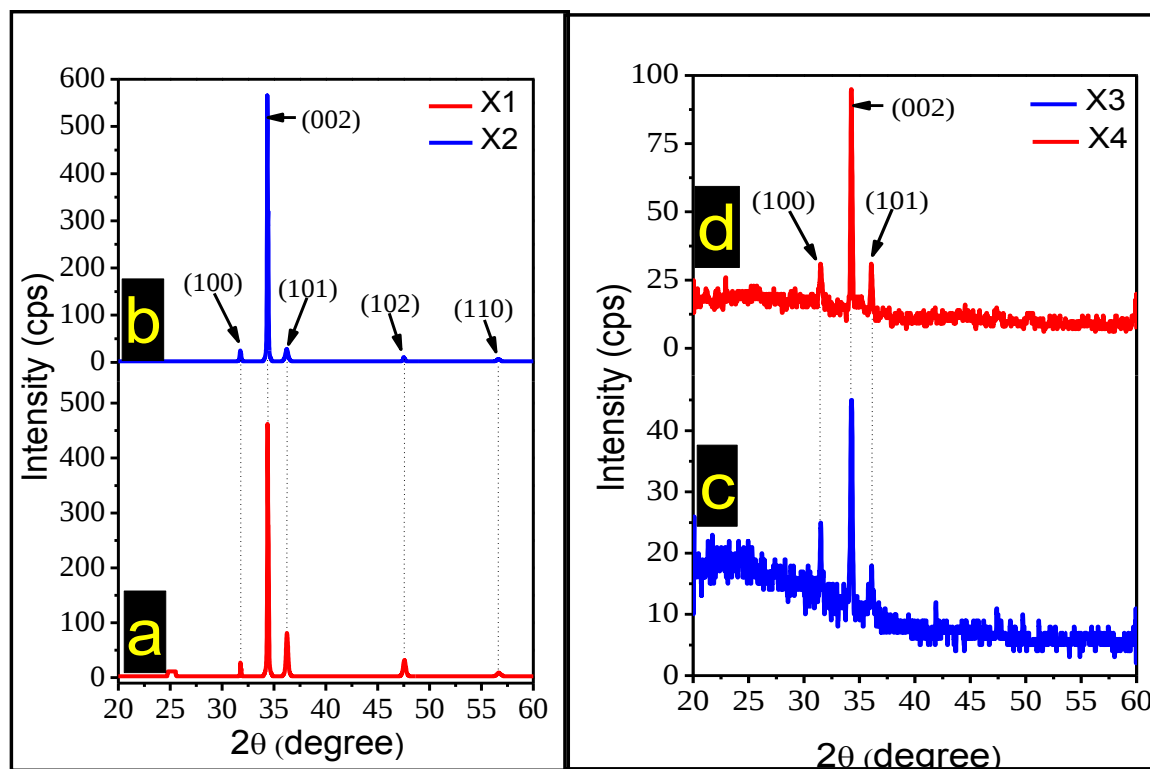


Figure 2: XRD spectra of films: (a) X1, (b) X2, (c) X3, and (d) X4.

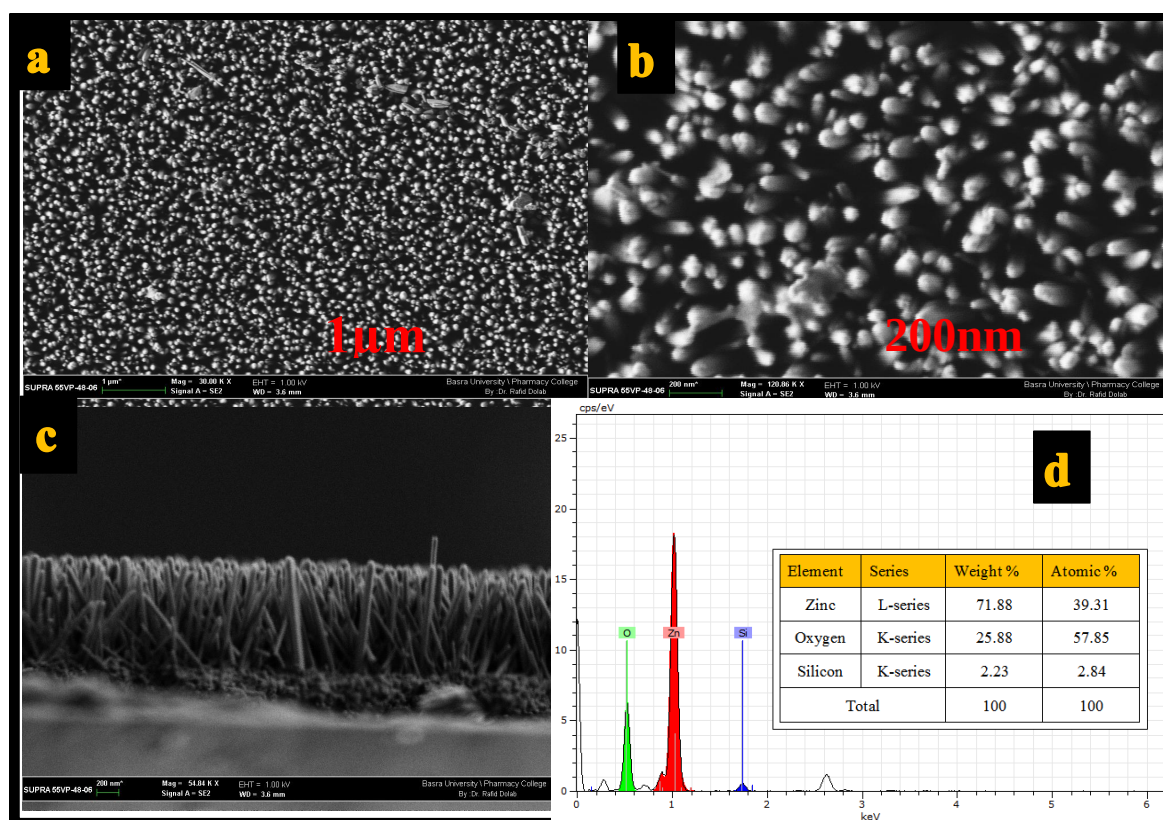


Figure 3: FE-SEM images of the film X1: (a) ZnO-NRs with scale bar 1μm, (b) enlarged view of nanorods with scale bar 200 nm, (c) Cross section of ZnO-NRs, and (f) EDX analysis of ZnO-NRs.

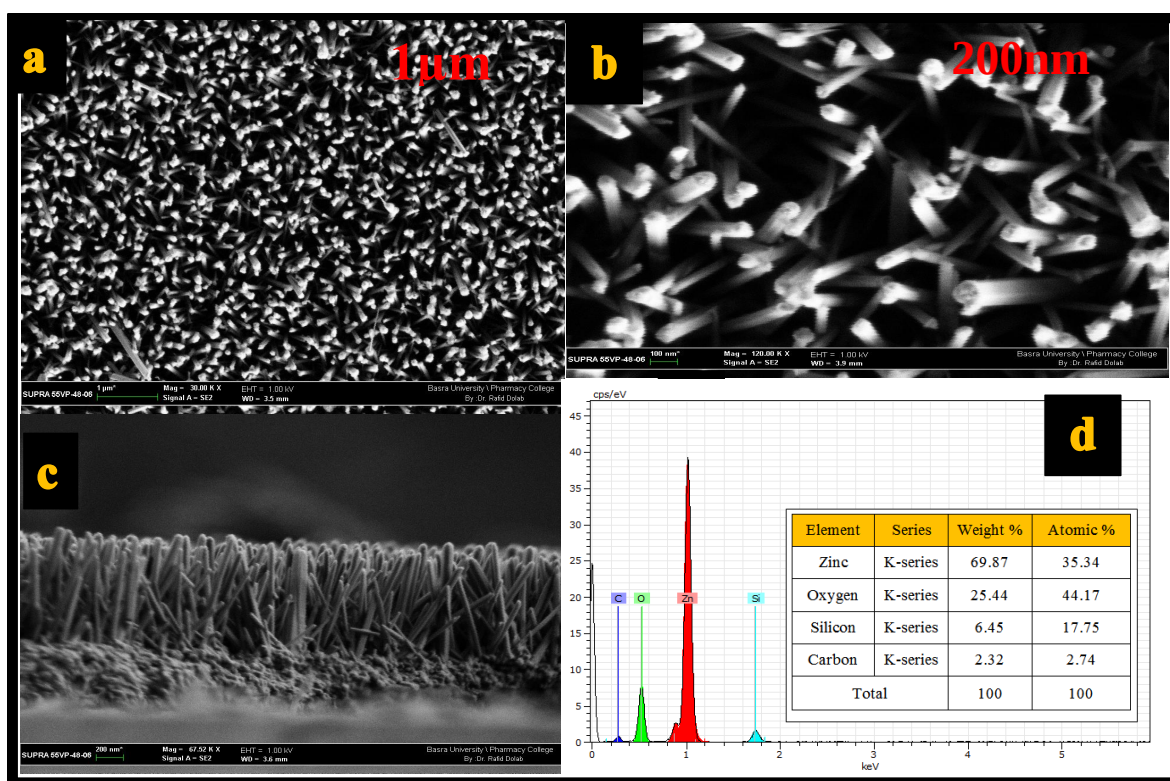


Figure 4: FE-SEM images of the film X2: (a) ZnO-NRs with scale bar $1\mu\text{m}$, (b) enlarged view of nanorods with scale bar 200 nm, (c) Cross section of ZnO-NRs, and (f) EDX analysis of ZnO-NRs.

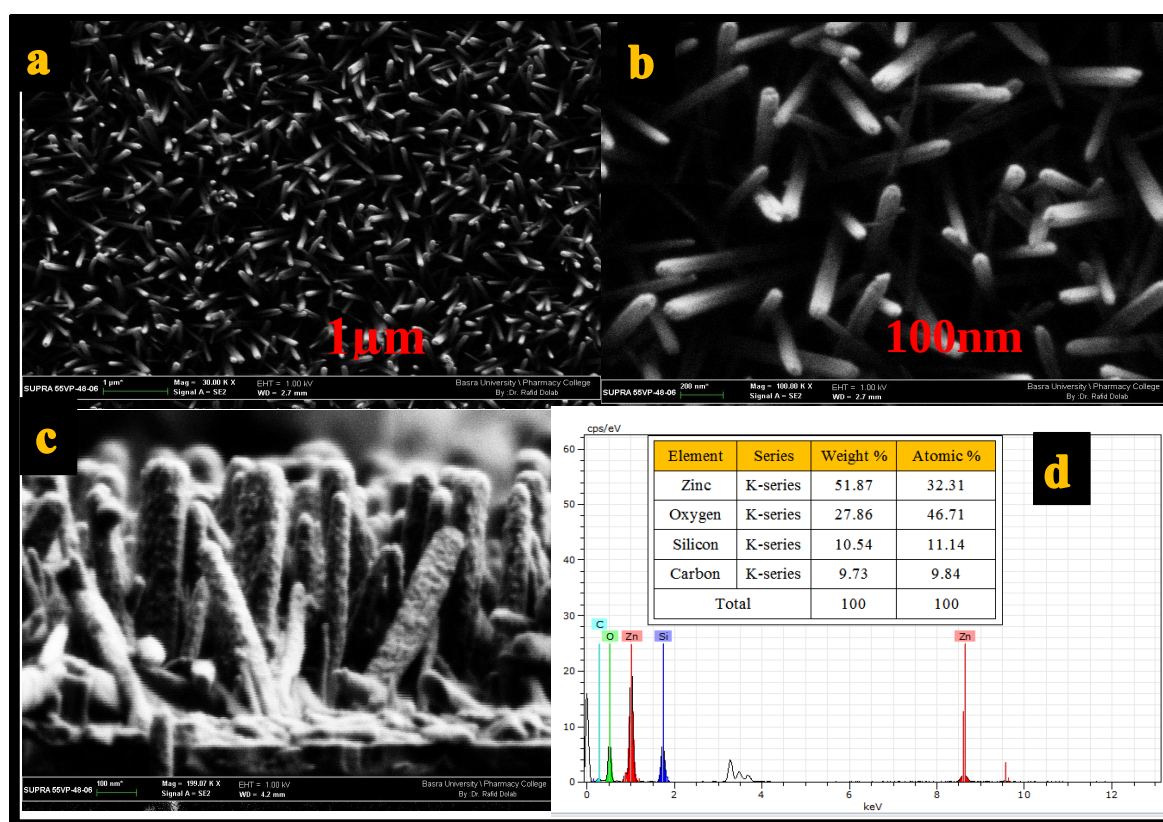


Figure 5: FE-SEM images of the film X3: (a) ZnO-NRs with scale bar $1\mu\text{m}$, (b) enlarged view of nanorods with scale bar 200 nm, (c) Cross section of ZnO-NRs, and (f) EDX analysis of ZnO-NRs.

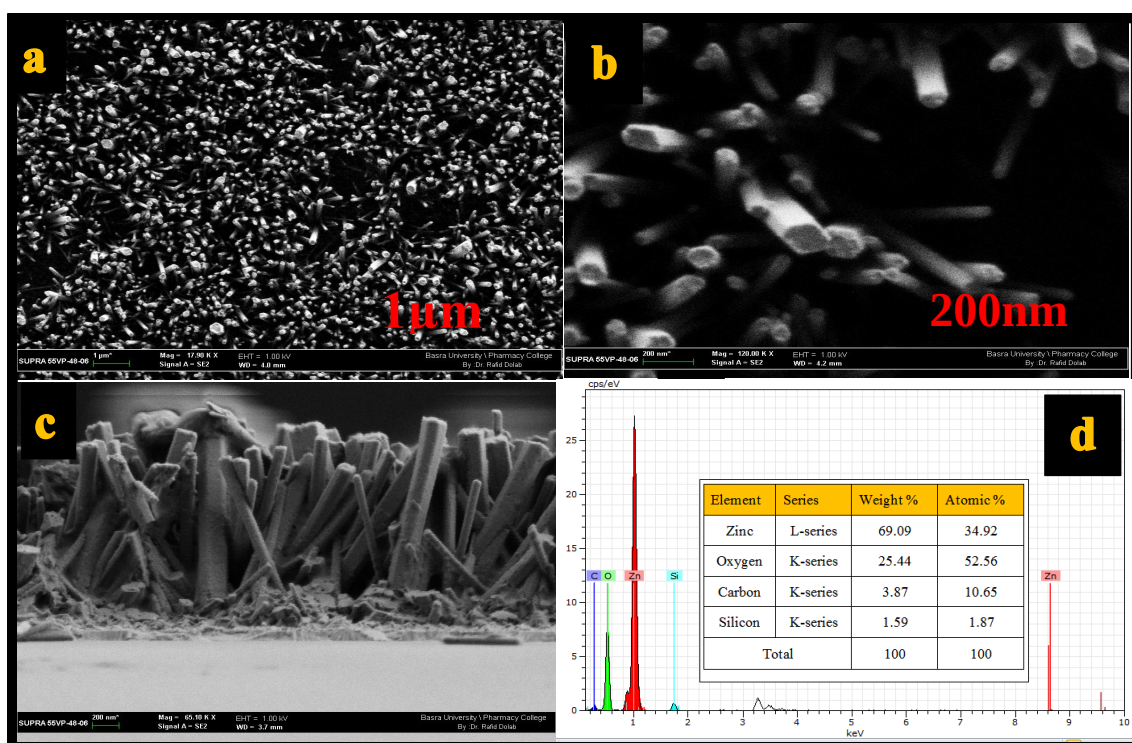


Figure 6: FE-SEM images of the film X4: (a) ZnO-NRs with scale bar 1μm, (b) enlarged view of nanorods with scale bar 200 nm, (c) Cross section of ZnO-NRs, and (f) EDX analysis of ZnO-NRs.

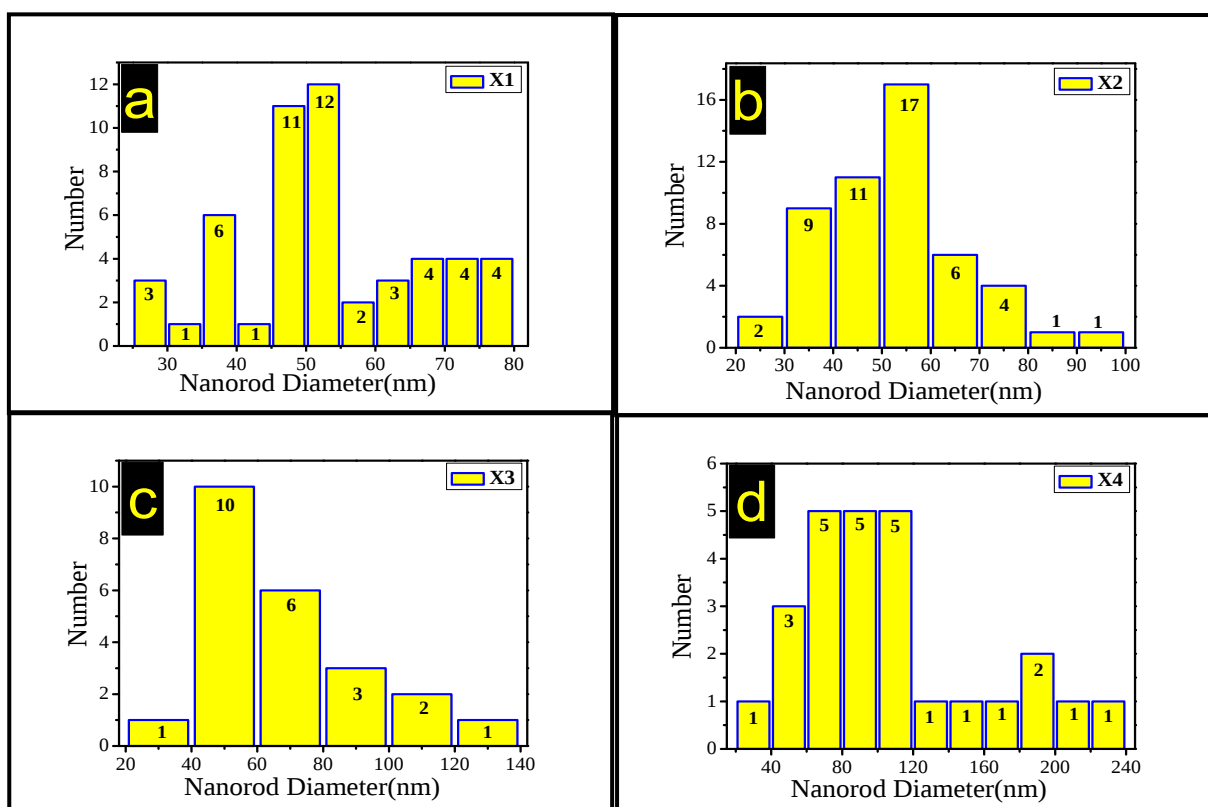


Figure 7: The diameters distribution of the films : (a) X1, (b) X2, (c) X3, and (d) X4.

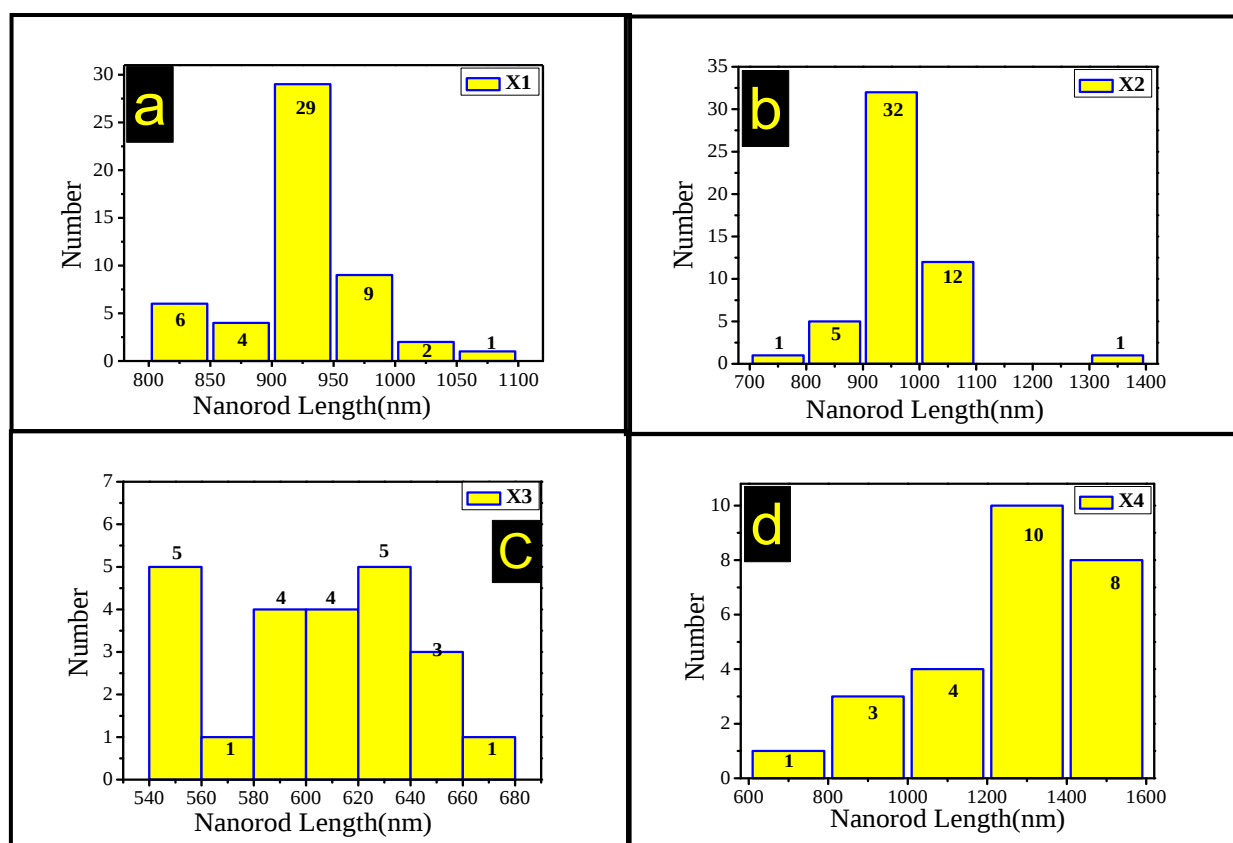


Figure 8: The lengths distribution of the films: (a) X1, (b) X2, (c) X3, and (d) X4.

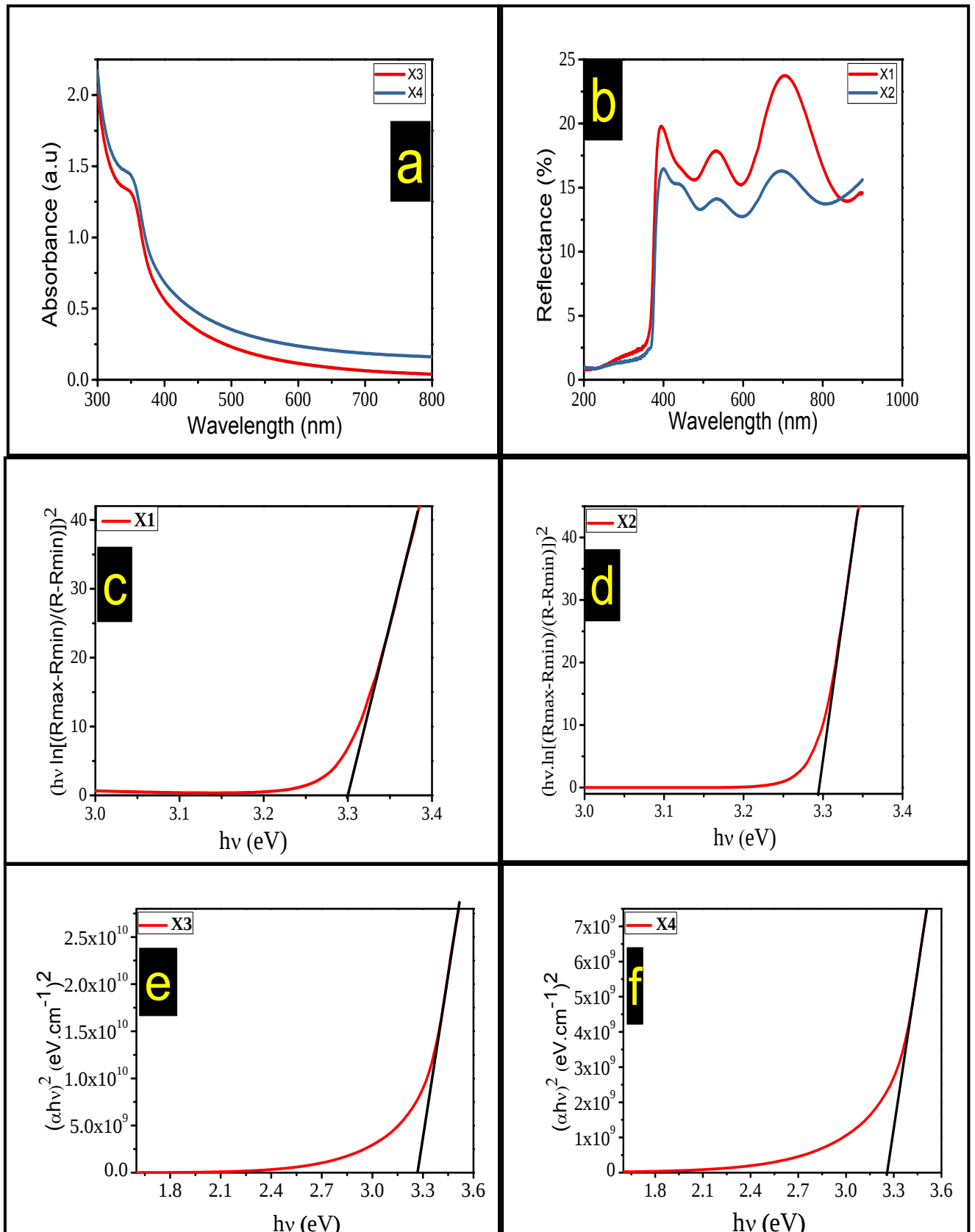


Figure 9: (a) The reflection spectrum of X1 and X2. (b) The absorption spectrum of X3 and X4. The optical band gap (E_g) of the films : (c) X1, (d) X2, (e) X3, and (f) X4.

3.4 Optical Properties

Figure 9(a) shows UV-V absorption of the films X3 and X4 with an absorption edge at about 379 nm and 380 nm, respectively, which are less than 380 nm that returning to bulk ZnO, indicating that ZnO nanostructures grown [35]. A high transmission in the films X3 and X4 at energies lower than a band gap, which is described by absorption spectrum. The reflection spectrum of the films X1 and X2 as shown in Figure 9(b). The optical band gap (E_g) of the films X3 and X4 were determined using the Tauc relationship using information on the photon energy ($h\nu$), and absorption coefficient (α), which depends on the absorbance (Abs.) and film thickness (d), as follows [36]:

$$\alpha h\nu = A(h\nu - E_g)^n \quad (3)$$

$$\alpha = \frac{2.303 \text{ Abs.}}{d} \quad (4)$$

Where A is a constant, and $n = 1/2$ for direct band gap material. When $(\alpha h\nu)^2$ is plotted as a function of ($h\nu$), the linear portion of the curve is extrapolated to $(\alpha h\nu)^2 = 0$. The band gap of the films X1 and X2 were determined using the reflection spectrum, which verified this method for several semiconducting materials such as CdS, ZnS, Si, ZnO etc. This determination method as follows [37, 38]:

$$2\alpha d = \ln \left[\frac{R_{\max} - R_{\min}}{R - R_{\min}} \right] \quad (5)$$

Where d is the thickness of the film, R is reflectance at any wavelength, and $R_{\max}$ and $R_{\min}$ are the maximum and minimum value of reflectance For direct band gap material. From Eqs. (3) and (5) it is clear that there is a proportionality relation between $\ln \left[\frac{R_{\max} - R_{\min}}{R - R_{\min}} \right]$ and α . This behavior is the same of the absorption spectrum

behavior Eq(3). Similarly, $\left(\ln \left[\frac{R_{\max} - R_{\min}}{R - R_{\min}} \right] \right)^2$ is plotted as a function of ($h\nu$), the linear portion of the curve is extrapolated to $\left(h\nu \ln \left[\frac{R_{\max} - R_{\min}}{R - R_{\min}} \right] \right)^2 = 0$. The band gap of the films X1, X2, X3, and X4 as shown in Figure 9(c), (d), (e), and (f), respectively, and inserted in Table 1. These values are near for the result of researchers [35, 39]. The absorbance of the films X3 and X4 was increased with increasing growth temperature due to increase in their average diameter and length, this agrees with [26]. The reflectance of the films X1 and X2 was decreased with increasing growth temperature

4. Conclusions

The ZnO-NRs films could be prepared by inexpensive and simple the hydrothermal processing. From the results of XRD and FESEM, the ZnO-NRs on Si substrate gives a better result from the glass substrate and the ZnO-NRs prepared at 120°C was better than prepared at 95°C. The absorption edge was decreased with the increase growth temperature. The lowest absorption edge at 375 nm is observed on Si substrates while the growth temperature is equal to 95°C. This high-quality ZnO-NRs can be applied to the gas sensor, and photodetector. The ZnO-NRs could be prepared on ITO substrate to be applied into a dye-sensitized solar cell.

Acknowledgments

The authors gratefully acknowledge support from a university of Basrah.

5. Reference

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